



Full wwPDB X-ray Structure Validation Report ⓘ

Sep 12, 2016 – 07:37 AM EDT

PDB ID : 5K5N
Title : Crystal structure of GSK-3beta complexed with PF-04802367, a highly selective brain-penetrant kinase inhibitor
Authors : Kurumbail, R.G.; Chang, J.S.
Deposited on : 2016-05-23
Resolution : 2.20 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

<http://wwpdb.org/validation/2016/XrayValidationReportHelp>

with specific help available everywhere you see the ⓘ symbol.

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

MolProbity : 4.02b-467
Mogul : 1.7.1 (RC1), CSD as537be (2016)
Xtriage (Phenix) : 1.9-1692
EDS : rb-20027939
Percentile statistics : 20151230.v01 (using entries in the PDB archive December 30th 2015)
Refmac : 5.8.0135
CCP4 : 6.5.0
Ideal geometry (proteins) : Engh & Huber (2001)
Ideal geometry (DNA, RNA) : Parkinson et al. (1996)
Validation Pipeline (wwPDB-VP) : rb-20027939

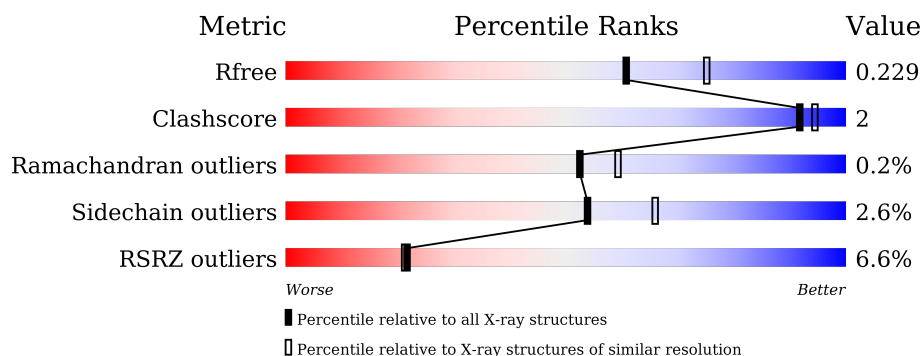
1 Overall quality at a glance

The following experimental techniques were used to determine the structure:

X-RAY DIFFRACTION

The reported resolution of this entry is 2.20 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}	91344	3774 (2.20-2.20)
Clashscore	102246	4477 (2.20-2.20)
Ramachandran outliers	100387	4404 (2.20-2.20)
Sidechain outliers	100360	4405 (2.20-2.20)
RSRZ outliers	91569	3781 (2.20-2.20)

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 ≥ 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	370	<div> <div>5%</div> <div> <div></div> <div>84%</div> <div>6%</div> <div>10%</div> </div> </div>
1	B	370	<div> <div>7%</div> <div> <div></div> <div>83%</div> <div>7%</div> <div>9%</div> </div> </div>

2 Entry composition

There are 4 unique types of molecules in this entry. The entry contains 5484 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 Glycogen synthase kinase-3 beta.

Mol	Chain	Residues	Atoms						ZeroOcc	AltConf	Trace
1	A	332	Total	C	N	O	P	S	0	0	0
			2637	1695	445	485	1	11			
1	B	335	Total	C	N	O	P	S	0	0	0
			2638	1695	446	485	1	11			

There are 28 discrepancies between the modelled and reference sequences:

