



Full wwPDB X-ray Structure Validation Report ⓘ

Oct 17, 2017 – 06:29 AM EDT

PDB ID : 3DU8
Title : Crystal structure of GSK-3 beta in complex with NMS-869553A
Authors : Bossi, R.T.
Deposited on : unknown
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 : 1.7.2 (RC1), CSD as538be (2017)
Xtriage (Phenix) : 1.9-1692
EDS : rb-20030345
Percentile statistics : 20161228.v01 (using entries in the PDB archive December 28th 2016)
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-20030345

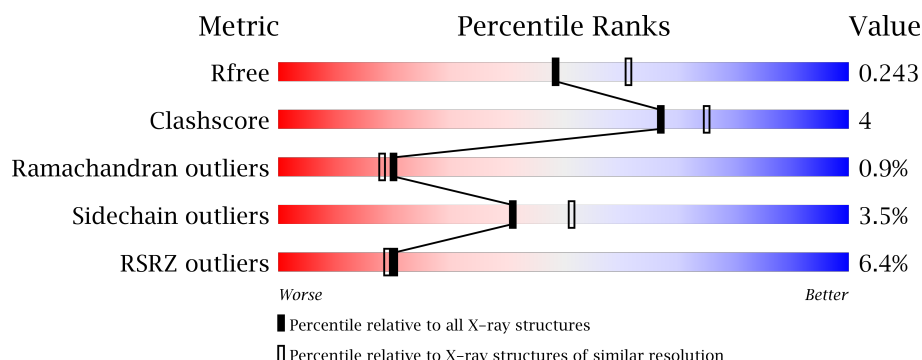
1 Overall quality at a glance

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}	100719	4002 (2.20-2.20)
Clashscore	112137	4730 (2.20-2.20)
Ramachandran outliers	110173	4656 (2.20-2.20)
Sidechain outliers	110143	4657 (2.20-2.20)
RSRZ outliers	101464	4033 (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	422	<div> <div>6%</div> <div>69%</div> <div>9%</div> <div>20%</div> </div>
1	B	422	<div> <div>4%</div> <div>70%</div> <div>9%</div> <div>20%</div> </div>

2 Entry composition [i](#)

There are 3 unique types of molecules in this entry. The entry contains 5644 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.

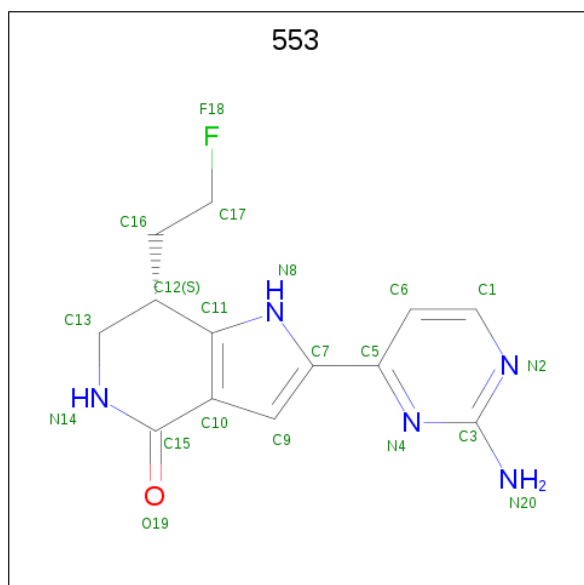
- Molecule 1 is a protein called Glycogen synthase kinase-3 beta.

Mol	Chain	Residues	Atoms					ZeroOcc	AltConf	Trace
1	A	336	Total	C	N	O	S	0	0	0
			2681	1727	460	483	11			
1	B	339	Total	C	N	O	S	0	0	0
			2709	1744	467	487	11			

There are 4 discrepancies between the modelled and reference sequences:

Chain	Residue	Modelled	Actual	Comment	Reference
A	-1	GLY	-	EXPRESSION TAG	UNP P49841
A	0	PRO	-	EXPRESSION TAG	UNP P49841
B	-1	GLY	-	EXPRESSION TAG	UNP P49841
B	0	PRO	-	EXPRESSION TAG	UNP P49841

- Molecule 2 is (7S)-2-(2-aminopyrimidin-4-yl)-7-(2-fluoroethyl)-1,5,6,7-tetrahydro-4H-pyrrolo[3,2-c]pyridin-4-one (three-letter code: 553) (formula: C₁₃H₁₄FN₅O).



Mol	Chain	Residues	Atoms					ZeroOcc	AltConf
2	A	1	Total	C	F	N	O	0	0
			20	13	1	5	1		
2	B	1	Total	C	F	N	O	0	0
			20	13	1	5	1		

- Molecule 3 is water.

