



Full wwPDB X-ray Structure Validation Report ⓘ

Feb 21, 2017 – 04:52 pm GMT

PDB ID : 5UBT
Title : CRYSTAL STRUCTURE OF PI3K DELTA IN COMPLEX WITH A 7-(3-(PIPERAZIN-1-YL)PHENYL)PYRROLO[2,1-F][1,2,4] TRIAZIN-4-AMINE DERIVATIVE
Authors : Sack, J.S.
Deposited on : 2016-12-21
Resolution : 2.83 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

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	:	recalc29047
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)	:	recalc29047

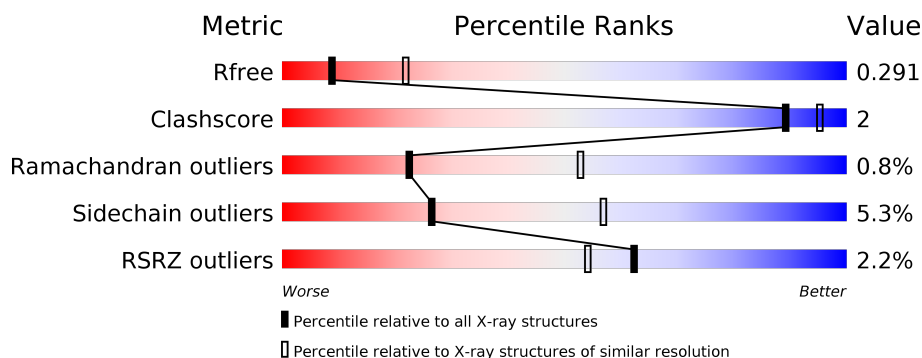
1 Overall quality at a glance

The following experimental techniques were used to determine the structure:

X-RAY DIFFRACTION

The reported resolution of this entry is 2.83 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}	100719	3466 (2.88-2.80)
Clashscore	112137	3975 (2.88-2.80)
Ramachandran outliers	110173	3902 (2.88-2.80)
Sidechain outliers	110143	3905 (2.88-2.80)
RSRZ outliers	101464	3501 (2.88-2.80)

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 ≥ 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	1013	<div> <div>2%</div> <div> <div></div> <div>81%</div> <div>9%</div> <div>10%</div> </div> </div>
2	B	168	<div> <div>%</div> <div> <div></div> <div>88%</div> <div>8%</div> <div></div> </div> </div>

2 Entry composition

There are 4 unique types of molecules in this entry. The entry contains 8531 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 Phosphatidylinositol 4,5-bisphosphate 3-kinase catalytic subunit delta isoform.

Mol	Chain	Residues	Atoms					ZeroOcc	AltConf	Trace
1	A	911	Total	C	N	O	S	319	0	0
			7106	4546	1207	1301	52			

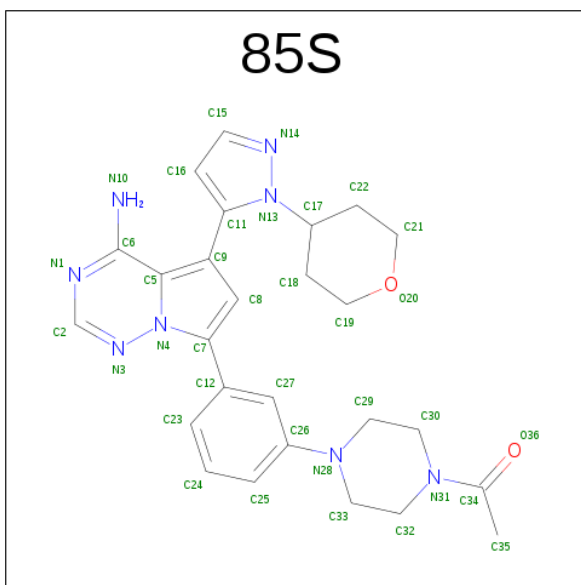
- Molecule 2 is a protein called Phosphatidylinositol 3-kinase regulatory subunit alpha.

Mol	Chain	Residues	Atoms					ZeroOcc	AltConf	Trace
2	B	163	Total	C	N	O	S	113	0	0
			1376	857	248	266	5			

There are 4 discrepancies between the modelled and reference sequences:

Chain	Residue	Modelled	Actual	Comment	Reference
B	469	ASP	GLU	engineered mutation	UNP P27986
B	519	THR	LYS	engineered mutation	UNP P27986
B	529	GLU	ASP	engineered mutation	UNP P27986
B	539	VAL	ILE	engineered mutation	UNP P27986

- Molecule 3 is 1-[4-(3-{4-amino-5-[1-(oxan-4-yl)-1H-pyrazol-5-yl]pyrrolo[2,1-f][1,2,4]triazin-7-yl}phenyl)piperazin-1-yl]ethan-1-one (three-letter code: 85S) (formula: C₂₆H₃₀N₈O₂).



