



Full wwPDB X-ray Structure Validation Report ⓘ

Sep 5, 2014 – 09:06 AM EDT

PDB ID : 4D0W
Title : Pyrrole-3-carboxamides as potent and selective JAK2 inhibitors
Authors : Bertrand, J.; Canevari, G.; Fasolini, M.; Brasca, M.G.; Nesi, M.; Avanzi, N.; Ballinari, D.; Bandiera, T.; Bindi, S.; Carenzi, D.; Casero, D.; Ceriani, L.; Ciomei, M.; Cirila, A.; Colombo, M.; Cribioli, S.; Cristiani, C.; Della Vedova, F.; Fachin, G.; Felder, E.R.; Galvani, A.; Isacchi, A.; Mirizzi, D.; Motto, I.; Panzeri, A.; Pesenti, E.; Vianello, P.; Gnocchi, P.; Donati, D.
Deposited on : 2014-04-30
Resolution : 1.77 Å (reported)

This is a full wwPDB 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/ValidationPDFNotes.html>

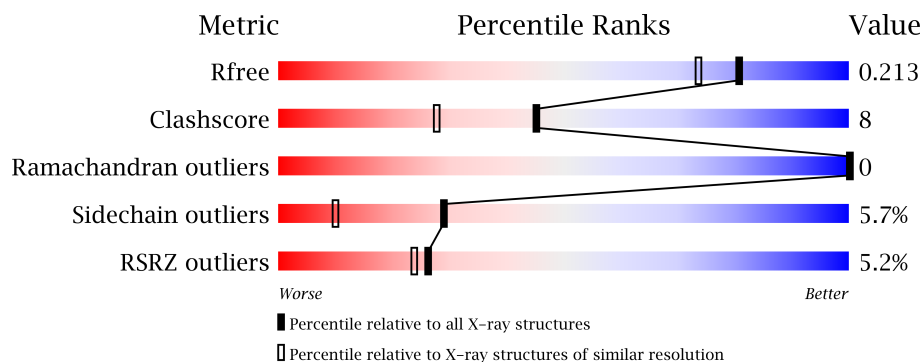
The following versions of software and data (see [references](#)) were used in the production of this report:

MolProbity : 4.02b-467
Mogul : 1.16 November 2013
Xtriage (Phenix) : dev-1439
EDS : stable23489
Percentile statistics : 21963
Refmac : 5.8.0049
CCP4 : 6.3.0 (Settle)
Ideal geometry (proteins) : Engh & Huber (2001)
Ideal geometry (DNA, RNA) : Parkinson et. al. (1996)
Validation Pipeline (wwPDB-VP) : stable23489

1 Overall quality at a glance

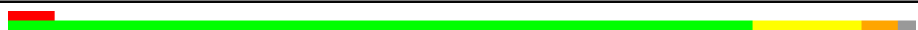
The reported resolution of this entry is 1.77 Å.

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}	66092	4987 (1.80-1.76)
Clashscore	79885	6152 (1.80-1.76)
Ramachandran outliers	78287	6074 (1.80-1.76)
Sidechain outliers	78261	6073 (1.80-1.76)
RSRZ outliers	66119	4990 (1.80-1.76)

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. The upper red bar (where present) indicates the fraction of residues that have poor fit to the electron density.

Mol	Chain	Length	Quality of chain
1	A	298	

2 Entry composition i

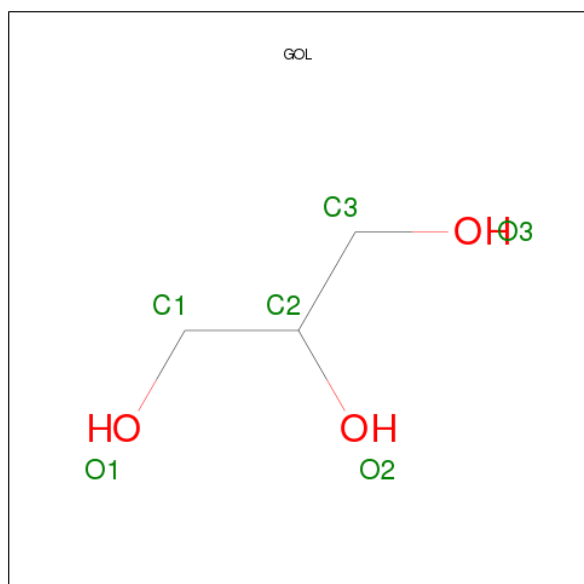
There are 4 unique types of molecules in this entry. The entry contains 2627 atoms, of which 0 are hydrogen and 0 are deuterium.

