wwPDB X-ray Structure Validation Summary Report (i)

Jun 17, 2024 – 02:15 PM EDT

PDB ID	:	3BHU
Title	:	Structure of phosphorylated Thr160 CDK2/cyclin A in complex with the inhibitor meriolin 5
Authors	:	Echalier, A.; Bettayeb, K.; Ferandin, Y.; Lozach, O.; Clement, M.; Valette, A.; Liger, F.; Marquet, B.; Morris, J.C.; Endicott, J.A.; Joseph, B.; Meijer, L.
Deposited on	:	2007-11-29
Resolution	:	2.30 Å(reported)

This is a wwPDB X-ray Structure Validation Summary Report for a publicly released PDB entry.

We welcome your comments at *validation@mail.wwpdb.org*
A user guide is available at
https://www.wwpdb.org/validation/2017/XrayValidationReportHelp
with specific help available everywhere you see the (i) symbol.

The types of validation reports are described at
http://www.wwpdb.org/validation/2017/FAQs#types.

The following versions of software and data (see references (1)) were used in the production of this report:

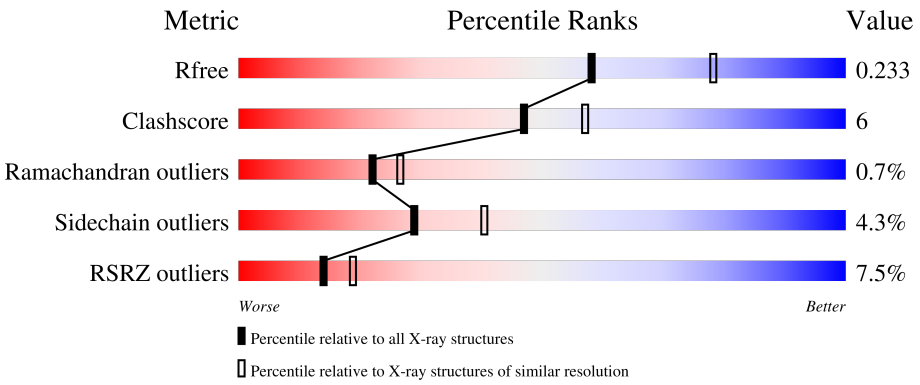
MolProbity	:	4.02b-467
Mogul	:	2022.3.0, CSD as543be (2022)
Xtriage (Phenix)	:	1.20.1
EDS	:	2.37.1
buster-report	:	1.1.7 (2018)
Percentile statistics	:	20191225.v01 (using entries in the PDB archive December 25th 2019)
Refmac	:	5.8.0158
CCP4	:	7.0.044 (Gargrove)
Ideal geometry (proteins)	:	Engh & Huber (2001)
Ideal geometry (DNA, RNA)	:	Parkinson et al. (1996)
Validation Pipeline (wwPDB-VP)	:	2.37.1

1 Overall quality at a glance (i)

The following experimental techniques were used to determine the structure:
X-RAY DIFFRACTION

The reported resolution of this entry is 2.30 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}	130704	5042 (2.30-2.30)
Clashscore	141614	5643 (2.30-2.30)
Ramachandran outliers	138981	5575 (2.30-2.30)
Sidechain outliers	138945	5575 (2.30-2.30)
RSRZ outliers	127900	4938 (2.30-2.30)

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 of the lower bar indicate the fraction of residues that contain outliers for >=3, 2, 1 and 0 types of geometric quality criteria respectively. 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 <=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	300	6% 80% 17% •••
1	C	300	10% 79% 10% •• 9%
2	B	262	5% 89% 10% •
2	D	262	8% 91% 7% •

2 Entry composition (i)

There are 5 unique types of molecules in this entry. The entry contains 9423 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 Cell division protein kinase 2.

