



# Full wwPDB X-ray Structure Validation Report ⓘ

Feb 1, 2016 – 09:18 AM GMT

PDB ID : 3HY9  
Title : Crystal Structure of the Catalytic Domain of ADAMTS-5 in Complex with an Amino-2-indanol compound  
Authors : Shieh, H.-S.; Williams, J.M.; Caspers, N.; Mathis, K.J.; Tortorella, M.D.; Tomasselli, A.  
Deposited on : 2009-06-22  
Resolution : 2.02 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](mailto: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.

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

MolProbity : 4.02b-467  
Mogul : 1.7 (RC4), CSD as536be (2015)  
Xtriage (Phenix) : 1.9-1692  
EDS : rb-20026688  
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) : trunk26865

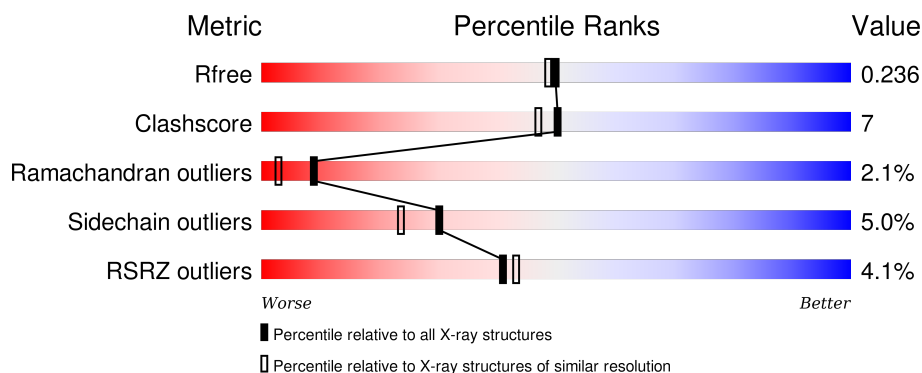
# 1 Overall quality at a glance

The following experimental techniques were used to determine the structure:

## *X-RAY DIFFRACTION*

The reported resolution of this entry is 2.02 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	7858 (2.04-2.00)
Clashscore	102246	9060 (2.04-2.00)
Ramachandran outliers	100387	8952 (2.04-2.00)
Sidechain outliers	100360	8951 (2.04-2.00)
RSRZ outliers	91569	7873 (2.04-2.00)

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  $\geq 3$ , 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	221	<div> <div>3%</div> <div>85%</div> <div>10%</div> <div>••</div> </div>
1	B	221	<div> <div>5%</div> <div>83%</div> <div>14%</div> <div>•</div> </div>

## 2 Entry composition

There are 5 unique types of molecules in this entry. The entry contains 3909 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 Catalytic Domain of ADAMTS-5.

Mol	Chain	Residues	Atoms					ZeroOcc	AltConf	Trace
1	A	217	Total	C	N	O	S	0	0	0
			1668	1034	299	324	11			
1	B	220	Total	C	N	O	S	0	0	0
			1682	1043	302	326	11			

There are 6 discrepancies between the modelled and reference sequences:

Chain	Residue	Modelled	Actual	Comment	Reference
A	260	MET	-	EXPRESSION TAG	UNP Q9UNA0
A	261	ALA	-	EXPRESSION TAG	UNP Q9UNA0
A	282	LYS	LEU	ENGINEERED	UNP Q9UNA0
B	260	MET	-	EXPRESSION TAG	UNP Q9UNA0
B	261	ALA	-	EXPRESSION TAG	UNP Q9UNA0
B	282	LYS	LEU	ENGINEERED	UNP Q9UNA0

- Molecule 2 is ZINC ION (three-letter code: ZN) (formula: Zn).