Mol	Chain	Residues	Atoms		ZeroOcc	AltConf
3	A	113	Total	O	0	0
			113	113		
3	B	101	Total	O	0	0
			101	101		

4 Data and refinement statistics

Property	Value	Source
Space group	P 21 21 21	Depositor
Cell constants a, b, c, α , β , γ	82.60Å 85.81Å 178.04Å 90.00° 90.00° 90.00°	Depositor
Resolution (Å)	30.00 – 2.20 29.67 – 2.20	Depositor EDS
% Data completeness (in resolution range)	90.5 (30.00-2.20) 90.5 (29.67-2.20)	Depositor EDS
R_{merge}	0.08	Depositor
R_{sym}	(Not available)	Depositor
$\langle I/\sigma(I) \rangle$ ¹	2.52 (at 2.20Å)	Xtriage
Refinement program	REFMAC	Depositor
R, R_{free}	0.214 , 0.246 0.210 , 0.243	Depositor DCC
R_{free} test set	2973 reflections (5.32%)	DCC
Wilson B-factor (Å ²)	39.6	Xtriage
Anisotropy	0.597	Xtriage
Bulk solvent k_{sol} (e/Å ³), B_{sol} (Å ²)	0.38 , 48.4	EDS
L-test for twinning ²	$\langle L \rangle = 0.50$, $\langle L^2 \rangle = 0.34$	Xtriage
Estimated twinning fraction	0.015 for k,h,-l	Xtriage
F_o, F_c correlation	0.95	EDS
Total number of atoms	5644	wwPDB-VP
Average B, all atoms (Å ²)	48.0	wwPDB-VP

Xtriage's analysis on translational NCS is as follows: *The largest off-origin peak in the Patterson function is 3.24% 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 [i](#)

5.1 Standard geometry [i](#)

Bond lengths and bond angles in the following residue types are not validated in this section: 553

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	0.82	1/2748 (0.0%)	0.77	2/3740 (0.1%)
1	B	0.83	2/2776 (0.1%)	0.77	0/3777
All	All	0.83	3/5524 (0.1%)	0.77	2/7517 (0.0%)

All (3) bond length outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(Å)	Ideal(Å)
1	B	317	CYS	CB-SG	-6.16	1.71	1.82
1	A	317	CYS	CB-SG	-5.67	1.72	1.81
1	B	222	TYR	CE2-CZ	5.07	1.45	1.38

All (2) bond angle outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(°)	Ideal(°)
1	A	110	VAL	CB-CA-C	-5.65	100.66	111.40
1	A	328	ARG	NE-CZ-NH2	-5.29	117.66	120.30

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	2681	0	2712	22	1

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Mol	Chain	Non-H	H(model)	H(added)	Clashes	Symm-Clashes
1	B	2709	0	2744	28	0
2	A	20	0	14	3	0
2	B	20	0	14	1	0
3	A	113	0	0	1	1
3	B	101	0	0	4	0
All	All	5644	0	5484	49	1

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 4.

All (49) 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:283:GLU:HG2	1:A:324:THR:HG23	1.27	1.12
1:B:285:ASN:HB3	1:B:286:PRO:HD3	1.09	1.04
1:B:285:ASN:HB3	1:B:286:PRO:CD	1.90	1.01
1:B:285:ASN:CB	1:B:286:PRO:HD3	1.94	0.96
1:A:285:ASN:HB3	1:A:286:PRO:HD3	1.50	0.92
1:B:220:ARG:O	1:B:221:TYR:HB2	1.98	0.64
1:B:299:HIS:HE1	1:B:303:LYS:HD3	1.65	0.61
1:B:295:GLN:HB2	3:B:631:HOH:O	2.02	0.59
1:A:99:GLN:HG3	3:A:655:HOH:O	2.05	0.56
1:A:60:LYS:HE3	1:A:72:GLN:NE2	2.21	0.56
1:B:220:ARG:O	1:B:221:TYR:CB	2.56	0.53
1:A:107:CYS:O	1:A:197:LYS:NZ	2.42	0.52
1:B:271:LYS:O	1:B:299:HIS:HB2	2.09	0.52
1:A:220:ARG:CD	1:B:220:ARG:HD3	2.41	0.51
1:A:285:ASN:HB3	1:A:286:PRO:CD	2.31	0.51
1:A:217:ILE:O	1:A:223:ARG:NH1	2.45	0.50
1:B:183:LYS:HG3	1:B:185:GLN:HG2	1.93	0.49
1:B:183:LYS:HE3	1:B:185:GLN:HG3	1.95	0.48
1:A:186:ASN:OD1	2:A:553:553:H17A	2.13	0.48
1:B:153:LEU:HG	1:B:157:TYR:HB2	1.96	0.48
1:A:133:ASP:O	2:A:553:553:H1	2.14	0.47
1:B:162:MET:HG3	1:B:247:LEU:HD13	1.95	0.47
1:B:167:ARG:HD3	3:B:565:HOH:O	2.14	0.47
1:A:74:LYS:HE2	1:A:79:GLY:HA2	1.97	0.47
1:B:42:ALA:HB1	1:B:114:TYR:HB3	1.97	0.46
1:B:285:ASN:CB	1:B:286:PRO:CD	2.69	0.46
1:B:60:LYS:HE3	1:B:72:GLN:NE2	2.31	0.46
1:B:133:ASP:O	2:B:553:553:H1	2.17	0.45