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 TYROSINE-PROTEIN KINASE JAK2.

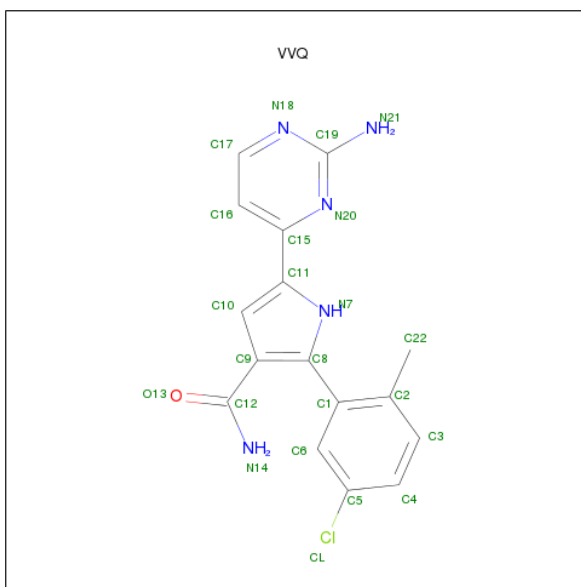
Mol	Chain	Residues	Atoms						ZeroOcc	AltConf	Trace
			Total	C	N	O	P	S			
1	A	291	2442	1548	424	452	2	16	0	5	0

- Molecule 2 is GLYCEROL (three-letter code: GOL) (formula: C₃H₈O₃).



Mol	Chain	Residues	Atoms			ZeroOcc	AltConf
			Total	C	O		
2	A	1	6	3	3	0	0

- Molecule 3 is 5-(2-AMINOPYRIMIDIN-4-YL)-2-(5-CHLORO-2-METHYLPHENYL)-1H-PYRROLE-3-CARBOXAMIDE (three-letter code: VVQ) (formula: C₁₆H₁₄ClN₅O).



Mol	Chain	Residues	Atoms					ZeroOcc	AltConf
3	A	1	Total	C	Cl	N	O	0	0
			23	16	1	5	1		

- Molecule 4 is water.

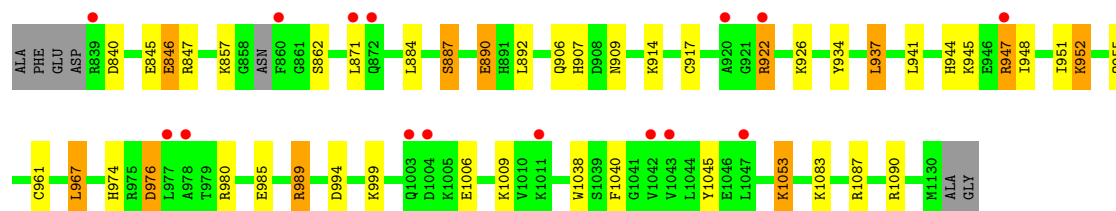
Mol	Chain	Residues	Atoms		ZeroOcc	AltConf
4	A	156	Total	O	0	0
			156	156		

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 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 ($RSRZ > 2$). 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: TYROSINE-PROTEIN KINASE JAK2

Chain A: 



4 Data and refinement statistics

Property	Value	Source
Space group	C 2 2 21	Depositor
Cell constants a, b, c, α , β , γ	93.25Å 102.10Å 68.23Å 90.00° 90.00° 90.00°	Depositor
Resolution (Å)	29.74 – 1.77 29.73 – 1.77	Depositor EDS
% Data completeness (in resolution range)	95.3 (29.74-1.77) 95.3 (29.73-1.77)	Depositor EDS
R_{merge}	0.06	Depositor
R_{sym}	(Not available)	Depositor
$\langle I/\sigma(I) \rangle$ ¹	1.78 (at 1.77Å)	Xtriage
Refinement program	REFMAC 5.6.0117	Depositor
R, R_{free}	0.173 , 0.215 0.173 , 0.213	Depositor DCC
R_{free} test set	1532 reflections (5.27%)	DCC
Wilson B-factor (Å ²)	22.3	Xtriage
Anisotropy	0.363	Xtriage
Bulk solvent k_{sol} (e/Å ³), B_{sol} (Å ²)	0.40 , 38.6	EDS
Estimated twinning fraction	No twinning to report.	Xtriage
L-test for twinning	$\langle L \rangle = 0.50$, $\langle L^2 \rangle = 0.33$	Xtriage
Outliers	0 of 30603 reflections	Xtriage
F_o, F_c correlation	0.96	EDS
Total number of atoms	2627	wwPDB-VP
Average B, all atoms (Å ²)	27.0	wwPDB-VP

Xtriage's analysis on translational NCS is as follows: *The largest off-origin peak in the Patterson function is 5.90% of the height of the origin peak. No significant pseudotranslation is detected.*

¹Intensities estimated from amplitudes.