Mol	Chain	Residues	Atoms				ZeroOcc	AltConf
3	A	1	Total	C	N	O	0	0
			36	26	8	2		

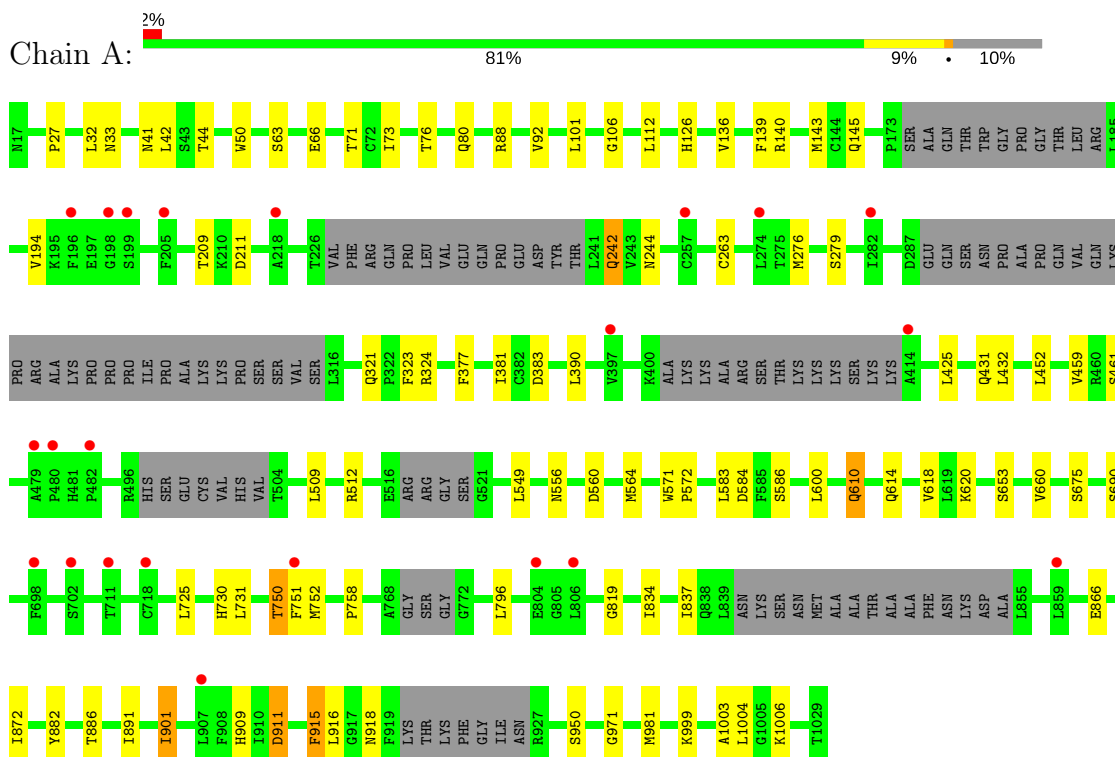
- Molecule 4 is water.

Mol	Chain	Residues	Atoms		ZeroOcc	AltConf
4	A	13	Total	O	0	0
			13	13		

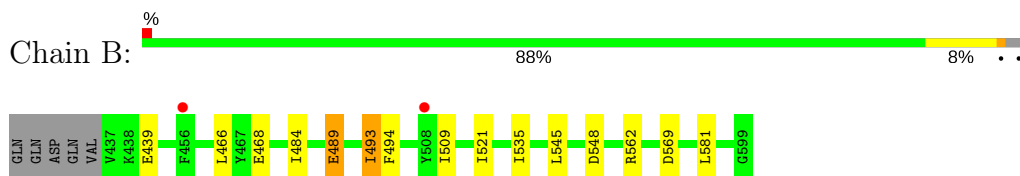
3 Residue-property plots

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: Phosphatidylinositol 4,5-bisphosphate 3-kinase catalytic subunit delta isoform