Mol	Chain	Residues	Atoms						ZeroOcc	AltConf	Trace
			Total	C	N	O	P	S			
1	A	298	2430	1576	413	431	1	9	0	6	0
1	C	272	2198	1425	374	391	1	7	0	3	0

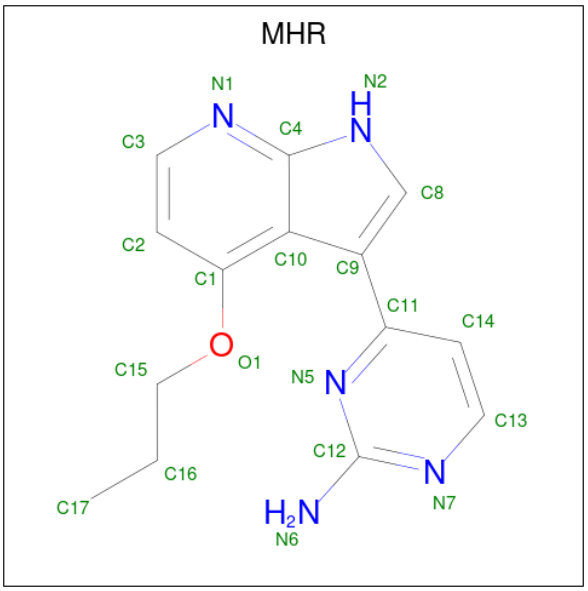
There are 4 discrepancies between the modelled and reference sequences:

Chain	Residue	Modelled	Actual	Comment	Reference
A	-1	GLY	-	EXPRESSION TAG	UNP P24941
A	0	SER	-	EXPRESSION TAG	UNP P24941
C	-1	GLY	-	EXPRESSION TAG	UNP P24941
C	0	SER	-	EXPRESSION TAG	UNP P24941

- Molecule 2 is a protein called Cyclin-A2.

Mol	Chain	Residues	Atoms					ZeroOcc	AltConf	Trace
			Total	C	N	O	S			
2	B	262	2136	1384	347	395	10	0	4	0
2	D	262	2123	1374	345	394	10	0	2	0

- Molecule 3 is 4-(4-propoxy-1H-pyrrolo[2,3-b]pyridin-3-yl)pyrimidin-2-amine (three-letter code: MHR) (formula: $C_{14}H_{15}N_5O$).



Mol	Chain	Residues	Atoms				ZeroOcc	AltConf
			Total	C	N	O		
3	A	1	20	14	5	1	0	0
			Total	C	N	O		
3	C	1	20	14	5	1	0	0

- Molecule 4 is MAGNESIUM ION (three-letter code: MG) (formula: Mg).

Mol	Chain	Residues	Atoms		ZeroOcc	AltConf
			Total	Mg		
4	B	1	1	1	0	0
			Total	Mg		
4	D	1	1	1	0	0

- Molecule 5 is water.

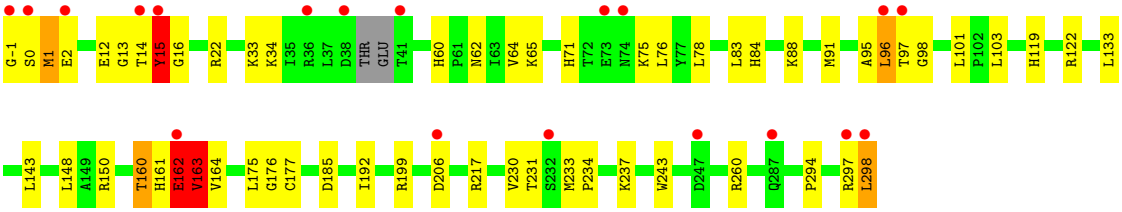
Mol	Chain	Residues	Atoms		ZeroOcc	AltConf
			Total	O		
5	A	176	176	176	0	0
			Total	O		
5	B	138	138	138	0	0
			Total	O		
5	C	90	90	90	0	0
			Total	O		
5	D	90	90	90	0	0

3 Residue-property plots (i)

These plots are drawn for all protein, RNA, DNA and oligosaccharide 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.

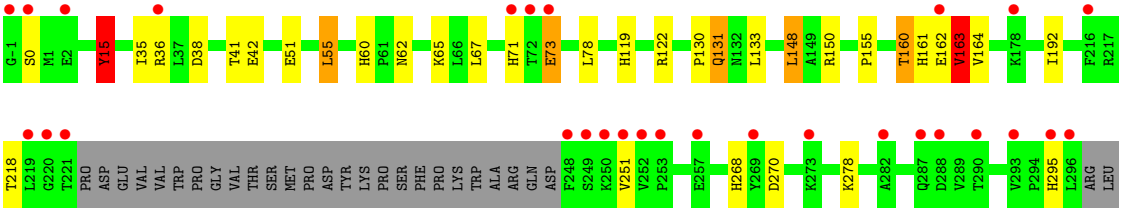
• Molecule 1: Cell division protein kinase 2

Chain A:



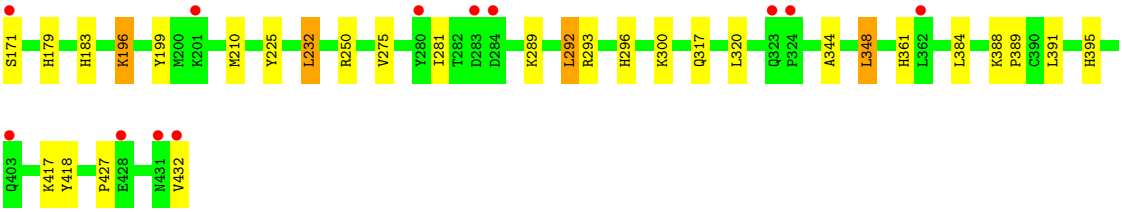
• Molecule 1: Cell division protein kinase 2

Chain C:



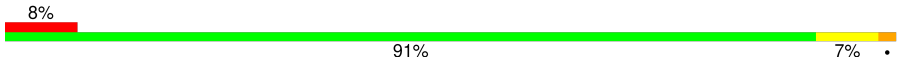
• Molecule 2: Cyclin-A2

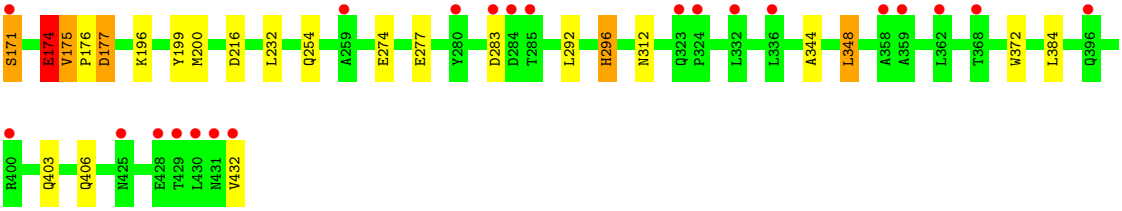
Chain B:



• Molecule 2: Cyclin-A2

Chain D:




S171
E174
V175
P176
D177
K196
Y199
M200
D216
L232
Q254
A259
E274
E277
Y280
D283
D284
T285
L292
H296
N312
Q323
P324
L332
L336
A344
L348
A358
A359
L362
T368
W372
L384
Q396
R400
Q403
Q406
M425
E428
T429
L430
M431
V432

4 Data and refinement statistics (i)

Property	Value	Source
Space group	P 21 21 21	Depositor
Cell constants a, b, c, α, β, γ	74.15Å 133.96Å 147.84Å 90.00° 90.00° 90.00°	Depositor
Resolution (Å)	20.00 – 2.30 19.89 – 2.30	Depositor EDS
% Data completeness (in resolution range)	99.2 (20.00-2.30) 99.2 (19.89-2.30)	Depositor EDS
R_{merge}	0.09	Depositor
R_{sym}	(Not available)	Depositor
$< I/\sigma(I) >$ [1]	2.69 (at 2.30Å)	Xtriage
Refinement program	REFMAC 5.2.0019	Depositor
R, R_{free}	0.194 , 0.240 0.190 , 0.233	Depositor DCC
R_{free} test set	3322 reflections (5.07%)	wwPDB-VP
Wilson B-factor (Å^2)	29.1	Xtriage
Anisotropy	0.263	Xtriage
Bulk solvent k_{sol}(e/Å^3), B_{sol}(Å^2)	0.39 , 33.3	EDS
L-test for twinning[2]	$< \|L\| > = 0.49$, $< L^2 > = 0.32$	Xtriage
Estimated twinning fraction	No twinning to report.	Xtriage
F_o,F_c correlation	0.93	EDS
Total number of atoms	9423	wwPDB-VP
Average B, all atoms (Å^2)	10.0	wwPDB-VP

Xtriage's analysis on translational NCS is as follows: *The largest off-origin peak in the Patterson function is 2.79% of the height of the origin peak. No significant pseudotranslation is detected.*

[1] Intensities estimated from amplitudes.

