



Full wwPDB X-ray Structure Validation Report ⓘ

Oct 16, 2017 – 09:07 AM EDT

PDB ID : 3EDQ
Title : Crystal structure of Caspase-3 with inhibitor AC-LDESD-CHO
Authors : Fu, G.
Deposited on : unknown
Resolution : 1.61 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

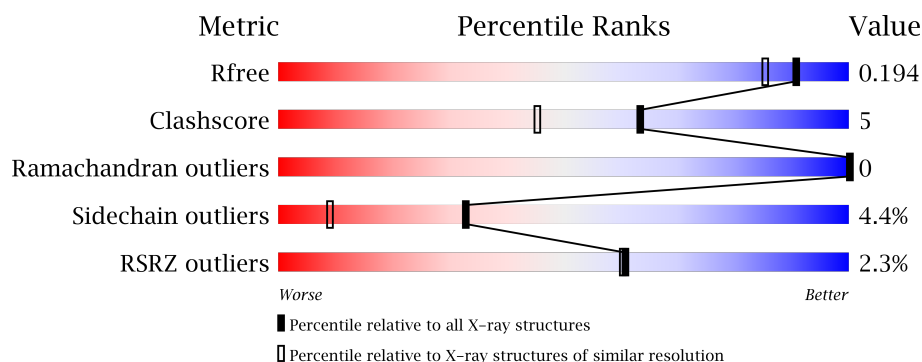
1 Overall quality at a glance

The following experimental techniques were used to determine the structure:

X-RAY DIFFRACTION

The reported resolution of this entry is 1.61 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	3539 (1.64-1.60)
Clashscore	112137	3855 (1.64-1.60)
Ramachandran outliers	110173	3764 (1.64-1.60)
Sidechain outliers	110143	3763 (1.64-1.60)
RSRZ outliers	101464	3562 (1.64-1.60)

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	147	<div> <div style="width: 100%; height: 10px; background-color: red;"></div> <div style="display: flex; justify-content: space-between;"> 0% 80% 13% 5% </div> </div>
1	C	147	<div> <div style="width: 100%; height: 10px; background-color: red;"></div> <div style="display: flex; justify-content: space-between;"> 4% 81% 11% . </div> </div>
2	B	108	<div> <div style="width: 100%; height: 10px; background-color: red;"></div> <div style="display: flex; justify-content: space-between;"> 2% 73% 10% . </div> </div>
2	D	108	<div> <div style="width: 100%; height: 10px; background-color: red;"></div> <div style="display: flex; justify-content: space-between;"> 0% 76% 9% . </div> </div>
3	E	6	<div> <div style="width: 100%; height: 10px; background-color: red;"></div> <div style="display: flex; justify-content: space-between;"> 100% </div> </div>

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Mol	Chain	Length	Quality of chain
3	F	6	 67% 33%

2 Entry composition

There are 4 unique types of molecules in this entry. The entry contains 4159 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 Caspase-3.

Mol	Chain	Residues	Atoms					ZeroOcc	AltConf	Trace
1	A	140	Total	C	N	O	S	0	1	0
			1118	692	201	216	9			
1	C	141	Total	C	N	O	S	0	1	0
			1127	697	204	217	9			

- Molecule 2 is a protein called Caspase-3.

Mol	Chain	Residues	Atoms					ZeroOcc	AltConf	Trace
2	B	93	Total	C	N	O	S	0	0	0
			777	511	125	136	5			
2	D	93	Total	C	N	O	S	0	0	0
			777	511	125	136	5			

There are 12 discrepancies between the modelled and reference sequences:

Chain	Residue	Modelled	Actual	Comment	Reference
B	278	HIS	-	EXPRESSION TAG	UNP P42574
B	279	HIS	-	EXPRESSION TAG	UNP P42574
B	280	HIS	-	EXPRESSION TAG	UNP P42574
B	281	HIS	-	EXPRESSION TAG	UNP P42574
B	282	HIS	-	EXPRESSION TAG	UNP P42574
B	283	HIS	-	EXPRESSION TAG	UNP P42574
D	278	HIS	-	EXPRESSION TAG	UNP P42574
D	279	HIS	-	EXPRESSION TAG	UNP P42574
D	280	HIS	-	EXPRESSION TAG	UNP P42574
D	281	HIS	-	EXPRESSION TAG	UNP P42574
D	282	HIS	-	EXPRESSION TAG	UNP P42574
D	283	HIS	-	EXPRESSION TAG	UNP P42574

- Molecule 3 is a protein called AC-LDESD-CHO peptide.

Mol	Chain	Residues	Atoms				ZeroOcc	AltConf	Trace
3	E	6	Total	C	N	O	0	0	0
			42	24	5	13			
3	F	6	Total	C	N	O	0	0	0
			42	24	5	13			

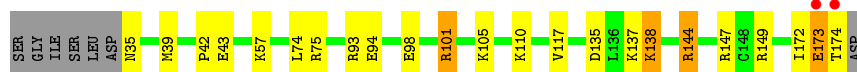
- Molecule 4 is water.

Mol	Chain	Residues	Atoms		ZeroOcc	AltConf
4	A	93	Total	O	0	0
			93	93		
4	B	44	Total	O	0	0
			44	44		
4	C	81	Total	O	0	0
			81	81		
4	D	48	Total	O	0	0
			48	48		
4	E	6	Total	O	0	0
			6	6		
4	F	4	Total	O	0	0
			4	4		

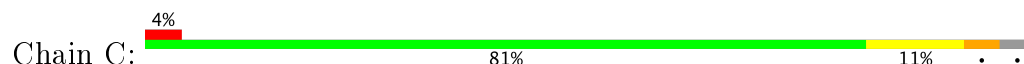
3 Residue-property plots [i](#)

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.

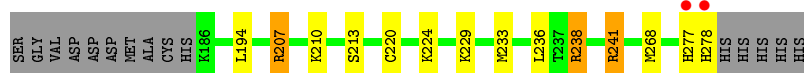
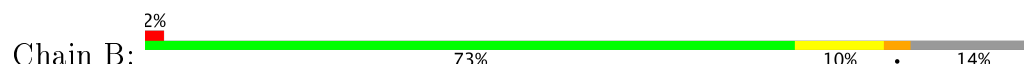
- Molecule 1: Caspase-3



- Molecule 1: Caspase-3



- Molecule 2: Caspase-3



- Molecule 2: Caspase-3



- Molecule 3: AC-LDES-CHO peptide



There are no outlier residues recorded for this chain.

- Molecule 3: AC-LDES-CHO peptide





4 Data and refinement statistics

Property	Value	Source
Space group	P 21 21 21	Depositor
Cell constants a, b, c, α , β , γ	67.38Å 93.56Å 97.62Å 90.00° 90.00° 90.00°	Depositor
Resolution (Å)	10.00 – 1.61 47.70 – 1.61	Depositor EDS
% Data completeness (in resolution range)	90.2 (10.00-1.61) 84.5 (47.70-1.61)	Depositor EDS
R_{merge}	0.07	Depositor
R_{sym}	(Not available)	Depositor
$\langle I/\sigma(I) \rangle$ ¹	3.07 (at 1.61Å)	Xtriage
Refinement program	SHELXL-97	Depositor
R, R_{free}	0.177 , 0.223 0.148 , 0.194	Depositor DCC
R_{free} test set	3626 reflections (5.61%)	DCC
Wilson B-factor (Å ²)	23.2	Xtriage
Anisotropy	0.661	Xtriage
Bulk solvent k_{sol} (e/Å ³), B_{sol} (Å ²)	0.40 , 57.0	EDS
L-test for twinning ²	$\langle L \rangle = 0.48$, $\langle L^2 \rangle = 0.31$	Xtriage
Estimated twinning fraction	0.000 for -h,l,k	Xtriage
F_o, F_c correlation	0.98	EDS
Total number of atoms	4159	wwPDB-VP
Average B, all atoms (Å ²)	32.0	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 45.27 % 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 1.3539e-04. The detected translational NCS is most likely also responsible for the elevated intensity ratio.*

¹ Intensities estimated from amplitudes.