5 Model quality

5.1 Standard geometry

Bond lengths and bond angles in the following residue types are not validated in this section: GOL, PTR, VVQ

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	1.13	3/2474 (0.1%)	1.07	8/3325 (0.2%)

All (3) bond length outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(Å)	Ideal(Å)
1	A	862	SER	CB-OG	-5.78	1.34	1.42
1	A	1038	TRP	CD2-CE2	5.30	1.47	1.41
1	A	1045	TYR	CG-CD1	5.04	1.45	1.39

All (8) bond angle outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(°)	Ideal(°)
1	A	840	ASP	CB-CG-OD1	8.28	125.75	118.30
1	A	976	ASP	CB-CG-OD1	7.11	124.70	118.30
1	A	967	LEU	CA-CB-CG	6.51	130.28	115.30
1	A	937	LEU	CB-CG-CD2	5.76	120.79	111.00
1	A	1006	GLU	OE1-CD-OE2	-5.57	116.61	123.30
1	A	1087	ARG	NE-CZ-NH1	5.52	123.06	120.30
1	A	1090	ARG	NE-CZ-NH2	-5.41	117.59	120.30
1	A	840	ASP	CB-CG-OD2	-5.32	113.51	118.30

There are no chirality outliers.

There are no planarity outliers.

5.2 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 hydrogens added by MolProbity. The Clashes column lists the number of clashes within the asymmetric unit,

and the number in parentheses is this value normalized per 1000 atoms of the molecule in the chain. The Symm-Clashes column gives symmetry related clashes, in the same way as for the Clashes column.

Mol	Chain	Non-H	H(model)	H(added)	Clashes	Symm-Clashes
1	A	2442	0	2428	41	0
2	A	6	0	8	0	0
3	A	23	0	0	0	0
4	A	156	0	0	4	0
All	All	2627	0	2436	41	0

Clashscore is defined as the number of clashes calculated for the entry per 1000 atoms (including hydrogens) of the entry. The overall clashscore for this entry is 8.

All (41) close contacts within the same asymmetric unit are listed below.

Atom-1	Atom-2	Distance(Å)	Clash(Å)
1:A:1053:LYS:H	1:A:1053:LYS:CD	1.86	0.87
1:A:944:HIS:O	1:A:947:ARG:HG3	1.78	0.83
1:A:887:SER:HB2	1:A:892:LEU:HD11	1.62	0.81
1:A:952:LYS:HZ2	1:A:955:GLN:HE22	1.32	0.77
1:A:846:GLU:HB2	1:A:917[A]:CYS:SG	2.30	0.71
1:A:974:HIS:HD2	1:A:976:ASP:H	1.41	0.69
1:A:1083:LYS:HG3	4:A:3133:HOH:O	1.93	0.68
1:A:914:LYS:HE2	4:A:3025:HOH:O	1.94	0.66
1:A:952:LYS:NZ	1:A:955:GLN:HE22	1.96	0.64
1:A:887:SER:CB	1:A:892:LEU:HD11	2.28	0.63
1:A:884:LEU:HD12	1:A:892:LEU:HD12	1.80	0.62
1:A:922:ARG:NH1	1:A:922:ARG:HG2	2.14	0.62
1:A:922:ARG:HH11	1:A:922:ARG:HG2	1.63	0.62
1:A:1053:LYS:HD3	1:A:1053:LYS:H	1.65	0.61
1:A:999:LYS:HE2	4:A:3093:HOH:O	2.02	0.60
1:A:917[B]:CYS:HB2	1:A:926:LYS:HB2	1.84	0.59
1:A:989:ARG:HH11	1:A:989:ARG:HG2	1.70	0.57
1:A:846:GLU:O	1:A:846:GLU:HG3	2.04	0.56
1:A:845:GLU:HG3	1:A:847:ARG:HG2	1.88	0.55
1:A:941:LEU:HD12	1:A:948:ILE:HD12	1.88	0.54
1:A:1053:LYS:N	1:A:1053:LYS:CD	2.67	0.54
1:A:941:LEU:CD1	1:A:948:ILE:HD12	2.37	0.53
1:A:1053:LYS:HD2	1:A:1053:LYS:H	1.73	0.52
1:A:922:ARG:HH11	1:A:922:ARG:CG	2.23	0.52
1:A:944:HIS:O	1:A:947:ARG:CG	2.56	0.52
1:A:934:TYR:N	1:A:934:TYR:HD1	2.09	0.51
1:A:976:ASP:OD1	1:A:980:ARG:NH2	2.45	0.50
1:A:845:GLU:CG	1:A:847:ARG:HG2	2.42	0.49