[2] Theoretical values of $< |L| >$, $< L^2 >$ for acentric reflections are 0.5, 0.333 respectively for untwinned datasets, and 0.375, 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: MHR, MG, TPO

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	0.57	0/2494	0.65	2/3380 (0.1%)
1	C	0.55	0/2245	0.62	1/3037 (0.0%)
2	B	0.47	0/2199	0.57	1/2989 (0.0%)
2	D	0.44	0/2179	0.64	3/2963 (0.1%)
All	All	0.51	0/9117	0.62	7/12369 (0.1%)

Chiral center outliers are detected by calculating the chiral volume of a chiral center and verifying if the center is modelled as a planar moiety or with the opposite hand.A planarity outlier is detected by checking planarity of atoms in a peptide group, atoms in a mainchain group or atoms of a sidechain that are expected to be planar.

Mol	Chain	#Chirality outliers	#Planarity outliers
1	C	0	1

There are no bond length outliers.

The worst 5 of 7 bond angle outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(^{o})	Ideal(^{o})
2	D	175	VAL	N-CA-C	-11.55	79.82	111.00
2	D	174	GLU	N-CA-C	7.36	130.86	111.00
2	B	232	LEU	CA-CB-CG	5.87	128.81	115.30
1	A	163	VAL	N-CA-C	-5.71	95.58	111.00
1	C	163	VAL	N-CA-C	5.26	125.21	111.00

There are no chirality outliers.

All (1) planarity outliers are listed below:

Mol	Chain	Res	Type	Group
1	C	15	TYR	Peptide

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	2430	0	2480	51	0
1	C	2198	0	2258	32	0
2	B	2136	0	2160	17	0
2	D	2123	0	2142	17	0
3	A	20	0	15	2	0
3	C	20	0	15	0	0
4	B	1	0	0	0	0
4	D	1	0	0	0	0
5	A	176	0	0	7	0
5	B	138	0	0	2	0
5	C	90	0	0	2	0
5	D	90	0	0	3	0
All	All	9423	0	9070	111	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 6.

The worst 5 of 111 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:15:TYR:CB	1:A:16:GLY:HA3	1.70	1.20
1:A:15:TYR:HB2	1:A:16:GLY:CA	1.72	1.18
1:A:162:GLU:HB2	1:A:163:VAL:HA	1.27	1.12
1:A:177[A]:CYS:SG	1:A:233:MET:HE3	2.07	0.95
1:C:15:TYR:HE1	1:C:35:ILE:CD1	1.81	0.93

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	299/300 (100%)	289 (97%)	5 (2%)	5 (2%)	9	8
1	C	270/300 (90%)	256 (95%)	11 (4%)	3 (1%)	14	15
2	B	264/262 (101%)	262 (99%)	2 (1%)	0	100	100
2	D	262/262 (100%)	257 (98%)	5 (2%)	0	100	100
All	All	1095/1124 (97%)	1064 (97%)	23 (2%)	8 (1%)	22	26

5 of 8 Ramachandran outliers are listed below:

Mol	Chain	Res	Type
1	A	162	GLU
1	A	1	MET
1	A	164	VAL
1	C	164	VAL
1	A	15	TYR

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	267/263 (102%)	253 (95%)	14 (5%)	23	32
1	C	240/263 (91%)	231 (96%)	9 (4%)	33	47
2	B	239/235 (102%)	232 (97%)	7 (3%)	42	58
2	D	237/235 (101%)	224 (94%)	13 (6%)	21	30
All	All	983/996 (99%)	940 (96%)	43 (4%)	29	39

5 of 43 residues with a non-rotameric sidechain are listed below:

Mol	Chain	Res	Type
1	C	148	LEU
2	D	232	LEU
1	C	150	ARG

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Mol	Chain	Res	Type
2	D	177	ASP
2	D	292	LEU

Sometimes sidechains can be flipped to improve hydrogen bonding and reduce clashes. 5 of 26 such sidechains are listed below:

Mol	Chain	Res	Type
1	C	119	HIS
1	C	295	HIS
2	D	406	GLN
1	C	287	GLN
2	D	173	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	$\#\|Z\| > 2$
1	TPO	C	160	1	8,10,11	2.32	3 (37%)	10,14,16	1.35	2 (20%)
1	TPO	A	160	1	8,10,11	0.80	0	10,14,16	1.08	1 (10%)

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	TPO	C	160	1	-	1/9/11/13	-

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Mol	Type	Chain	Res	Link	Chirals	Torsions	Rings
1	TPO	A	160	1	-	0/9/11/13	-

All (3) bond length outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(Å)	Ideal(Å)
1	C	160	TPO	P-O3P	-3.86	1.40	1.54
1	C	160	TPO	P-O2P	-3.50	1.41	1.54
1	C	160	TPO	P-O1P	-3.20	1.40	1.50

All (3) bond angle outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(^{o})	Ideal(^{o})
1	C	160	TPO	O3P-P-O2P	2.96	118.91	107.80
1	C	160	TPO	O3P-P-OG1	-2.19	97.33	105.85
1	A	160	TPO	O3P-P-O2P	2.05	115.51	107.80

There are no chirality outliers.

