



# Full wwPDB X-ray Structure Validation Report ⓘ

Feb 13, 2017 – 08:59 pm GMT

PDB ID : 1S18  
Title : Structure and protein design of human apyrase  
Authors : Dai, J.; Liu, J.; Deng, Y.; Smith, T.M.; Lu, M.  
Deposited on : 2004-01-05  
Resolution : 1.70 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 : 1.7.2 (RC1), CSD as538be (2017)  
Xtriage (Phenix) : 1.9-1692  
EDS : trunk28620  
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) : recalc28949

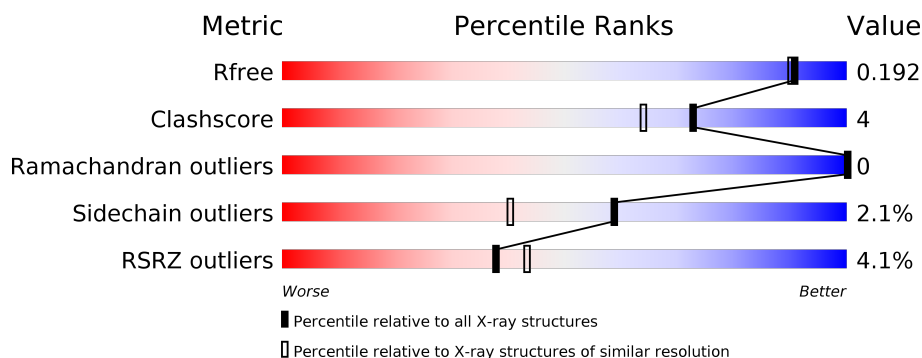
# 1 Overall quality at a glance

The following experimental techniques were used to determine the structure:

*X-RAY DIFFRACTION*



The reported resolution of this entry is 1.70 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	3453 (1.70-1.70)
Clashscore	112137	3876 (1.70-1.70)
Ramachandran outliers	110173	3815 (1.70-1.70)
Sidechain outliers	110143	3815 (1.70-1.70)
RSRZ outliers	101464	3491 (1.70-1.70)

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	331	
1	B	331	

The following table lists non-polymeric compounds, carbohydrate monomers and non-standard residues in protein, DNA, RNA chains that are outliers for geometric or electron-density-fit criteria:

Mol	Type	Chain	Res	Chirality	Geometry	Clashes	Electron density
2	CA	B	1002	-	-	-	X
3	ACT	A	3001	-	-	-	X
3	ACT	B	3003	-	-	X	X
4	TRS	A	2001	-	-	-	X
4	TRS	A	2005	-	-	-	X
4	TRS	B	2003	-	-	-	X
4	TRS	B	2004	-	-	-	X
4	TRS	B	2007	-	-	-	X

## 2 Entry composition [i](#)

There are 5 unique types of molecules in this entry. The entry contains 5597 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.

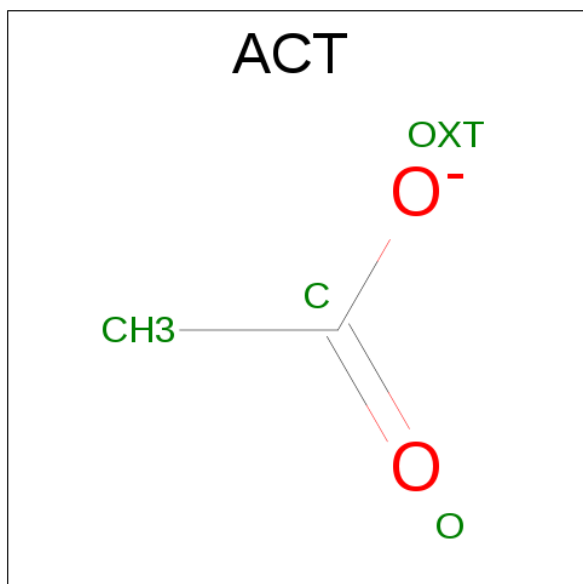
- Molecule 1 is a protein called apyrase.

Mol	Chain	Residues	Atoms					ZeroOcc	AltConf	Trace
1	A	317	Total	C	N	O	S	0	0	0
			2510	1599	421	487	3			
1	B	315	Total	C	N	O	S	0	0	0
			2488	1584	417	484	3			

- Molecule 2 is CALCIUM ION (three-letter code: CA) (formula: Ca).