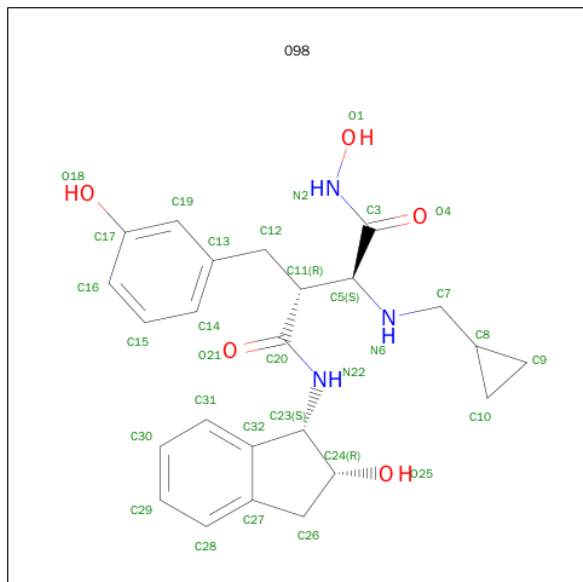
Mol	Chain	Residues	Atoms		ZeroOcc	AltConf
2	B	1	Total	Zn	0	0
			1	1		
2	A	1	Total	Zn	0	0
			1	1		

- Molecule 3 is CALCIUM ION (three-letter code: CA) (formula: Ca).

Mol	Chain	Residues	Atoms		ZeroOcc	AltConf
3	B	3	Total	Ca	0	0
			3	3		
3	A	3	Total	Ca	0	0
			3	3		

- Molecule 4 is (3R)-N 2 -(CYCLOPROPYLMETHYL)-N 1 -HYDROXY-3-(3-HYDROXYB

ENZYL)-N 4 -[(1S,2R)-2-HYDROXY-2,3-DIHYDRO-1H-INDEN-1-YL]-L-ASPARTAMIDE (three-letter code: 098) (formula: C<sub>24</sub>H<sub>29</sub>N<sub>3</sub>O<sub>5</sub>).



Mol	Chain	Residues	Atoms				ZeroOcc	AltConf
4	A	1	Total	C	N	O	0	0
			32	24	3	5		
4	B	1	Total	C	N	O	0	0
			32	24	3	5		


- Molecule 5 is water.

Mol	Chain	Residues	Atoms		ZeroOcc	AltConf
5	A	235	Total	O	0	0
			235	235		
5	B	252	Total	O	0	0
			252	252		

### 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 errors 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 ( $\text{RSRZ} > 2$ ). 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: Catalytic Domain of ADAMTS-5

Chain A: 



#### • Molecule 1: Catalytic Domain of ADAMTS-5

Chain B: 



## 4 Data and refinement statistics

Property	Value	Source
Space group	P 1 21 1	Depositor
Cell constants a, b, c, $\alpha$ , $\beta$ , $\gamma$	52.69Å 44.44Å 76.46Å 90.00° 90.19° 90.00°	Depositor
Resolution (Å)	31.02 – 2.02 31.02 – 2.02	Depositor EDS
% Data completeness (in resolution range)	99.7 (31.02-2.02) 99.7 (31.02-2.02)	Depositor EDS
$R_{merge}$	0.04	Depositor
$R_{sym}$	0.04	Depositor
$\langle I/\sigma(I) \rangle$ <sup>1</sup>	3.98 (at 2.01Å)	Xtriage
Refinement program	REFMAC 5.2.0019	Depositor
R, $R_{free}$	0.159 , 0.238 0.160 , 0.236	Depositor DCC
$R_{free}$ test set	1205 reflections (5.42%)	DCC
Wilson B-factor (Å <sup>2</sup> )	18.6	Xtriage
Anisotropy	0.140	Xtriage
Bulk solvent $k_{sol}$ (e/Å <sup>3</sup> ), $B_{sol}$ (Å <sup>2</sup> )	0.32 , 50.9	EDS
Estimated twinning fraction	0.037 for h,-k,-l	Xtriage
L-test for twinning <sup>2</sup>	$\langle  L  \rangle = 0.48$ , $\langle L^2 \rangle = 0.31$	Xtriage
Outliers	0 of 23440 reflections	Xtriage
$F_o, F_c$ correlation	0.95	EDS
Total number of atoms	3909	wwPDB-VP
Average B, all atoms (Å <sup>2</sup> )	20.0	wwPDB-VP

Xtriage's analysis on translational NCS is as follows: *The largest off-origin peak in the Patterson function is 7.10% of the height of the origin peak. No significant pseudotranslation is detected.*

<sup>1</sup>Intensities estimated from amplitudes.