² 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: ASA, ACE

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	0.50	0/1136	1.33	15/1521 (1.0%)
1	C	0.47	0/1146	1.24	3/1535 (0.2%)
2	B	0.60	0/802	1.25	4/1083 (0.4%)
2	D	0.53	0/802	1.30	5/1083 (0.5%)
3	E	0.57	0/31	0.98	0/41
3	F	0.52	0/31	1.30	0/41
All	All	0.52	0/3948	1.28	27/5304 (0.5%)

There are no bond length outliers.

All (27) bond angle outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(°)	Ideal(°)
2	D	241	ARG	NE-CZ-NH1	10.16	125.38	120.30
1	A	147	ARG	NE-CZ-NH2	10.15	125.37	120.30
1	A	144	ARG	NE-CZ-NH2	9.82	125.21	120.30
2	B	241	ARG	NE-CZ-NH1	9.73	125.17	120.30
1	A	144	ARG	NE-CZ-NH1	-9.50	115.55	120.30
1	A	75	ARG	NE-CZ-NH1	9.30	124.95	120.30
2	B	238	ARG	NE-CZ-NH2	-9.08	115.76	120.30
1	C	144	ARG	NE-CZ-NH1	-8.43	116.08	120.30
1	A	75	ARG	CD-NE-CZ	7.80	134.52	123.60
2	D	241	ARG	NE-CZ-NH2	-7.72	116.44	120.30
1	C	75	ARG	NE-CZ-NH1	-7.28	116.66	120.30
1	A	149	ARG	NE-CZ-NH2	7.17	123.89	120.30
1	A	147	ARG	NE-CZ-NH1	-7.01	116.79	120.30
2	D	253	ASP	CB-CG-OD1	6.55	124.19	118.30
2	D	194	LEU	CA-CB-CG	6.50	130.26	115.30
1	A	147	ARG	CD-NE-CZ	5.85	131.79	123.60
1	A	75	ARG	NE-CZ-NH2	-5.84	117.38	120.30
1	A	93	ARG	NE-CZ-NH2	-5.50	117.55	120.30

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Mol	Chain	Res	Type	Atoms	Z	Observed(°)	Ideal(°)
2	B	207	ARG	NE-CZ-NH1	-5.33	117.64	120.30
1	A	135	ASP	CB-CG-OD1	5.22	123.00	118.30
1	A	43	GLU	OE1-CD-OE2	-5.17	117.10	123.30
1	C	147	ARG	CD-NE-CZ	5.14	130.79	123.60
1	A	93	ARG	NE-CZ-NH1	5.09	122.84	120.30
1	A	101	ARG	NE-CZ-NH1	5.09	122.85	120.30
1	A	94	GLU	OE1-CD-OE2	-5.05	117.24	123.30
2	B	194	LEU	CA-CB-CG	5.03	126.87	115.30
2	D	204	TYR	CB-CG-CD1	5.02	124.01	121.00