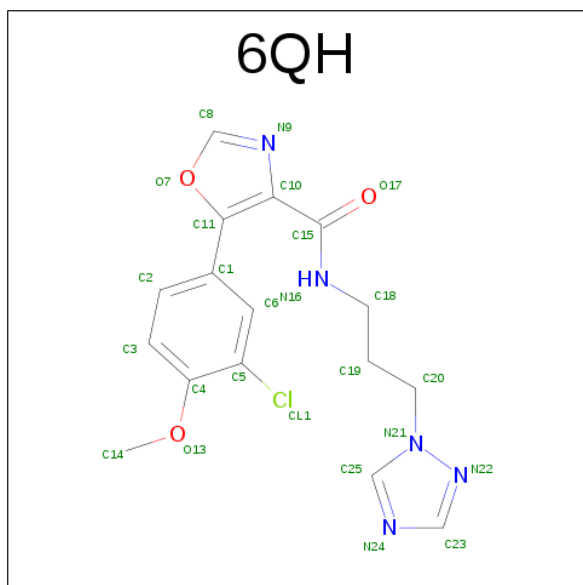
Chain	Residue	Modelled	Actual	Comment	Reference
A	15	HIS	-	expression tag	UNP P49841
A	16	HIS	-	expression tag	UNP P49841
A	17	HIS	-	expression tag	UNP P49841
A	18	HIS	-	expression tag	UNP P49841
A	19	HIS	-	expression tag	UNP P49841
A	20	HIS	-	expression tag	UNP P49841
A	21	SER	-	expression tag	UNP P49841
A	22	SER	-	expression tag	UNP P49841
A	23	GLY	-	expression tag	UNP P49841
A	24	LEU	-	expression tag	UNP P49841
A	25	VAL	-	expression tag	UNP P49841
A	26	PRO	-	expression tag	UNP P49841
A	27	ARG	-	expression tag	UNP P49841
A	28	GLY	VAL	engineered mutation	UNP P49841
B	15	HIS	-	expression tag	UNP P49841
B	16	HIS	-	expression tag	UNP P49841
B	17	HIS	-	expression tag	UNP P49841
B	18	HIS	-	expression tag	UNP P49841
B	19	HIS	-	expression tag	UNP P49841
B	20	HIS	-	expression tag	UNP P49841
B	21	SER	-	expression tag	UNP P49841
B	22	SER	-	expression tag	UNP P49841
B	23	GLY	-	expression tag	UNP P49841
B	24	LEU	-	expression tag	UNP P49841
B	25	VAL	-	expression tag	UNP P49841

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Chain	Residue	Modelled	Actual	Comment	Reference
B	26	PRO	-	expression tag	UNP P49841
B	27	ARG	-	expression tag	UNP P49841
B	28	GLY	VAL	engineered mutation	UNP P49841

- Molecule 2 is 5-(3-chloranyl-4-methoxy-phenyl)- {N}-[3-(1,2,4-triazol-1-yl)propyl]-1,3-oxazole-4-carboxamide (three-letter code: 6QH) (formula: C₁₆H₁₆ClN₅O₃).



Mol	Chain	Residues	Atoms					ZeroOcc	AltConf
2	A	1	Total	C	Cl	N	O	0	0
			25	16	1	5	3		
2	B	1	Total	C	Cl	N	O	0	0
			25	16	1	5	3		

- Molecule 3 is SULFATE ION (three-letter code: SO4) (formula: O₄S).