<sup>2</sup>Theoretical values of  $\langle |L| \rangle$ ,  $\langle L^2 \rangle$  for acentric reflections are 0.5, 0.375 respectively for untwinned datasets, and 0.333, 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: 098, ZN, CA

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	0.49	0/1698	0.60	1/2296 (0.0%)
1	B	0.55	0/1712	0.68	1/2316 (0.0%)
All	All	0.52	0/3410	0.64	2/4612 (0.0%)

There are no bond length outliers.

All (2) bond angle outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(°)	Ideal(°)
1	A	443	LEU	CA-CB-CG	5.59	128.16	115.30
1	B	367	ARG	NE-CZ-NH2	-5.54	117.53	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	1668	0	1607	21	0
1	B	1682	0	1613	25	0
2	A	1	0	0	0	0
2	B	1	0	0	0	0
3	A	3	0	0	0	0
3	B	3	0	0	0	0
4	A	32	0	27	1	0

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Mol	Chain	Non-H	H(model)	H(added)	Clashes	Symm-Clashes
4	B	32	0	27	2	0
5	A	235	0	0	13	0
5	B	252	0	0	7	0
All	All	3909	0	3274	47	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 7.

All (47) 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:393:SER:HB3	5:A:5242:HOH:O	1.67	0.93
1:B:378:THR:HG21	5:B:6027:HOH:O	1.76	0.83
1:B:325:ASP:C	1:B:327:SER:H	1.83	0.81
1:A:443:LEU:HB2	5:A:5024:HOH:O	1.86	0.76
1:B:325:ASP:O	1:B:327:SER:N	2.19	0.75
1:B:299:ASN:ND2	1:B:313:LEU:H	1.90	0.70
1:B:281:ARG:HD2	5:B:6067:HOH:O	1.93	0.67
1:A:299:ASN:HD22	1:A:313:LEU:H	1.40	0.67
1:B:378:THR:HG23	4:B:801:098:H7	1.78	0.65
1:B:344:TRP:HA	1:B:347:GLN:HE21	1.61	0.65
1:B:325:ASP:C	1:B:327:SER:N	2.52	0.63
1:A:299:ASN:ND2	1:A:313:LEU:H	1.99	0.59
1:B:402:LEU:HG	1:B:403:HIS:CE1	2.38	0.59
1:A:478:LYS:HG3	5:B:5105:HOH:O	2.03	0.59
1:A:468:HIS:HE1	5:A:5034:HOH:O	1.86	0.58
1:A:424:LYS:HE2	5:A:5012:HOH:O	2.03	0.57
1:A:390:PRO:HA	5:A:5242:HOH:O	2.05	0.56
1:B:322:GLY:HA2	5:B:6086:HOH:O	2.06	0.55
1:A:468:HIS:HD2	5:A:5078:HOH:O	1.89	0.54
1:A:281:ARG:HD2	5:A:5090:HOH:O	2.07	0.53
1:A:312:ARG:NH1	5:A:5161:HOH:O	2.40	0.53
1:A:465:ASP:CG	5:A:5157:HOH:O	2.47	0.52
1:A:285:ARG:NH1	1:B:422:ASP:O	2.43	0.50
1:B:267:ARG:HH12	1:B:470:ASN:HD22	1.60	0.50
1:B:468:HIS:HE1	5:B:6057:HOH:O	1.96	0.49
1:B:371:CYS:HA	1:B:376:CYS:HA	1.94	0.49
1:A:312:ARG:HD3	5:A:5169:HOH:O	2.13	0.49
1:A:477:ARG:HD2	5:A:5173:HOH:O	2.12	0.48
1:B:367:ARG:HD2	5:B:6032:HOH:O	2.13	0.48
1:B:324:LYS:O	1:B:325:ASP:CB	2.61	0.48