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	1118	0	1115	12	0
1	C	1127	0	1120	18	0
2	B	777	0	754	8	0
2	D	777	0	754	2	0
3	E	42	0	32	0	0
3	F	42	0	32	1	0
4	A	93	0	0	4	0
4	B	44	0	0	1	0
4	C	81	0	0	2	0
4	D	48	0	0	1	0
4	E	6	0	0	0	0
4	F	4	0	0	0	0
All	All	4159	0	3807	38	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 (38) 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:277:HIS:O	2:B:278:HIS:HB2	1.50	1.06
1:C:42:PRO:HG2	1:C:110:LYS:HD3	1.57	0.86
1:A:42:PRO:HG2	1:A:110:LYS:HD3	1.69	0.75
1:A:39:MET:H	2:B:277:HIS:CE1	2.11	0.68
1:C:93:ARG:HB2	1:C:134:VAL:HG22	1.74	0.68
2:B:220:CYS:O	2:B:224:LYS:HG3	1.96	0.66
2:B:207:ARG:HA	2:B:213:SER:HA	1.83	0.59
2:B:233:MET:HE1	2:B:236:LEU:HD12	1.90	0.54
1:C:42:PRO:HG2	1:C:110:LYS:HB3	1.91	0.53
2:D:207:ARG:HA	2:D:213:SER:HA	1.91	0.52
1:C:56:HIS:HB3	1:C:59:THR:HG23	1.92	0.51
1:A:101:ARG:O	1:A:105:LYS:HG3	2.10	0.51
1:A:138:LYS:NZ	1:A:138:LYS:HB2	2.26	0.51
1:C:74:LEU:HD13	1:C:117:VAL:HG11	1.94	0.49
2:B:277:HIS:O	2:B:278:HIS:CB	2.37	0.48
1:A:173:GLU:HG3	1:A:173:GLU:H	1.36	0.48
1:A:137:LYS:HE2	4:D:324:HOH:O	2.13	0.48
1:A:35:ASN:N	4:A:241:HOH:O	2.48	0.47
1:A:172:ILE:HG23	1:A:172:ILE:O	2.16	0.46
1:C:59:THR:HB	1:C:61:MET:SD	2.56	0.45
1:C:144:ARG:HD2	4:C:236:HOH:O	2.15	0.45
1:C:34:ASP:HA	4:C:226:HOH:O	2.17	0.45
2:B:241:ARG:NH2	1:C:35:ASN:OD1	2.49	0.44
1:A:74:LEU:HD13	1:A:117:VAL:HG11	1.99	0.43
1:A:144:ARG:HD2	1:A:144:ARG:HH11	1.48	0.43
1:C:57:LYS:HD3	1:C:57:LYS:HA	1.24	0.43
1:C:51:ASN:HD22	1:C:89:ASN:HD22	1.67	0.43
1:A:57:LYS:HG3	4:A:230:HOH:O	2.19	0.43
1:A:138:LYS:NZ	4:A:228:HOH:O	2.52	0.42
3:F:1:ACE:H1	3:F:2:LEU:HA	1.82	0.42
4:A:241:HOH:O	2:D:238:ARG:NH2	2.53	0.42
4:B:308:HOH:O	1:C:35:ASN:HB3	2.20	0.42
2:B:238:ARG:HH22	1:C:35:ASN:ND2	2.17	0.42
1:C:173:GLU:HG3	1:C:173:GLU:H	1.47	0.41
1:C:82:LYS:HD3	1:C:82:LYS:HA	1.97	0.41
1:C:51:ASN:HD22	1:C:89:ASN:ND2	2.19	0.41
1:C:56:HIS:ND1	1:C:57:LYS:N	2.69	0.40
1:C:138:LYS:HZ3	1:C:138:LYS:HG2	1.76	0.40

There are no symmetry-related clashes.

5.3 Torsion angles

5.3.1 Protein backbone

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	139/147 (95%)	138 (99%)	1 (1%)	0	100	100
1	C	140/147 (95%)	136 (97%)	4 (3%)	0	100	100
2	B	91/108 (84%)	89 (98%)	2 (2%)	0	100	100
2	D	91/108 (84%)	90 (99%)	1 (1%)	0	100	100
3	E	4/6 (67%)	4 (100%)	0	0	100	100
3	F	4/6 (67%)	4 (100%)	0	0	100	100
All	All	469/522 (90%)	461 (98%)	8 (2%)	0	100	100

There are no Ramachandran outliers to report.