All (1) torsion outliers are listed below:

Mol	Chain	Res	Type	Atoms
1	C	160	TPO	O-C-CA-CB

There are no ring outliers.

2 monomers are involved in 4 short contacts:

Mol	Chain	Res	Type	Clashes	Symm-Clashes
1	C	160	TPO	2	0
1	A	160	TPO	2	0

5.5 Carbohydrates (i)

There are no monosaccharides in this entry.

5.6 Ligand geometry (i)

Of 4 ligands modelled in this entry, 2 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	$\#\|Z\| > 2$
3	MHR	C	299	-	21,22,22	1.50	3 (14%)	18,30,30	2.71	9 (50%)
3	MHR	A	299	-	21,22,22	1.52	3 (14%)	18,30,30	2.68	9 (50%)

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	MHR	C	299	-	-	3/4/8/8	0/3/3/3
3	MHR	A	299	-	-	3/4/8/8	0/3/3/3

The worst 5 of 6 bond length outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(Å)	Ideal(Å)
3	C	299	MHR	C8-C9	4.32	1.51	1.40
3	A	299	MHR	C8-C9	4.30	1.51	1.40
3	C	299	MHR	C1-C10	3.59	1.49	1.41
3	A	299	MHR	C1-C10	3.37	1.49	1.41
3	A	299	MHR	C14-C13	2.34	1.39	1.36

The worst 5 of 18 bond angle outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(^{o})	Ideal(^{o})
3	A	299	MHR	N7-C12-N5	-5.26	119.89	125.69
3	C	299	MHR	C13-N7-C12	5.21	121.39	116.21
3	C	299	MHR	N7-C12-N5	-4.98	120.20	125.69
3	A	299	MHR	C13-N7-C12	4.59	120.78	116.21
3	C	299	MHR	C14-C13-N7	-4.34	119.58	123.83

There are no chirality outliers.

5 of 6 torsion outliers are listed below:

Mol	Chain	Res	Type	Atoms
3	C	299	MHR	C10-C1-O1-C15
3	C	299	MHR	O1-C15-C16-C17

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Mol	Chain	Res	Type	Atoms
3	C	299	MHR	C2-C1-O1-C15
3	A	299	MHR	C10-C1-O1-C15
3	A	299	MHR	C2-C1-O1-C15

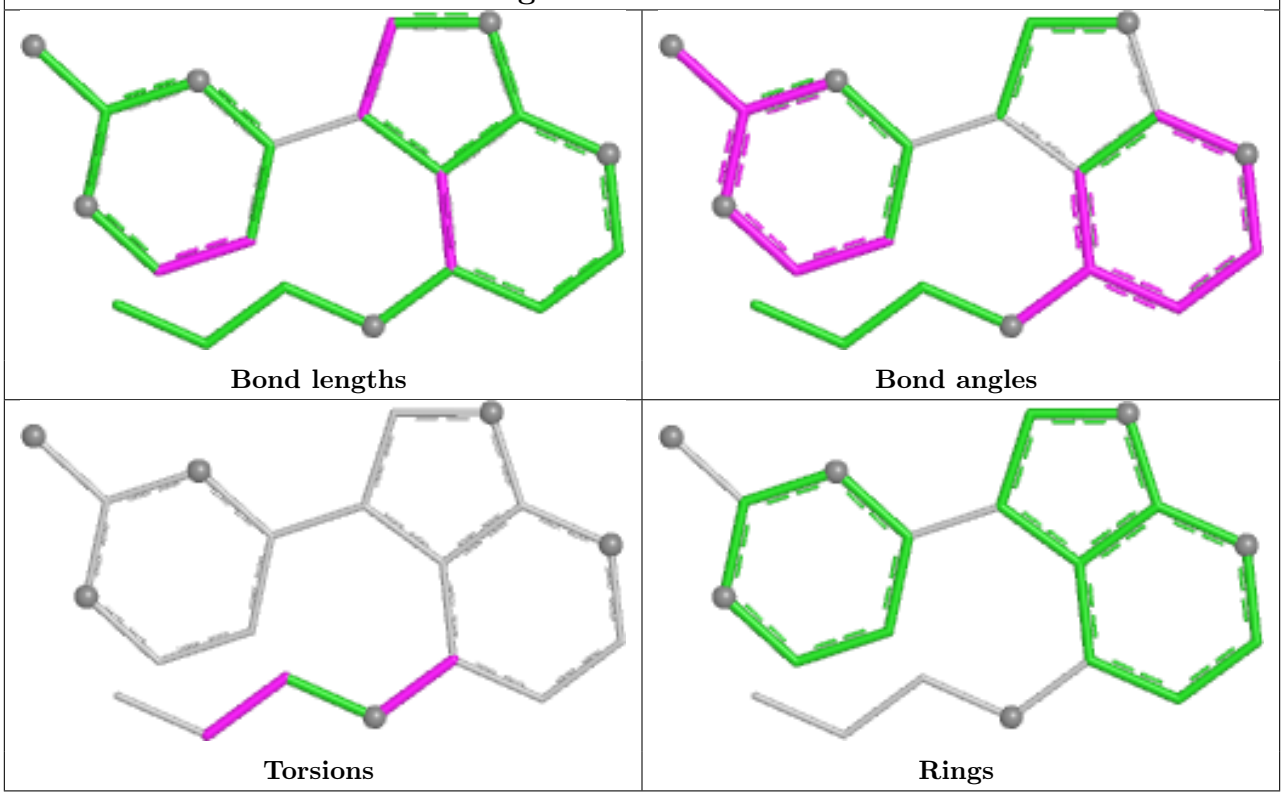
There are no ring outliers.