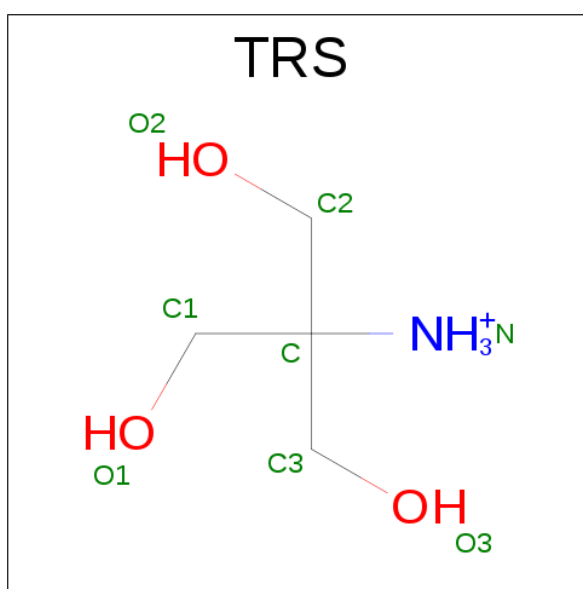
Mol	Chain	Residues	Atoms		ZeroOcc	AltConf
2	B	1	Total	Ca	0	0
			1	1		
2	A	1	Total	Ca	0	0
			1	1		

- Molecule 3 is ACETATE ION (three-letter code: ACT) (formula: C<sub>2</sub>H<sub>3</sub>O<sub>2</sub>).



Mol	Chain	Residues	Atoms	ZeroOcc	AltConf
3	A	1	Total C O 4 2 2	0	0
3	B	1	Total C O 4 2 2	0	0
3	B	1	Total C O 4 2 2	0	0
3	B	1	Total C O 4 2 2	0	0

- Molecule 4 is 2-AMINO-2-HYDROXYMETHYL-PROPANE-1,3-DIOL (three-letter code: TRS) (formula:  $C_4H_{12}NO_3$ ).



Mol	Chain	Residues	Atoms	ZeroOcc	AltConf
4	A	1	Total C N O 8 4 1 3	0	0
4	A	1	Total C N O 8 4 1 3	0	0
4	B	1	Total C N O 8 4 1 3	0	0
4	B	1	Total C N O 8 4 1 3	0	0
4	A	1	Total C N O 8 4 1 3	0	0
4	B	1	Total C N O 8 4 1 3	0	0
4	B	1	Total C N O 8 4 1 3	0	0

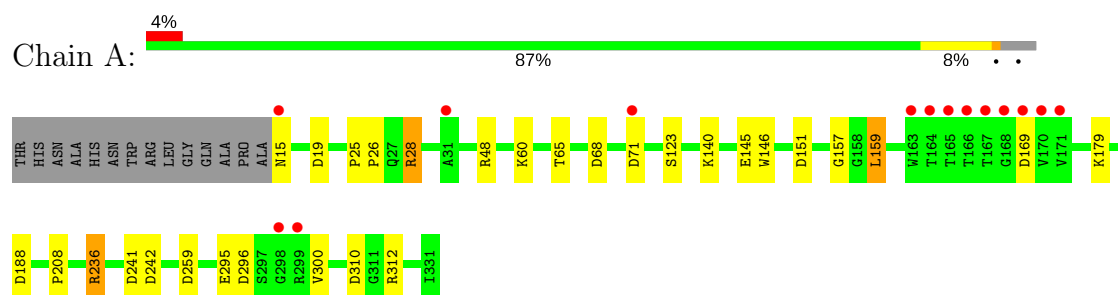
- Molecule 5 is water.

Mol	Chain	Residues	Atoms		ZeroOcc	AltConf
5	A	277	Total 277	O 277	0	0
5	B	248	Total 248	O 248	0	0

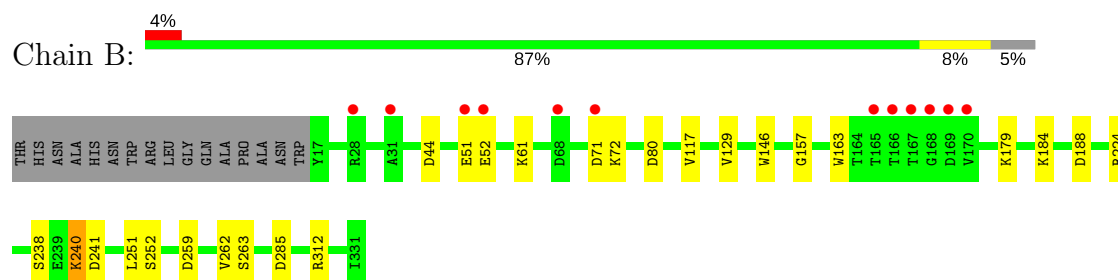
### 3 Residue-property plots [i](#)