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	127/132 (96%)	123 (97%)	4 (3%)	45	17
1	C	128/132 (97%)	121 (94%)	7 (6%)	25	5
2	B	83/96 (86%)	80 (96%)	3 (4%)	40	13
2	D	83/96 (86%)	78 (94%)	5 (6%)	22	4
3	E	4/4 (100%)	4 (100%)	0	100	100
3	F	4/4 (100%)	4 (100%)	0	100	100
All	All	429/464 (92%)	410 (96%)	19 (4%)	33	9

All (19) residues with a non-rotameric sidechain are listed below:

Mol	Chain	Res	Type
1	A	98	GLU
1	A	138	LYS
1	A	173	GLU
1	A	174	THR
2	B	210	LYS
2	B	229	LYS
2	B	268	MET
1	C	56	HIS
1	C	57	LYS
1	C	62	THR
1	C	80	ASN
1	C	93	ARG
1	C	138	LYS
1	C	173	GLU
2	D	194	LEU
2	D	229	LYS
2	D	268	MET
2	D	273	LEU
2	D	277	HIS

Some sidechains can be flipped to improve hydrogen bonding and reduce clashes. All (3) such sidechains are listed below:

Mol	Chain	Res	Type
1	A	89	ASN
2	B	277	HIS
1	C	89	ASN

5.3.3 RNA [i](#)

There are no RNA molecules in this entry.

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

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	$\# Z > 2$
3	ASA	E	6	1,3	4,7,7	1.26	0	3,8,8	2.77	1 (33%)
3	ASA	F	6	1,3	4,7,7	1.18	0	3,8,8	2.59	1 (33%)

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	ASA	E	6	1,3	-	0/2/6/6	0/0/0/0
3	ASA	F	6	1,3	-	0/2/6/6	0/0/0/0

There are no bond length outliers.

All (2) bond angle outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed($^{\circ}$)	Ideal($^{\circ}$)
3	E	6	ASA	CB-CA-C	-4.55	102.64	111.41
3	F	6	ASA	CB-CA-C	-4.07	103.56	111.41

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](#)

There are no ligands in this entry.

5.7 Other polymers [i](#)

There are no such residues in this entry.

5.8 Polymer linkage issues

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, 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	140/147 (95%)	-0.29	2 (1%) 75 76	20, 30, 42, 70	0
1	C	141/147 (95%)	-0.13	6 (4%) 36 33	22, 33, 64, 89	0
2	B	93/108 (86%)	-0.02	2 (2%) 62 62	18, 25, 41, 100	0
2	D	93/108 (86%)	-0.32	1 (1%) 80 81	18, 26, 42, 82	0
3	E	4/6 (66%)	-0.61	0 100 100	30, 33, 34, 45	0
3	F	4/6 (66%)	-0.39	0 100 100	34, 38, 41, 47	0
All	All	475/522 (90%)	-0.20	11 (2%) 61 60	18, 29, 47, 100	0

All (11) RSRZ outliers are listed below:

Mol	Chain	Res	Type	RSRZ
1	A	174	THR	6.3
1	C	174	THR	5.6
1	C	57	LYS	5.3
2	B	278	HIS	4.3
2	B	277	HIS	4.0
1	C	58	SER	3.2
1	C	56	HIS	3.1
2	D	278	HIS	2.8
1	C	62	THR	2.5
1	C	60	GLY	2.2
1	A	173	GLU	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(\AA^2)	Q<0.9
3	ASA	F	6	8/8	0.97	0.07	-	29,30,34,34	0
3	ASA	E	6	8/8	0.97	0.06	-	25,27,29,29	0

6.3 Carbohydrates [i](#)

There are no carbohydrates in this entry.

6.4 Ligands [i](#)

There are no ligands in this entry.

6.5 Other polymers [i](#)

There are no such residues in this entry.