1 monomer is involved in 2 short contacts:

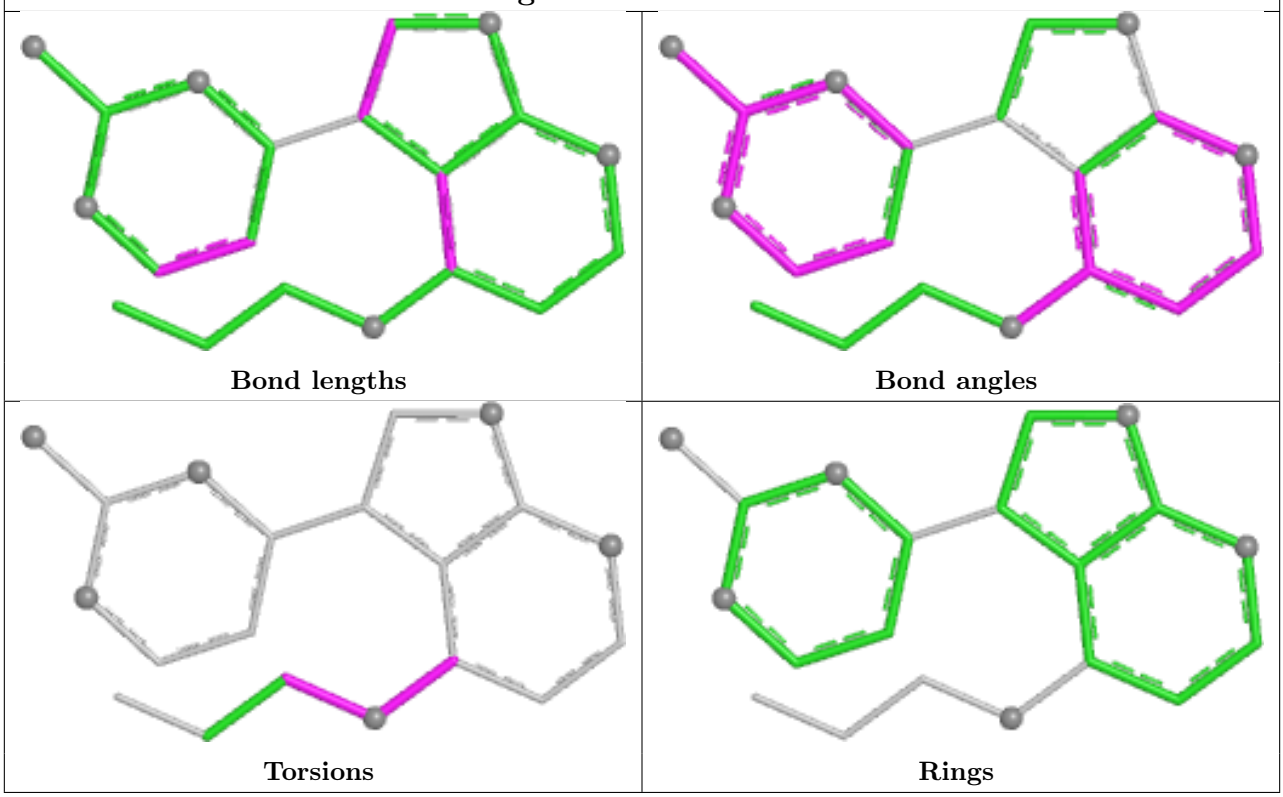
Mol	Chain	Res	Type	Clashes	Symm-Clashes
3	A	299	MHR	2	0