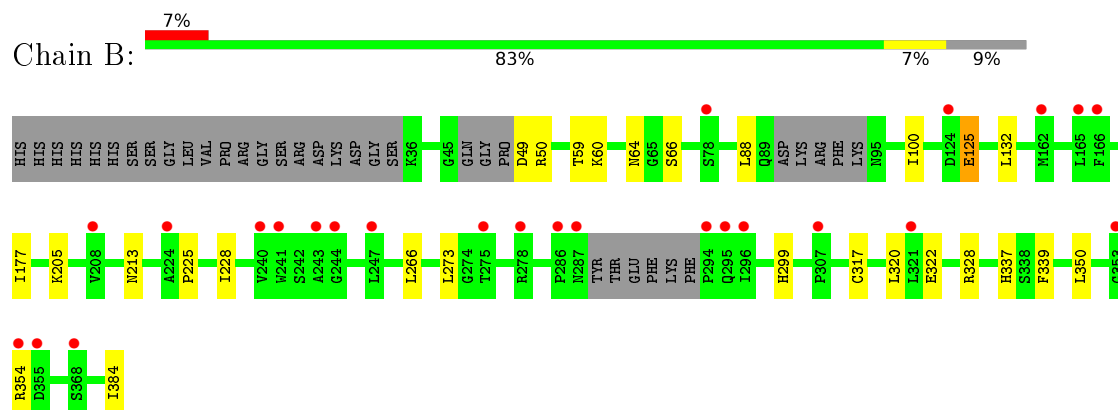


Mol	Chain	Residues	Atoms			ZeroOcc	AltConf
3	A	1	Total	O	S	0	0
			5	4	1		
3	B	1	Total	O	S	0	0
			5	4	1		

- Molecule 4 is water.

Mol	Chain	Residues	Atoms		ZeroOcc	AltConf
4	A	86	Total	O	0	0
			86	86		
4	B	63	Total	O	0	0
			63	63		

- Molecule 1: Glycogen synthase kinase-3 beta



4 Data and refinement statistics

Property	Value	Source
Space group	P 1 21 1	Depositor
Cell constants a, b, c, α , β , γ	66.29 Å 99.91 Å 66.91 Å 90.00° 102.90° 90.00°	Depositor
Resolution (Å)	27.03 – 2.20 25.89 – 2.20	Depositor EDS
% Data completeness (in resolution range)	97.8 (27.03-2.20) 97.8 (25.89-2.20)	Depositor EDS
R_{merge}	(Not available)	Depositor
R_{sym}	(Not available)	Depositor
$\langle I/\sigma(I) \rangle$ ¹	2.15 (at 2.20 Å)	Xtriage
Refinement program	BUSTER 2.11.6	Depositor
R, R_{free}	0.204 , 0.229 0.203 , 0.229	Depositor DCC
R_{free} test set	2121 reflections (5.29%)	DCC
Wilson B-factor (Å ²)	46.2	Xtriage
Anisotropy	0.080	Xtriage
Bulk solvent k_{sol} (e/Å ³), B_{sol} (Å ²)	0.34 , 48.8	EDS
L-test for twinning ²	$\langle L \rangle = 0.49$, $\langle L^2 \rangle = 0.33$	Xtriage
Estimated twinning fraction	0.023 for l,-k,h	Xtriage
F_o, F_c correlation	0.96	EDS
Total number of atoms	5484	wwPDB-VP
Average B, all atoms (Å ²)	58.0	wwPDB-VP

Xtriage's analysis on translational NCS is as follows: *The largest off-origin peak in the Patterson function is 4.77% of the height of the origin peak. No significant pseudotranslation is detected.*

¹Intensities estimated from amplitudes.

²Theoretical 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: 6QH, SO4, PTR

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	0.44	0/2682	0.61	0/3652
1	B	0.44	0/2683	0.62	0/3655
All	All	0.44	0/5365	0.61	0/7307