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Atom-1	Atom-2	Distance(Å)	Clash(Å)
1:A:974:HIS:CD2	1:A:976:ASP:H	2.25	0.49
1:A:934:TYR:N	1:A:934:TYR:CD1	2.81	0.47
1:A:961[A]:CYS:SG	1:A:1040:PHE:CZ	3.07	0.47
1:A:989:ARG:NH1	1:A:989:ARG:HG2	2.30	0.47
1:A:917[A]:CYS:HB3	1:A:926:LYS:HB2	1.97	0.46
1:A:952:LYS:HZ2	1:A:955:GLN:NE2	2.06	0.46
1:A:1009:LYS:HB2	1:A:1009:LYS:HE3	1.52	0.45
1:A:907:HIS:HE1	1:A:909:ASN:HD22	1.62	0.45
1:A:985:GLU:OE1	1:A:989:ARG:NH1	2.49	0.45
1:A:890[A]:GLU:HG2	1:A:890[A]:GLU:H	1.54	0.43
1:A:952:LYS:NZ	1:A:955:GLN:NE2	2.64	0.42
1:A:914:LYS:CE	4:A:3025:HOH:O	2.58	0.42
1:A:974:HIS:HE1	1:A:994:ASP:O	2.03	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	290/298 (97%)	284 (98%)	6 (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	270/269 (100%)	254 (94%)	16 (6%)	28	9

All (16) residues with a non-rotameric sidechain are listed below:

Mol	Chain	Res	Type
1	A	846	GLU
1	A	857	LYS
1	A	871	LEU
1	A	887	SER
1	A	890[A]	GLU
1	A	890[B]	GLU
1	A	906	GLN
1	A	922	ARG
1	A	937	LEU
1	A	945	LYS
1	A	947	ARG
1	A	951	ILE
1	A	952	LYS
1	A	967	LEU
1	A	989	ARG
1	A	1053	LYS

Some sidechains can be flipped to improve hydrogen bonding and reduce clashes. All (6) such sidechains are listed below:

Mol	Chain	Res	Type
1	A	848	HIS
1	A	853	GLN
1	A	885	GLN
1	A	909	ASN
1	A	955	GLN
1	A	974	HIS

5.3.3 RNA ⓘ

There are no RNA chains in this entry.

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

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	$\# Z > 2$
1	PTR	A	1007	1	16,16,17	4.80	1 (6%)	20,22,24	1.87	5 (25%)
1	PTR	A	1008	1	16,16,17	5.00	2 (12%)	20,22,24	2.56	6 (30%)

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
1	PTR	A	1007	1	-	0/9/11/13	0/1/1/1
1	PTR	A	1008	1	-	0/9/11/13	0/1/1/1

All (3) bond length outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(Å)	Ideal(Å)
1	A	1008	PTR	O-C	19.63	1.24	1.11
1	A	1007	PTR	O-C	18.89	1.24	1.11
1	A	1008	PTR	CD1-CG	2.11	1.43	1.38

All (11) bond angle outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(°)	Ideal(°)
1	A	1008	PTR	C-CA-N	-9.12	104.72	113.83
1	A	1007	PTR	C-CA-N	-4.34	109.50	113.83
1	A	1007	PTR	OH-P-O1P	-3.31	95.47	108.78
1	A	1007	PTR	CD2-CG-CD1	2.81	122.72	118.13
1	A	1008	PTR	OH-CZ-CE2	-2.55	111.36	119.22
1	A	1008	PTR	O3P-P-O2P	2.46	116.60	107.38
1	A	1007	PTR	O3P-P-O2P	2.43	116.48	107.38
1	A	1007	PTR	CE1-CD1-CG	-2.27	117.78	121.04
1	A	1008	PTR	CD2-CG-CD1	2.16	121.66	118.13
1	A	1008	PTR	CB-CA-N	-2.06	103.87	112.80
1	A	1008	PTR	OH-CZ-CE1	2.02	125.42	119.22

There are no chirality outliers.