The following is a two-dimensional graphical depiction of Mogul quality analysis of bond lengths, bond angles, torsion angles, and ring geometry for all instances of the Ligand of Interest. In addition, ligands with molecular weight > 250 and outliers as shown on the validation Tables will also be included. For torsion angles, if less then 5% of the Mogul distribution of torsion angles is within 10 degrees of the torsion angle in question, then that torsion angle is considered an outlier. Any bond that is central to one or more torsion angles identified as an outlier by Mogul will be highlighted in the graph. For rings, the root-mean-square deviation (RMSD) between the ring in question and similar rings identified by Mogul is calculated over all ring torsion angles. If the average RMSD is greater than 60 degrees and the minimal RMSD between the ring in question and any Mogul-identified rings is also greater than 60 degrees, then that ring is considered an outlier. The outliers are highlighted in purple. The color gray indicates Mogul did not find sufficient equivalents in the CSD to analyse the geometry.

Ligand MHR C 299



Ligand MHR A 299



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, 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	297/300 (99%)	0.22	19 (6%)	19	25	2, 7, 23, 32	0
1	C	271/300 (90%)	0.47	29 (10%)	6	8	2, 9, 34, 60	0
2	B	262/262 (100%)	0.12	12 (4%)	32	39	2, 8, 18, 30	0
2	D	262/262 (100%)	0.35	22 (8%)	11	15	4, 9, 22, 46	0
All	All	1092/1124 (97%)	0.29	82 (7%)	14	19	2, 8, 25, 60	0

The worst 5 of 82 RSRZ outliers are listed below:

Mol	Chain	Res	Type	RSRZ
1	C	251	VAL	7.2
1	C	249	SER	7.0
1	C	221	THR	6.5
2	D	432	VAL	6.1
2	D	284	ASP	5.6

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. 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	B-factors(Å^2)	Q<0.9
1	TPO	A	160	11/12	0.98	0.10	4,8,10,10	0
1	TPO	C	160	11/12	0.98	0.08	8,11,14,14	0

6.3 Carbohydrates (i)

There are no monosaccharides in this entry.

6.4 Ligands (i)

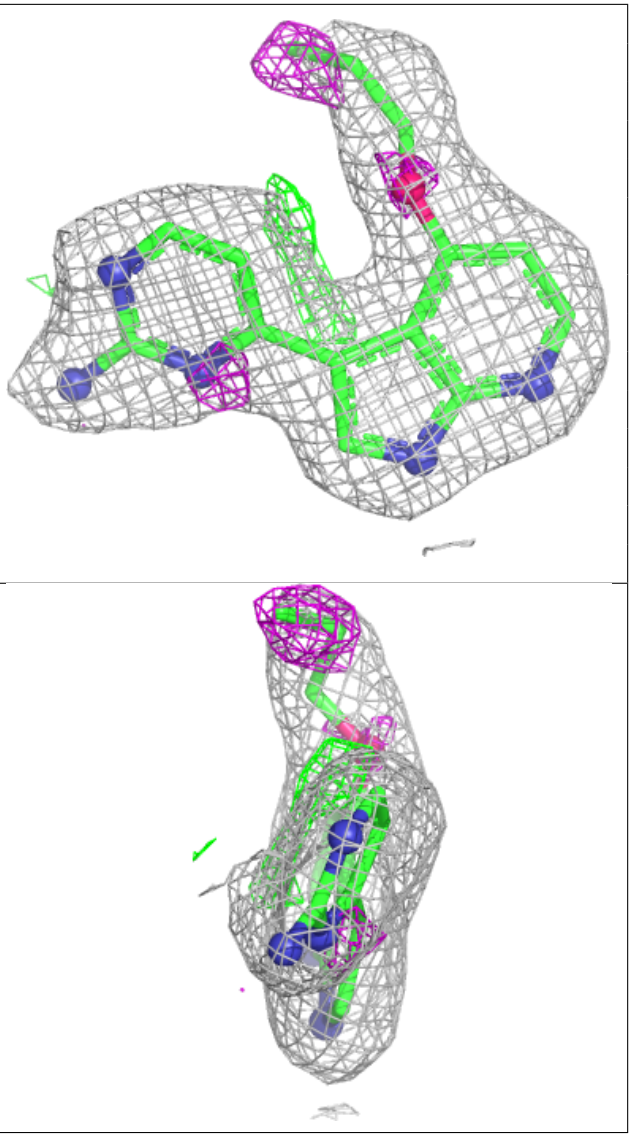
In the following table, the Atoms column lists the number of modelled atoms in the group and the number defined in the chemical component dictionary. The B-factors column lists the minimum, median, 95th percentile and maximum values of B factors of atoms in the group. The column labelled 'Q< 0.9' lists the number of atoms with occupancy less than 0.9.