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Atom-1	Atom-2	Interatomic distance (Å)	Clash overlap (Å)
1:B:217:ILE:O	1:B:223:ARG:NH1	2.49	0.45
2:A:553:553:H13A	2:A:553:553:H17	1.77	0.44
1:B:369:SER:CB	3:B:586:HOH:O	2.66	0.44
1:B:91:LYS:HA	1:B:91:LYS:HD3	1.76	0.44
1:B:349:LYS:HE2	1:B:355:ASP:OD1	2.19	0.43
1:A:220:ARG:O	1:A:221:TYR:HB2	2.19	0.43
1:A:310:PRO:HA	1:A:311:PRO:HD3	1.87	0.43
1:A:220:ARG:HD2	1:B:220:ARG:HD3	1.99	0.43
1:A:220:ARG:O	1:A:221:TYR:CB	2.67	0.43
1:A:313:ALA:HB2	1:A:339:PHE:CE1	2.54	0.42
1:B:217:ILE:O	1:B:218:CYS:HB3	2.19	0.42
1:B:381:HIS:HA	1:B:384:ILE:HD12	2.02	0.42
1:B:369:SER:HB3	3:B:586:HOH:O	2.18	0.42
1:A:100:ILE:O	1:A:104:LEU:HG	2.21	0.41
1:A:113:ARG:NE	1:A:133:ASP:OD1	2.48	0.41
1:A:369:SER:O	1:A:370:ASN:HB2	2.20	0.41
1:A:301:TRP:HB3	1:A:314:ILE:CG2	2.51	0.41
1:A:46:GLN:HE21	1:A:46:GLN:HB3	1.59	0.40
1:B:214:VAL:HB	1:B:216:TYR:CE2	2.57	0.40
1:B:323:TYR:O	1:B:325:PRO:HD3	2.21	0.40
1:A:180:ARG:HD2	1:A:217:ILE:HG12	2.03	0.40

All (1) symmetry-related close contacts are listed below. The label for Atom-2 includes the symmetry operator and encoded unit-cell translations to be applied.

Atom-1	Atom-2	Interatomic distance (Å)	Clash overlap (Å)
1:A:50:ARG:NH2	3:A:648:HOH:O[4_545]	1.84	0.36

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	330/422 (78%)	312 (94%)	15 (4%)	3 (1%)	20	18
1	B	333/422 (79%)	316 (95%)	14 (4%)	3 (1%)	20	18
All	All	663/844 (79%)	628 (95%)	29 (4%)	6 (1%)	20	18

All (6) Ramachandran outliers are listed below:

Mol	Chain	Res	Type
1	A	285	ASN
1	B	285	ASN
1	A	65	GLY
1	A	221	TYR
1	B	221	TYR
1	B	65	GLY

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	299/365 (82%)	288 (96%)	11 (4%)	39	49
1	B	302/365 (83%)	292 (97%)	10 (3%)	43	54
All	All	601/730 (82%)	580 (96%)	21 (4%)	41	51

All (21) residues with a non-rotameric sidechain are listed below:

Mol	Chain	Res	Type
1	A	46	GLN
1	A	64	ASN
1	A	66	SER
1	A	92	ARG
1	A	94	LYS
1	A	110	VAL
1	A	147	SER
1	A	151	GLN
1	A	152	THR
1	A	306	ARG

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Mol	Chain	Res	Type
1	A	349	LYS
1	B	35	SER
1	B	46	GLN
1	B	66	SER
1	B	94	LYS
1	B	110	VAL
1	B	185	GLN
1	B	306	ARG
1	B	349	LYS
1	B	370	ASN
1	B	383	ARG

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

Mol	Chain	Res	Type
1	A	46	GLN
1	A	72	GLN
1	A	299	HIS
1	A	365	GLN
1	B	46	GLN
1	B	64	ASN
1	B	72	GLN
1	B	99	GLN
1	B	108	ASN
1	B	299	HIS
1	B	365	GLN
1	B	370	ASN

5.3.3 RNA ⓘ

There are no RNA molecules in this entry.

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

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

5.5 Carbohydrates ⓘ

There are no carbohydrates in this entry.