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: apyrase



#### • Molecule 1: apyrase



## 4 Data and refinement statistics

Property	Value	Source
Space group	P 1	Depositor
Cell constants a, b, c, $\alpha$ , $\beta$ , $\gamma$	43.16Å 52.50Å 77.92Å 99.44° 106.58° 99.89°	Depositor
Resolution (Å)	72.55 – 1.70 37.64 – 1.70	Depositor EDS
% Data completeness (in resolution range)	95.1 (72.55-1.70) 89.5 (37.64-1.70)	Depositor EDS
$R_{merge}$	0.06	Depositor
$R_{sym}$	(Not available)	Depositor
$\langle I/\sigma(I) \rangle$ <sup>1</sup>	3.13 (at 1.70Å)	Xtriage
Refinement program	REFMAC 5.1.24	Depositor
R, $R_{free}$	0.157 , 0.191 0.158 , 0.192	Depositor DCC
$R_{free}$ test set	3323 reflections (5.33%)	DCC
Wilson B-factor (Å <sup>2</sup> )	14.8	Xtriage
Anisotropy	0.111	Xtriage
Bulk solvent $k_{sol}$ (e/Å <sup>3</sup> ), $B_{sol}$ (Å <sup>2</sup> )	0.39 , 50.4	EDS
L-test for twinning <sup>2</sup>	$\langle  L  \rangle = 0.50$ , $\langle L^2 \rangle = 0.33$	Xtriage
Estimated twinning fraction	No twinning to report.	Xtriage
$F_o, F_c$ correlation	0.96	EDS
Total number of atoms	5597	wwPDB-VP
Average B, all atoms (Å <sup>2</sup> )	19.0	wwPDB-VP

Xtriage's analysis on translational NCS is as follows: *The largest off-origin peak in the Patterson function is 7.27% 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, 0.333 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: CA, TRS, ACT

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	0.76	0/2574	0.88	8/3496 (0.2%)
1	B	0.74	0/2550	0.88	3/3462 (0.1%)
All	All	0.75	0/5124	0.88	11/6958 (0.2%)

There are no bond length outliers.

All (11) bond angle outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(°)	Ideal(°)
1	B	259	ASP	CB-CG-OD2	6.63	124.27	118.30
1	A	310	ASP	CB-CG-OD2	6.29	123.96	118.30
1	A	259	ASP	CB-CG-OD2	6.03	123.72	118.30
1	A	242	ASP	CB-CG-OD2	5.97	123.67	118.30
1	A	296	ASP	CB-CG-OD2	5.65	123.38	118.30
1	A	151	ASP	CB-CG-OD2	5.60	123.34	118.30
1	A	19	ASP	CB-CG-OD2	5.58	123.32	118.30
1	A	188	ASP	CB-CG-OD2	5.45	123.20	118.30
1	A	169	ASP	CB-CG-OD2	5.42	123.18	118.30
1	B	188	ASP	CB-CG-OD2	5.27	123.04	118.30
1	B	285	ASP	CB-CG-OD2	5.15	122.94	118.30

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	2510	0	2418	16	0
1	B	2488	0	2402	18	0
2	A	1	0	0	0	0
2	B	1	0	0	0	0
3	A	4	0	3	0	0
3	B	12	0	9	3	0
4	A	24	0	36	3	0
4	B	32	0	48	2	0
5	A	277	0	0	11	0
5	B	248	0	0	2	0
All	All	5597	0	4916	39	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 4.