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	2637	0	2636	11	0
1	B	2638	0	2627	12	0
2	A	25	0	0	0	0
2	B	25	0	0	0	0
3	A	5	0	0	0	0
3	B	5	0	0	0	0
4	A	86	0	0	0	0
4	B	63	0	0	0	0
All	All	5484	0	5263	22	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 (22) 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:263:VAL:HG23	1:B:64:ASN:HB2	1.81	0.61
1:B:337:HIS:HD2	1:B:339:PHE:H	1.47	0.60
1:A:155:VAL:HG12	1:A:159:LYS:HE3	1.83	0.60
1:A:322:GLU:HB2	1:A:328:ARG:HG2	1.89	0.54
1:A:225:PRO:HA	1:A:228:ILE:HD12	1.89	0.54
1:B:322:GLU:HB2	1:B:328:ARG:HG2	1.89	0.53
1:A:228:ILE:HG21	1:A:266:LEU:HB2	1.91	0.52
1:A:332:LEU:HD11	1:A:367:LEU:HA	1.92	0.52
1:B:225:PRO:HA	1:B:228:ILE:HD12	1.93	0.51
1:B:88:LEU:HD11	1:B:125:GLU:HG2	1.93	0.51
1:B:320:LEU:O	1:B:328:ARG:HD2	2.11	0.51
1:A:320:LEU:O	1:A:328:ARG:HD2	2.10	0.51
1:A:337:HIS:HD2	1:A:339:PHE:H	1.57	0.51
1:B:350:LEU:HD12	1:B:354:ARG:HB2	1.97	0.46
1:B:228:ILE:HG21	1:B:266:LEU:HB2	1.97	0.45
1:A:337:HIS:CD2	1:A:339:PHE:H	2.33	0.45
1:B:337:HIS:CD2	1:B:339:PHE:H	2.30	0.45
1:A:138:THR:HG22	1:A:188:LEU:HD23	2.01	0.43
1:B:205:LYS:NZ	1:B:213:ASN:HD22	2.17	0.43
1:B:100:ILE:HG23	1:B:177:ILE:HD13	1.99	0.43
1:A:270:ILE:HG21	1:A:294:PRO:HD2	2.03	0.40
1:B:49:ASP:HB3	1:B:50:ARG:H	1.72	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	321/370 (87%)	315 (98%)	6 (2%)	0	100	100
1	B	326/370 (88%)	318 (98%)	7 (2%)	1 (0%)	46	50

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Mol	Chain	Analysed	Favoured	Allowed	Outliers	Percentiles
All	All	647/740 (87%)	633 (98%)	13 (2%)	1 (0%)	52 59

All (1) Ramachandran outliers are listed below:

Mol	Chain	Res	Type
1	B	125	GLU

5.3.2 Protein sidechains ⓘ

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	292/328 (89%)	285 (98%)	7 (2%)	57 69
1	B	289/328 (88%)	281 (97%)	8 (3%)	51 63
All	All	581/656 (89%)	566 (97%)	15 (3%)	54 66

All (15) residues with a non-rotameric sidechain are listed below:

Mol	Chain	Res	Type
1	A	59	THR
1	A	69	VAL
1	A	77	ASP
1	A	95	ASN
1	A	152	THR
1	A	275	THR
1	A	332	LEU
1	B	59	THR
1	B	60	LYS
1	B	66	SER
1	B	132	LEU
1	B	273	LEU
1	B	299	HIS
1	B	317	CYS
1	B	384	ILE

Some sidechains can be flipped to improve hydrogen bonding and reduce clashes. All (7) such

sidechains are listed below:

Mol	Chain	Res	Type
1	A	72	GLN
1	A	173	HIS
1	A	213	ASN
1	A	337	HIS
1	B	145	HIS
1	B	213	ASN
1	B	337	HIS

5.3.3 RNA ⓘ

There are no RNA molecules in this entry.

5.4 Non-standard residues in protein, DNA, RNA chains ⓘ

2 non-standard protein/DNA/RNA residues are modelled in this entry.

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	$\# Z > 2$
1	PTR	A	216	1	13,16,17	1.37	2 (15%)	19,22,24	1.14	0
1	PTR	B	216	1	13,16,17	1.56	4 (30%)	19,22,24	0.94	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
1	PTR	A	216	1	-	0/9/11/13	0/1/1/1
1	PTR	B	216	1	-	0/9/11/13	0/1/1/1

All (6) bond length outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(Å)	Ideal(Å)
1	B	216	PTR	CE1-CZ	2.07	1.42	1.38
1	A	216	PTR	CD2-CG	2.10	1.43	1.38
1	B	216	PTR	CE2-CD2	2.33	1.43	1.38
1	B	216	PTR	CE2-CZ	2.38	1.43	1.38
1	A	216	PTR	CE1-CZ	2.43	1.43	1.38
1	B	216	PTR	CD2-CG	2.44	1.44	1.38

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.

5.5 Carbohydrates [i](#)

There are no carbohydrates in this entry.

5.6 Ligand geometry [i](#)

4 ligands are modelled in this entry.