- Molecule 2: Phosphatidylinositol 3-kinase regulatory subunit alpha



4 Data and refinement statistics

Property	Value	Source
Space group	P 21 21 21	Depositor
Cell constants a, b, c, α , β , γ	90.66Å 108.60Å 142.90Å 90.00° 90.00° 90.00°	Depositor
Resolution (Å)	37.86 – 2.83 36.10 – 2.83	Depositor EDS
% Data completeness (in resolution range)	97.1 (37.86-2.83) 97.1 (36.10-2.83)	Depositor EDS
R_{merge}	0.03	Depositor
R_{sym}	(Not available)	Depositor
$\langle I/\sigma(I) \rangle$ ¹	1.75 (at 2.81Å)	Xtriage
Refinement program	BUSTER 2.11.7	Depositor
R, R_{free}	0.215 , 0.268 0.233 , 0.291	Depositor DCC
R_{free} test set	688 reflections (2.11%)	DCC
Wilson B-factor (Å ²)	91.1	Xtriage
Anisotropy	0.478	Xtriage
Bulk solvent k_{sol} (e/Å ³), B_{sol} (Å ²)	0.28 , 82.4	EDS
L-test for twinning ²	$\langle L \rangle = 0.48$, $\langle L^2 \rangle = 0.32$	Xtriage
Estimated twinning fraction	No twinning to report.	Xtriage
F_o, F_c correlation	0.94	EDS
Total number of atoms	8531	wwPDB-VP
Average B, all atoms (Å ²)	104.0	wwPDB-VP

Xtriage's analysis on translational NCS is as follows: *The largest off-origin peak in the Patterson function is 3.33% 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: 85S

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	0/7257	0.62	0/9836
2	B	0.43	0/1394	0.59	0/1862
All	All	0.44	0/8651	0.62	0/11698

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	7106	0	6897	30	0
2	B	1376	0	1356	6	0
3	A	36	0	0	0	0
4	A	13	0	0	0	0
All	All	8531	0	8253	34	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 (34) 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)
2:B:484:ILE:HG13	2:B:545:LEU:HD23	1.77	0.65
2:B:466:LEU:HD21	2:B:562:ARG:HB3	1.82	0.61
1:A:549:LEU:HG	1:A:564:MET:HE3	1.82	0.61
1:A:549:LEU:HG	1:A:564:MET:CE	2.32	0.59
1:A:76:THR:O	1:A:126:HIS:HE1	1.87	0.58
1:A:390:LEU:HB2	1:A:425:LEU:HD21	1.89	0.55
1:A:583:LEU:HD11	1:A:600:LEU:HD11	1.89	0.54
1:A:999:LYS:HA	1:A:1004:LEU:HD12	1.88	0.54
1:A:610:GLN:HG2	1:A:796:LEU:HD13	1.91	0.52
1:A:571:TRP:CD1	1:A:572:PRO:HD2	2.46	0.51
1:A:614:GLN:HG3	1:A:981:MET:HG2	1.96	0.47
1:A:653:SER:HA	1:A:819:GLY:HA2	1.96	0.47
1:A:27:PRO:HD3	1:A:101:LEU:HB2	1.97	0.46
1:A:73:ILE:HD12	2:B:489:GLU:HG3	1.97	0.46
1:A:752:MET:HB2	1:A:758:PRO:HD2	1.98	0.46
1:A:971:GLY:HA3	1:A:1004:LEU:HD21	1.98	0.45
1:A:882:TYR:HB3	1:A:909:HIS:CD2	2.52	0.45
1:A:139:PHE:CZ	1:A:143:MET:HG3	2.52	0.45
1:A:323:PHE:HD2	1:A:377:PHE:CD2	2.35	0.45
1:A:321:GLN:HB3	1:A:324:ARG:HH12	1.81	0.44
1:A:1003:ALA:HB1	1:A:1006:LYS:HD2	1.97	0.44
1:A:42:LEU:HD11	1:A:92:VAL:HG21	2.00	0.44
2:B:509:ILE:HG23	2:B:521:ILE:HG12	2.00	0.44
1:A:41:ASN:HA	1:A:88:ARG:HA	2.00	0.43
1:A:583:LEU:HB3	1:A:618:VAL:HG11	2.01	0.43
1:A:73:ILE:HD11	2:B:493:ILE:HD12	2.00	0.43
1:A:750:THR:HG22	1:A:751:PHE:H	1.83	0.43
2:B:494:PHE:HB3	2:B:535:ILE:HG12	2.00	0.43
1:A:886:THR:HA	1:A:891:ILE:HD12	2.01	0.42
1:A:620:LYS:HE2	1:A:660:VAL:HG11	2.00	0.42
1:A:383:ASP:HB3	1:A:556:ASN:O	2.19	0.41
1:A:76:THR:O	1:A:126:HIS:CE1	2.69	0.41
1:A:136:VAL:O	1:A:140:ARG:HG3	2.20	0.41
1:A:834:ILE:HD11	1:A:901:ILE:HG23	2.03	0.41