All (39) 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:B:71:ASP:HB2	1:B:312:ARG:HH22	1.02	1.15
5:A:3259:HOH:O	1:B:80:ASP:HB2	1.66	0.96
1:B:71:ASP:HB2	1:B:312:ARG:NH2	1.88	0.88
1:B:263:SER:HA	4:B:2006:TRS:H31	1.70	0.74
1:A:145:GLU:HG3	1:A:159:LEU:HD13	1.69	0.73
1:B:51:GLU:HG2	1:B:52:GLU:N	2.04	0.72
1:A:295:GLU:HG3	1:A:300:VAL:HG22	1.72	0.71
4:A:2001:TRS:H22	5:A:3145:HOH:O	1.94	0.68
3:B:3003:ACT:H1	5:B:3046:HOH:O	1.98	0.64
1:A:208:PRO:HB2	1:A:236:ARG:HB3	1.85	0.59
1:A:236:ARG:NH2	1:A:241:ASP:OD2	2.36	0.58
1:A:71:ASP:HB3	1:A:312:ARG:HH12	1.71	0.56
1:A:65:THR:HG23	5:A:3125:HOH:O	2.06	0.55
1:B:251:LEU:HD23	1:B:262:VAL:HG13	1.88	0.54
1:A:159:LEU:HD23	5:A:3253:HOH:O	2.07	0.53
1:B:146:TRP:CE2	1:B:157:GLY:HA3	2.44	0.52
1:B:51:GLU:HG2	1:B:52:GLU:H	1.73	0.51
1:B:51:GLU:CG	1:B:52:GLU:N	2.73	0.49
3:B:3003:ACT:H3	5:B:3235:HOH:O	2.11	0.49
1:B:240:LYS:HG2	1:B:241:ASP:H	1.80	0.47
1:A:28:ARG:NH2	1:A:68:ASP:OD1	2.40	0.46
1:B:117:VAL:HB	1:B:129:VAL:HG22	1.96	0.46
5:A:3259:HOH:O	1:B:80:ASP:CB	2.41	0.46
1:A:48:ARG:HG3	5:A:3050:HOH:O	2.16	0.45

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Atom-1	Atom-2	Interatomic distance (Å)	Clash overlap (Å)
1:B:251:LEU:CD2	1:B:262:VAL:HG13	2.46	0.45
1:A:71:ASP:CB	1:A:312:ARG:HH12	2.30	0.45
1:B:240:LYS:HE3	1:B:240:LYS:HB3	1.78	0.45
1:A:146:TRP:CE2	1:A:157:GLY:HA3	2.52	0.44
1:B:51:GLU:CG	1:B:52:GLU:H	2.30	0.44
4:A:2005:TRS:H12	5:A:3021:HOH:O	2.16	0.44
1:B:61:LYS:NZ	4:B:2007:TRS:H31	2.33	0.44
1:B:163:TRP:HH2	3:B:3004:ACT:H3	1.83	0.43
1:A:15:ASN:HB3	5:A:3254:HOH:O	2.18	0.43
4:A:2001:TRS:C2	5:A:3145:HOH:O	2.61	0.43
1:A:123:SER:HB3	5:A:3046:HOH:O	2.19	0.42
1:A:60:LYS:NZ	5:A:3225:HOH:O	2.44	0.41
1:A:28:ARG:HH22	1:A:68:ASP:CG	2.21	0.41
1:A:25:PRO:HA	1:A:26:PRO:HD3	1.96	0.41
1:B:224:ARG:NE	1:B:252:SER:OG	2.42	0.41

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	315/331 (95%)	312 (99%)	3 (1%)	0	100	100
1	B	313/331 (95%)	310 (99%)	3 (1%)	0	100	100
All	All	628/662 (95%)	622 (99%)	6 (1%)	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	267/277 (96%)	262 (98%)	5 (2%)	62	45
1	B	265/277 (96%)	259 (98%)	6 (2%)	56	36
All	All	532/554 (96%)	521 (98%)	11 (2%)	59	40

All (11) residues with a non-rotameric sidechain are listed below:

Mol	Chain	Res	Type
1	A	28	ARG
1	A	140	LYS
1	A	159	LEU
1	A	179	LYS
1	A	236	ARG
1	B	44	ASP
1	B	72	LYS
1	B	179	LYS
1	B	184	LYS
1	B	238	SER
1	B	240	LYS

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	195	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 13 ligands modelled in this entry, 2 are monoatomic - leaving 11 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	TRS	A	2001	-	7,7,7	1.00	0	9,9,9	1.40	2 (22%)
4	TRS	A	2002	-	7,7,7	0.42	0	9,9,9	0.62	0
4	TRS	A	2005	-	7,7,7	0.60	0	9,9,9	0.72	0
3	ACT	A	3001	-	1,3,3	1.07	0	0,3,3	0.00	-
4	TRS	B	2003	-	7,7,7	1.23	1 (14%)	9,9,9	1.34	0
4	TRS	B	2004	-	7,7,7	0.65	0	9,9,9	0.42	0
4	TRS	B	2006	-	7,7,7	0.70	0	9,9,9	1.07	0
4	TRS	B	2007	-	7,7,7	0.35	0	9,9,9	0.61	0
3	ACT	B	3002	-	1,3,3	1.29	0	0,3,3	0.00	-
3	ACT	B	3003	-	1,3,3	1.52	0	0,3,3	0.00	-
3	ACT	B	3004	-	1,3,3	1.08	0	0,3,3	0.00	-