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Atom-1	Atom-2	Interatomic distance (Å)	Clash overlap (Å)
1:B:322:GLY:O	1:B:323:ASP:O	2.31	0.48
1:B:475:LEU:N	1:B:476:PRO:CD	2.77	0.48
1:B:444:THR:HG22	1:B:445:SER:N	2.31	0.46
1:A:442:ILE:H	1:A:442:ILE:HD13	1.82	0.45
1:B:267:ARG:HH12	1:B:470:ASN:ND2	2.15	0.45
1:B:272:LEU:CD1	1:B:317:LYS:HG3	2.47	0.45
1:B:416:LEU:HB3	1:B:460:ILE:HD11	1.99	0.44
1:B:475:LEU:N	1:B:476:PRO:HD3	2.33	0.44
1:B:272:LEU:HD13	1:B:317:LYS:HG3	1.99	0.44
1:A:356:GLU:HG2	1:A:477:ARG:HD3	1.99	0.43
1:A:275:ALA:HB2	1:A:291:LEU:HD11	2.01	0.42
1:A:324:LYS:C	1:A:326:LYS:H	2.23	0.42
4:B:801:098:H31	5:B:6063:HOH:O	2.19	0.41
1:B:378:THR:HG22	1:B:380:GLY:H	1.86	0.41
1:A:371:CYS:HA	1:A:376:CYS:HA	2.02	0.40
1:A:310:HIS:HE1	5:A:5094:HOH:O	2.03	0.40
4:A:801:098:H15	5:A:5190:HOH:O	2.21	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	215/221 (97%)	208 (97%)	3 (1%)	4 (2%)	10	3
1	B	218/221 (99%)	208 (95%)	5 (2%)	5 (2%)	8	2
All	All	433/442 (98%)	416 (96%)	8 (2%)	9 (2%)	9	3

All (9) Ramachandran outliers are listed below:

Mol	Chain	Res	Type
1	B	262	SER

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Mol	Chain	Res	Type
1	B	323	ASP
1	B	326	LYS
1	A	327	SER
1	A	324	LYS
1	A	326	LYS
1	A	325	ASP
1	B	325	ASP
1	B	475	LEU

### 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	181/188 (96%)	172 (95%)	9 (5%)	30	24
1	B	180/188 (96%)	171 (95%)	9 (5%)	30	24
All	All	361/376 (96%)	343 (95%)	18 (5%)	30	24

All (18) residues with a non-rotameric sidechain are listed below:

Mol	Chain	Res	Type
1	A	328	LEU
1	A	375	SER
1	A	394	CYS
1	A	402	LEU
1	A	424	LYS
1	A	442	ILE
1	A	443	LEU
1	A	460	ILE
1	A	465	ASP
1	B	285	ARG
1	B	351	LEU
1	B	378	THR
1	B	394	CYS
1	B	402	LEU
1	B	433	THR

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Mol	Chain	Res	Type
1	B	443	LEU
1	B	460	ILE
1	B	478	LYS

Some sidechains can be flipped to improve hydrogen bonding and reduce clashes. All (10) such sidechains are listed below:

Mol	Chain	Res	Type
1	A	289	HIS
1	A	299	ASN
1	A	309	ASN
1	A	468	HIS
1	B	268	GLN
1	B	299	ASN
1	B	347	GLN
1	B	403	HIS
1	B	468	HIS
1	B	470	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 ⓘ

Of 10 ligands modelled in this entry, 8 are monoatomic - leaving 2 for Mogul analysis.