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	891/1013 (88%)	841 (94%)	42 (5%)	8 (1%)	20	49
2	B	161/168 (96%)	159 (99%)	2 (1%)	0	100	100
All	All	1052/1181 (89%)	1000 (95%)	44 (4%)	8 (1%)	22	53

All (8) Ramachandran outliers are listed below:

Mol	Chain	Res	Type
1	A	242	GLN
1	A	279	SER
1	A	911	ASP
1	A	918	ASN
1	A	730	HIS
1	A	915	PHE
1	A	381	ILE
1	A	106	GLY

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	753/898 (84%)	712 (95%)	41 (5%)	26	56
2	B	147/159 (92%)	140 (95%)	7 (5%)	30	61
All	All	900/1057 (85%)	852 (95%)	48 (5%)	26	57

All (48) residues with a non-rotameric sidechain are listed below:

Mol	Chain	Res	Type
1	A	32	LEU
1	A	33	ASN
1	A	44	THR
1	A	50	TRP
1	A	63	SER
1	A	66	GLU
1	A	71	THR
1	A	80	GLN
1	A	112	LEU
1	A	145	GLN
1	A	194	VAL
1	A	209	THR
1	A	211	ASP
1	A	242	GLN
1	A	244	ASN
1	A	263	CYS
1	A	276	MET
1	A	431	GLN
1	A	432	LEU
1	A	452	LEU
1	A	459	VAL
1	A	461	SER
1	A	509	LEU
1	A	512	ARG
1	A	560	ASP
1	A	584	ASP
1	A	586	SER
1	A	610	GLN
1	A	675	SER
1	A	690	SER
1	A	725	LEU
1	A	731	LEU
1	A	750	THR
1	A	837	ILE
1	A	866	GLU
1	A	872	ILE
1	A	901	ILE
1	A	911	ASP
1	A	915	PHE
1	A	916	LEU
1	A	950	SER
2	B	439	GLU
2	B	468	GLU

Continued on next page...

Continued from previous page...

Mol	Chain	Res	Type
2	B	489	GLU
2	B	493	ILE
2	B	548	ASP
2	B	569	ASP
2	B	581	LEU

Some sidechains can be flipped to improve hydrogen bonding and reduce clashes. All (1) such sidechains are listed below:

Mol	Chain	Res	Type
1	A	610	GLN

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 [i](#)

1 ligand is 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	$\# Z > 2$
3	85S	A	4000	-	37,41,41	0.58	0	39,59,59	0.63	1 (2%)

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
3	85S	A	4000	-	-	0/12/38/38	0/6/6/6

There are no bond length outliers.

All (1) bond angle outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(°)	Ideal(°)
3	A	4000	85S	C9-C11-N13	2.01	126.22	123.38

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	911/1013 (89%)	0.03	22 (2%) 59 50	64, 101, 143, 176	87 (9%)
2	B	163/168 (97%)	-0.13	2 (1%) 79 73	78, 103, 128, 142	33 (20%)
All	All	1074/1181 (90%)	0.00	24 (2%) 62 54	64, 101, 142, 176	120 (11%)

All (24) RSRZ outliers are listed below:

Mol	Chain	Res	Type	RSRZ
1	A	711	THR	3.6
1	A	205	PHE	3.6
1	A	718	CYS	3.5
1	A	480	PRO	3.5
1	A	479	ALA	3.4
2	B	508	TYR	3.4
1	A	282	ILE	3.3
1	A	859	LEU	3.0
1	A	198	GLY	3.0
1	A	907	LEU	3.0
1	A	414	ALA	2.7
2	B	456	PHE	2.6
1	A	698	PHE	2.5
1	A	751	PHE	2.5
1	A	199	SER	2.4
1	A	274	LEU	2.4
1	A	482	PRO	2.3
1	A	806	LEU	2.3
1	A	196	PHE	2.3
1	A	804	GLU	2.2
1	A	702	SER	2.1
1	A	257	CYS	2.1
1	A	397	VAL	2.1
1	A	218	ALA	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(\AA^2)	Q<0.9
3	85S	A	4000	36/36	0.90	0.23	0.36	91,103,131,132	0

6.5 Other polymers [i](#)

There are no such residues in this entry.