There are no torsion outliers.

There are no ring outliers.

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	GOL	A	2131	-	5,5,5	0.55	0	5,5,5	0.94	0
3	VVQ	A	2132	-	25,25,25	2.69	7 (28%)	34,36,36	3.86	20 (58%)

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	GOL	A	2131	-	-	0/4/4/4	0/0/0/0
3	VVQ	A	2132	-	-	1/6/12/12	0/3/3/3

All (7) bond length outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(Å)	Ideal(Å)
3	A	2132	VVQ	C1-C8	-8.80	1.37	1.48
3	A	2132	VVQ	C22-C2	-5.10	1.41	1.51
3	A	2132	VVQ	C9-C12	-4.64	1.44	1.50
3	A	2132	VVQ	C15-N20	4.20	1.40	1.34
3	A	2132	VVQ	C19-N18	4.00	1.41	1.35
3	A	2132	VVQ	C11-C15	-2.41	1.36	1.48
3	A	2132	VVQ	C5-CL	2.39	1.80	1.74

All (20) bond angle outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(°)	Ideal(°)
3	A	2132	VVQ	C15-N20-C19	10.39	121.10	116.30
3	A	2132	VVQ	C6-C1-C2	-9.70	112.82	119.06
3	A	2132	VVQ	O13-C12-N14	-6.90	112.73	122.59
3	A	2132	VVQ	C3-C4-C5	-6.32	112.49	119.22
3	A	2132	VVQ	N18-C19-N20	-5.72	119.98	125.85
3	A	2132	VVQ	C3-C2-C1	5.00	122.93	118.25
3	A	2132	VVQ	O13-C12-C9	4.59	125.88	120.26
3	A	2132	VVQ	C17-N18-C19	4.22	120.12	116.34
3	A	2132	VVQ	C17-C16-C15	3.57	120.56	117.20
3	A	2132	VVQ	C4-C5-CL	-3.57	113.41	119.34
3	A	2132	VVQ	C16-C17-N18	-3.32	120.21	123.88
3	A	2132	VVQ	C11-C15-N20	3.32	118.62	115.09
3	A	2132	VVQ	C16-C15-N20	-2.99	117.87	122.01
3	A	2132	VVQ	C6-C1-C8	2.98	122.92	118.64
3	A	2132	VVQ	C22-C2-C3	-2.95	114.33	120.33
3	A	2132	VVQ	C4-C5-C6	2.88	125.38	121.53
3	A	2132	VVQ	N21-C19-N20	2.34	121.11	117.19
3	A	2132	VVQ	C1-C8-N7	-2.28	117.77	121.32
3	A	2132	VVQ	C1-C6-C5	2.13	122.65	119.03
3	A	2132	VVQ	C8-C1-C2	2.12	124.01	119.01

There are no chirality outliers.

All (1) torsion outliers are listed below:

Mol	Chain	Res	Type	Atoms
3	A	2132	VVQ	O13-C12-C9-C8

There are no ring outliers.

5.7 Other polymers ⓘ

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 ⓘ

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	291/298 (97%)	0.21	15 (5%) 26 24	14, 24, 46, 77	0

All (15) RSRZ outliers are listed below:

Mol	Chain	Res	Type	RSRZ
1	A	860	PHE	5.2
1	A	839	ARG	4.6
1	A	920	ALA	3.6
1	A	1042	VAL	3.0
1	A	977	LEU	2.8
1	A	1003	GLN	2.7
1	A	1011	LYS	2.6
1	A	1004	ASP	2.5
1	A	922	ARG	2.5
1	A	1043	VAL	2.3
1	A	947	ARG	2.3
1	A	871	LEU	2.2
1	A	872	GLN	2.1
1	A	978	ALA	2.1
1	A	1047	LEU	2.0

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

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	RSR	LLDF	B-factors(\AA^2)	Q<0.9
1	PTR	A	1008	16/17	0.12	0.44	19,28,60,62	0
1	PTR	A	1007	16/17	0.12	-0.07	22,29,60,68	0

6.3 Carbohydrates

There are no carbohydrates in this entry.

6.4 Ligands

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	RSR	LLDF	B-factors(\AA^2)	Q<0.9
2	GOL	A	2131	6/6	0.18	1.49	36,40,42,43	6
3	VVQ	A	2132	23/23	0.07	-0.96	15,17,21,28	0

6.5 Other polymers

There are no such residues in this entry.