5.6 Ligand geometry

2 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	553	A	553	-	19,22,22	2.17	2 (10%)	19,31,31	3.37	10 (52%)
2	553	B	553	-	19,22,22	2.18	2 (10%)	19,31,31	3.47	12 (63%)

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	553	A	553	-	-	0/4/20/20	0/3/3/3
2	553	B	553	-	-	0/4/20/20	0/3/3/3

All (4) bond length outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(Å)	Ideal(Å)
2	B	553	553	C11-C12	-3.17	1.48	1.52
2	A	553	553	C11-C12	-3.14	1.48	1.52
2	A	553	553	C10-C11	8.26	1.47	1.38
2	B	553	553	C10-C11	8.47	1.48	1.38

All (22) bond angle outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(°)	Ideal(°)
2	A	553	553	N2-C3-N4	-5.81	119.73	125.82
2	B	553	553	N2-C3-N4	-5.67	119.87	125.82
2	A	553	553	C16-C12-C11	-4.52	103.81	112.00
2	B	553	553	C6-C1-N2	-4.17	119.11	123.92
2	B	553	553	C16-C12-C11	-4.09	104.59	112.00
2	A	553	553	C6-C1-N2	-2.54	120.99	123.92
2	B	553	553	O19-C15-C10	-2.52	119.06	123.28

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Mol	Chain	Res	Type	Atoms	Z	Observed(°)	Ideal(°)
2	A	553	553	O19-C15-C10	-2.37	119.31	123.28
2	A	553	553	C10-C9-C7	-2.29	102.94	105.98
2	B	553	553	C10-C9-C7	-2.21	103.05	105.98
2	B	553	553	C10-C11-N8	-2.16	106.62	110.15
2	B	553	553	C7-C5-N4	2.14	118.98	116.29
2	B	553	553	C9-C7-C5	2.17	132.46	129.30
2	A	553	553	C7-C5-N4	3.36	120.51	116.29
2	A	553	553	C9-C10-C11	3.39	108.59	104.10
2	B	553	553	C9-C10-C11	3.46	108.69	104.10
2	A	553	553	N20-C3-N2	4.23	121.45	117.39
2	A	553	553	C1-N2-C3	4.51	121.06	116.33
2	B	553	553	C1-N2-C3	5.22	121.80	116.33
2	B	553	553	N20-C3-N2	5.34	122.52	117.39
2	B	553	553	C5-N4-C3	8.11	120.75	116.36
2	A	553	553	C5-N4-C3	8.18	120.79	116.36

There are no chirality outliers.

There are no torsion outliers.

There are no ring outliers.

2 monomers are involved in 4 short contacts:

Mol	Chain	Res	Type	Clashes	Symm-Clashes
2	A	553	553	3	0
2	B	553	553	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

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	336/422 (79%)	0.19	25 (7%) 15 14	27, 43, 76, 99	0
1	B	339/422 (80%)	0.12	18 (5%) 27 26	28, 46, 75, 96	0
All	All	675/844 (79%)	0.16	43 (6%) 20 19	27, 44, 75, 99	0

All (43) RSRZ outliers are listed below:

Mol	Chain	Res	Type	RSRZ
1	A	93	PHE	6.5
1	A	35	SER	6.0
1	B	35	SER	5.0
1	B	92	ARG	4.8
1	A	92	ARG	4.8
1	B	298	ALA	4.7
1	B	93	PHE	4.6
1	B	297	LYS	4.0
1	A	278	ARG	3.9
1	A	300	PRO	3.9
1	B	300	PRO	3.9
1	B	295	GLN	3.9
1	B	91	LYS	3.8
1	B	299	HIS	3.7
1	B	148	ARG	3.5
1	B	278	ARG	3.2
1	B	119	SER	3.2
1	A	299	HIS	3.1
1	A	297	LYS	3.1
1	A	279	GLU	3.1
1	A	47	GLY	3.1
1	A	91	LYS	3.0
1	A	282	ARG	2.9
1	A	150	LYS	2.9

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Mol	Chain	Res	Type	RSRZ
1	A	285	ASN	2.8
1	A	286	PRO	2.8
1	B	36	LYS	2.8
1	A	283	GLU	2.7
1	B	385	GLN	2.7
1	B	48	PRO	2.6
1	A	302	THR	2.5
1	A	165	LEU	2.5
1	A	295	GLN	2.4
1	A	208	VAL	2.4
1	B	127	TYR	2.3
1	B	279	GLU	2.3
1	A	298	ALA	2.3
1	A	148	ARG	2.2
1	B	282	ARG	2.1
1	A	36	LYS	2.1
1	A	320	LEU	2.1
1	A	198	LEU	2.0
1	A	50	ARG	2.0

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. 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	553	B	553	20/20	0.95	0.12	-0.48	42,46,53,55	0
2	553	A	553	20/20	0.95	0.09	-1.40	33,40,57,60	0

6.5 Other polymers [i](#)

There are no such residues in this entry.