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	6QH	A	4001	-	19,27,27	0.35	0	19,36,36	0.34	0
3	SO4	A	4002	-	4,4,4	0.41	0	6,6,6	0.40	0
2	6QH	B	401	-	19,27,27	0.37	0	19,36,36	0.30	0
3	SO4	B	402	-	4,4,4	0.35	0	6,6,6	0.23	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	6QH	A	4001	-	-	0/11/17/17	0/2/3/3
3	SO4	A	4002	-	-	0/0/0/0	0/0/0/0
2	6QH	B	401	-	-	0/11/17/17	0/2/3/3
3	SO4	B	402	-	-	0/0/0/0	0/0/0/0

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.

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 ⓘ

6.1 Protein, DNA and RNA chains ⓘ

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, 95th 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 ²)	Q<0.9
1	A	331/370 (89%)	0.25	19 (5%) 27 27	31, 55, 89, 131	0
1	B	334/370 (90%)	0.33	25 (7%) 17 17	35, 55, 91, 134	0
All	All	665/740 (89%)	0.29	44 (6%) 22 21	31, 55, 89, 134	0

All (44) RSRZ outliers are listed below:

Mol	Chain	Res	Type	RSRZ
1	A	294	PRO	8.0
1	B	296	ILE	4.5
1	B	294	PRO	4.5
1	B	307	PRO	4.2
1	A	289	THR	4.2
1	A	286	PRO	4.1
1	B	124	ASP	3.9
1	A	288	TYR	3.8
1	A	293	PHE	3.7
1	B	165	LEU	3.6
1	B	287	ASN	3.6
1	B	224	ALA	3.5
1	B	241	TRP	3.5
1	B	243	ALA	3.3
1	B	286	PRO	3.3
1	B	295	GLN	3.1
1	B	275	THR	3.0
1	B	244	GLY	3.0
1	B	353	GLY	2.9
1	A	308	ARG	2.9
1	A	209	ARG	2.8
1	B	321	LEU	2.8
1	B	162	MET	2.8
1	A	126	VAL	2.7

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Mol	Chain	Res	Type	RSRZ
1	B	355	ASP	2.6
1	A	169	LEU	2.6
1	B	278	ARG	2.6
1	A	210	GLY	2.6
1	B	354	ARG	2.6
1	A	125	GLU	2.5
1	A	119	SER	2.5
1	A	77	ASP	2.5
1	A	165	LEU	2.4
1	B	247	LEU	2.4
1	B	166	PHE	2.4
1	A	243	ALA	2.4
1	B	368	SER	2.3
1	A	307	PRO	2.3
1	A	124	ASP	2.3
1	A	37	VAL	2.3
1	B	240	VAL	2.2
1	A	240	VAL	2.2
1	B	208	VAL	2.1
1	B	78	SER	2.1

6.2 Non-standard residues in protein, DNA, RNA chains [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. LLDF column lists the quality of electron density of the group with respect to its neighbouring residues in protein, DNA or RNA chains. The B-factors column lists the minimum, median, 95th 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	LLDF	B-factors(Å ²)	Q<0.9
1	PTR	B	216	16/17	0.96	0.13	-	55,57,64,64	0
1	PTR	A	216	16/17	0.97	0.11	-	44,46,48,48	0

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. LLDF column lists the quality of electron density of the group with respect to its neighbouring residues in protein, DNA or RNA chains. The B-factors column lists the minimum, median, 95th 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	LLDF	B-factors(Å ²)	Q<0.9
2	6QH	B	401	25/25	0.92	0.14	0.21	34,43,52,63	0
2	6QH	A	4001	25/25	0.94	0.12	-0.24	36,43,49,60	0
3	SO4	A	4002	5/5	0.98	0.11	-1.25	49,52,55,56	0
3	SO4	B	402	5/5	0.97	0.09	-1.42	54,54,55,57	0

6.5 Other polymers [i](#)

There are no such residues in this entry.