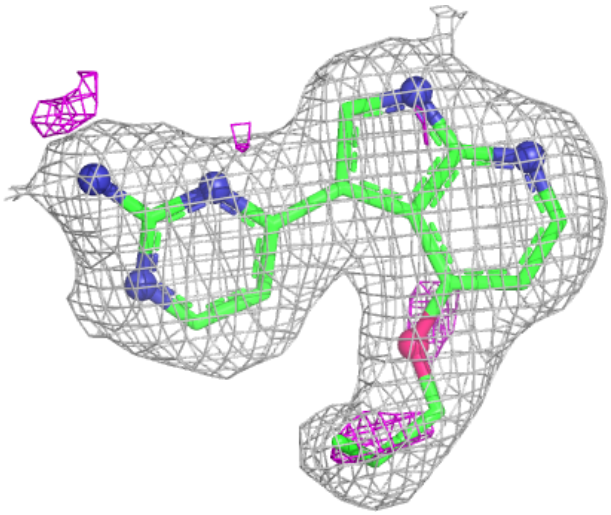
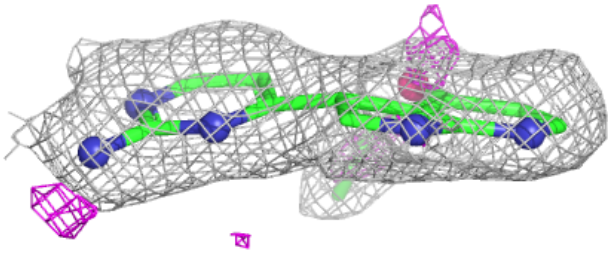
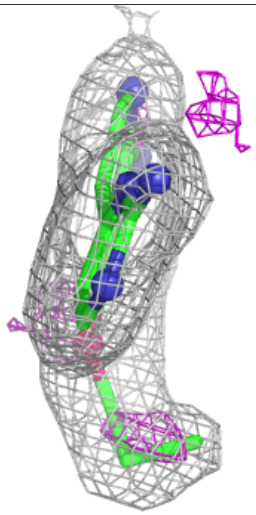
Mol	Type	Chain	Res	Atoms	RSCC	RSR	B-factors(Å^2)	Q<0.9
4	MG	D	1	1/1	0.61	0.28	15,15,15,15	0
4	MG	B	1	1/1	0.82	0.16	19,19,19,19	0
3	MHR	C	299	20/20	0.93	0.13	3,5,11,12	0
3	MHR	A	299	20/20	0.95	0.11	2,2,2,3	0

The following is a graphical depiction of the model fit to experimental electron density of all instances of the Ligand of Interest. In addition, ligands with molecular weight > 250 and outliers as shown on the geometry validation Tables will also be included. Each fit is shown from different orientation to approximate a three-dimensional view.

Electron density around MHR C 299:

$2mF_o$-DF_c (at 0.7 rmsd) in gray
mF_o-DF_c (at 3 rmsd) in purple (negative)
and green (positive)



Electron density around MHR A 299: 2mF$_o$-DF$_c$ (at 0.7 rmsd) in gray mF$_o$-DF$_c$ (at 3 rmsd) in purple (negative) and green (positive)	
	

6.5 Other polymers (i)

There are no such residues in this entry.