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	$\# Z  > 2$
4	098	A	801	2	34,35,35	1.08	3 (8%)	38,49,49	1.70	7 (18%)
4	098	B	801	2	34,35,35	1.07	2 (5%)	38,49,49	1.50	6 (15%)

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
4	098	A	801	2	-	0/27/41/41	0/3/4/4
4	098	B	801	2	-	0/27/41/41	0/3/4/4

All (5) bond length outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(Å)	Ideal(Å)
4	A	801	098	C24-C23	-2.98	1.52	1.55
4	B	801	098	C24-C23	-2.78	1.52	1.55
4	B	801	098	C32-C23	-2.76	1.48	1.51
4	A	801	098	C32-C23	-2.54	1.49	1.51
4	A	801	098	C20-N22	2.10	1.38	1.34

All (13) bond angle outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(°)	Ideal(°)
4	A	801	098	O1-N2-C3	-6.47	110.72	119.86
4	B	801	098	O1-N2-C3	-5.78	111.70	119.86
4	B	801	098	C5-C3-N2	-2.96	109.88	116.42
4	A	801	098	C5-C3-N2	-2.77	110.29	116.42
4	A	801	098	C30-C31-C32	-2.71	117.49	121.02
4	A	801	098	C11-C5-C3	-2.69	105.88	111.36
4	A	801	098	C29-C28-C27	-2.32	117.30	120.90
4	A	801	098	C11-C20-N22	-2.31	113.24	116.08
4	B	801	098	C17-C19-C13	-2.23	119.05	120.45
4	B	801	098	C30-C31-C32	-2.17	118.19	121.02
4	B	801	098	C11-C20-N22	-2.14	113.44	116.08
4	A	801	098	O21-C20-N22	2.25	127.34	122.93
4	B	801	098	O4-C3-C5	2.27	125.36	120.68

There are no chirality outliers.

There are no torsion outliers.

There are no ring outliers.

2 monomers are involved in 3 short contacts:

Mol	Chain	Res	Type	Clashes	Symm-Clashes
4	A	801	098	1	0
4	B	801	098	2	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 [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<sup>th</sup> 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 <sup>2</sup> )	Q<0.9
1	A	217/221 (98%)	-0.17	7 (3%) 51 53	11, 19, 34, 44	1 (0%)
1	B	220/221 (99%)	-0.18	11 (5%) 32 34	8, 15, 32, 42	1 (0%)
All	All	437/442 (98%)	-0.18	18 (4%) 41 43	8, 17, 34, 44	2 (0%)

All (18) RSRZ outliers are listed below:

Mol	Chain	Res	Type	RSRZ
1	B	261	ALA	7.8
1	B	262	SER	7.5
1	A	325	ASP	6.3
1	B	324	LYS	4.8
1	B	325	ASP	4.5
1	B	351	LEU	4.3
1	B	323	ASP	4.3
1	A	324	LYS	4.1
1	A	374	HIS	4.0
1	A	323	ASP	3.3
1	B	326	LYS	3.2
1	A	327	SER	3.0
1	B	374	HIS	2.9
1	B	432	SER	2.7
1	B	322	GLY	2.6
1	A	322	GLY	2.2
1	B	263	ILE	2.2
1	A	402	LEU	2.1

### 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, 95<sup>th</sup> 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( $\text{\AA}^2$ )	Q<0.9
4	098	B	801	32/32	0.90	0.15	1.01	14,24,33,33	0
4	098	A	801	32/32	0.91	0.15	0.91	22,27,31,31	0
3	CA	B	903	1/1	0.99	0.04	-1.81	15,15,15,15	0
3	CA	B	902	1/1	1.00	0.05	-2.18	13,13,13,13	0
3	CA	A	904	1/1	0.98	0.04	-2.72	18,18,18,18	0
3	CA	B	904	1/1	1.00	0.04	-2.75	11,11,11,11	0
3	CA	A	903	1/1	1.00	0.05	-5.89	13,13,13,13	0
3	CA	A	902	1/1	0.99	0.03	-16.92	12,12,12,12	0
2	ZN	A	901	1/1	1.00	0.04	-	14,14,14,14	0
2	ZN	B	901	1/1	1.00	0.03	-	12,12,12,12	0

### 6.5 Other polymers [i](#)

There are no such residues in this entry.