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	TRS	A	2001	-	-	0/9/9/9	0/0/0/0
4	TRS	A	2002	-	-	0/9/9/9	0/0/0/0
4	TRS	A	2005	-	-	0/9/9/9	0/0/0/0
3	ACT	A	3001	-	-	0/0/0/0	0/0/0/0
4	TRS	B	2003	-	-	0/9/9/9	0/0/0/0
4	TRS	B	2004	-	-	0/9/9/9	0/0/0/0
4	TRS	B	2006	-	-	0/9/9/9	0/0/0/0
4	TRS	B	2007	-	-	0/9/9/9	0/0/0/0
3	ACT	B	3002	-	-	0/0/0/0	0/0/0/0
3	ACT	B	3003	-	-	0/0/0/0	0/0/0/0
3	ACT	B	3004	-	-	0/0/0/0	0/0/0/0

All (1) bond length outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(Å)	Ideal(Å)
4	B	2003	TRS	O2-C2	2.45	1.50	1.42

All (2) bond angle outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(°)	Ideal(°)
4	A	2001	TRS	C3-C-C2	-2.14	105.01	111.06
4	A	2001	TRS	C3-C-N	2.48	113.00	107.73

There are no chirality outliers.

There are no torsion outliers.

There are no ring outliers.

6 monomers are involved in 8 short contacts:

Mol	Chain	Res	Type	Clashes	Symm-Clashes
4	A	2001	TRS	2	0
4	A	2005	TRS	1	0
4	B	2006	TRS	1	0
4	B	2007	TRS	1	0
3	B	3003	ACT	2	0
3	B	3004	ACT	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, 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	317/331 (95%)	-0.16	14 (4%) 35 40	8, 16, 34, 48	0
1	B	315/331 (95%)	-0.23	12 (3%) 41 47	8, 16, 32, 39	0
All	All	632/662 (95%)	-0.19	26 (4%) 38 43	8, 16, 32, 48	0

All (26) RSRZ outliers are listed below:

Mol	Chain	Res	Type	RSRZ
1	A	170	VAL	5.7
1	A	169	ASP	4.9
1	A	167	THR	4.8
1	A	166	THR	4.5
1	A	165	THR	4.4
1	B	167	THR	4.4
1	A	31	ALA	4.2
1	B	166	THR	3.9
1	A	171	VAL	3.7
1	B	169	ASP	3.4
1	A	298	GLY	3.3
1	A	164	THR	3.2
1	B	165	THR	3.1
1	B	31	ALA	2.9
1	B	68	ASP	2.7
1	A	299	ARG	2.6
1	B	51	GLU	2.6
1	B	52	GLU	2.6
1	A	168	GLY	2.5
1	B	168	GLY	2.4
1	A	15	ASN	2.2
1	B	28	ARG	2.1
1	B	170	VAL	2.1
1	A	71	ASP	2.0

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Mol	Chain	Res	Type	RSRZ
1	A	163	TRP	2.0
1	B	71	ASP	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, 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(Å <sup>2</sup> )	Q<0.9
4	TRS	B	2007	8/8	0.74	0.30	10.80	46,47,48,49	0
4	TRS	A	2005	8/8	0.83	0.20	8.44	22,27,30,32	0
3	ACT	B	3003	4/4	0.75	0.23	6.97	39,40,40,41	0
4	TRS	B	2003	8/8	0.84	0.16	4.71	16,27,28,31	0
4	TRS	B	2004	8/8	0.82	0.18	4.27	38,43,44,44	0
2	CA	B	1002	1/1	0.99	0.18	4.05	3,3,3,3	0
4	TRS	A	2001	8/8	0.86	0.19	4.00	17,27,30,33	0
3	ACT	A	3001	4/4	0.89	0.11	2.13	49,49,49,49	0
4	TRS	A	2002	8/8	0.89	0.14	1.95	39,44,45,46	0
4	TRS	B	2006	8/8	0.68	0.26	1.52	36,42,43,44	0
3	ACT	B	3002	4/4	0.82	0.15	0.70	47,47,47,48	0
2	CA	A	1001	1/1	1.00	0.09	-0.51	8,8,8,8	0
3	ACT	B	3004	4/4	0.85	0.15	-	47,47,47,48	0

## 6.5 Other polymers [i](#)

There are no such residues in this entry.