wwPDB X-ray Structure Validation Summary Report (i)

Sep 17, 2023 – 03:16 AM EDT

PDB ID	:	4PL3
Title	:	Crystal structure of murine IRE1 in complex with MKC9989 inhibitor
Authors	:	Sanches, M.; Duffy, N.; Talukdar, M.; Thevakumaran, N.; Chiovitti, D.; Alawar, R.; Patterson, J.B.; Sicheri, F.
Deposited on	:	2014-05-16
Resolution	:	2.90 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 (i)) were used in the production of this report:

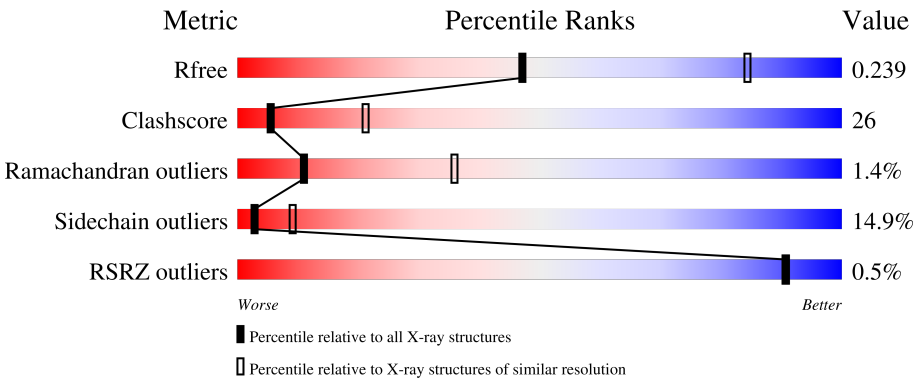
MolProbity	:	4.02b-467
Mogul	:	1.8.5 (274361), CSD as541be (2020)
Xtriage (Phenix)	:	1.13
EDS	:	2.35.1
buster-report	:	1.1.7 (2018)
Percentile statistics	:	20191225.v01 (using entries in the PDB archive December 25th 2019)
Refmac	:	5.8.0158
CCP4	:	7.0.044 (Gargrove)
Ideal geometry (proteins)	:	Engh & Huber (2001)
Ideal geometry (DNA, RNA)	:	Parkinson et al. (1996)
Validation Pipeline (wwPDB-VP)	:	2.35.1

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 2.90 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	1957 (2.90-2.90)
Clashscore	141614	2172 (2.90-2.90)
Ramachandran outliers	138981	2115 (2.90-2.90)
Sidechain outliers	138945	2117 (2.90-2.90)
RSRZ outliers	127900	1906 (2.90-2.90)

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	435	46% 36% 7% 11%
1	B	435	% 43% 37% 7% 12%

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	31J	A	1001	-	-	X	-

2 Entry composition (i)

There are 4 unique types of molecules in this entry. The entry contains 12482 atoms, of which 6152 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.

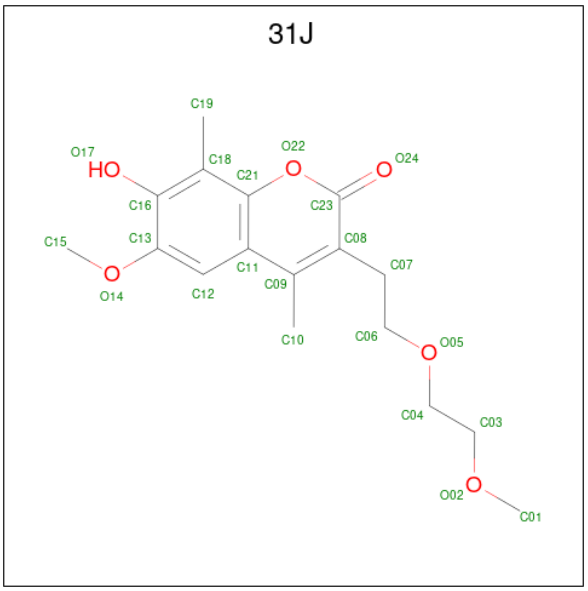
- Molecule 1 is a protein called Serine/threonine-protein kinase/endoribonuclease IRE1.

Mol	Chain	Residues	Atoms						ZeroOcc	AltConf	Trace
			Total	C	H	N	O	S			
1	A	388	6201	2003	3072	541	566	19	0	0	0
1	B	383	6155	1984	3056	541	555	19	0	0	0

There are 16 discrepancies between the modelled and reference sequences:

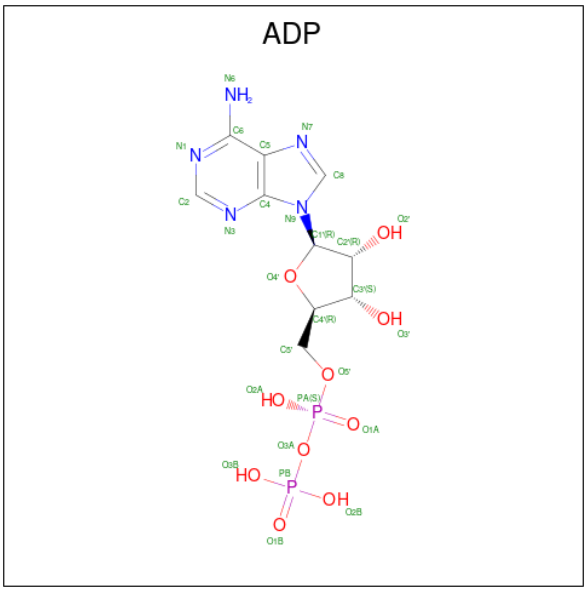
Chain	Residue	Modelled	Actual	Comment	Reference
A	543	GLY	-	expression tag	UNP Q9EQY0
A	544	ALA	-	expression tag	UNP Q9EQY0
A	545	MET	-	expression tag	UNP Q9EQY0
A	546	ASP	-	expression tag	UNP Q9EQY0
A	547	PRO	-	expression tag	UNP Q9EQY0
A	548	GLU	-	expression tag	UNP Q9EQY0
A	549	PHE	-	expression tag	UNP Q9EQY0
A	772	TYR	ASN	engineered mutation	UNP Q9EQY0
B	543	GLY	-	expression tag	UNP Q9EQY0
B	544	ALA	-	expression tag	UNP Q9EQY0
B	545	MET	-	expression tag	UNP Q9EQY0
B	546	ASP	-	expression tag	UNP Q9EQY0
B	547	PRO	-	expression tag	UNP Q9EQY0
B	548	GLU	-	expression tag	UNP Q9EQY0
B	549	PHE	-	expression tag	UNP Q9EQY0
B	772	TYR	ASN	engineered mutation	UNP Q9EQY0

- Molecule 2 is 7-hydroxy-6-methoxy-3-[2-(2-methoxyethoxy)ethyl]-4,8-dimethyl-2H-chromen-2-one (three-letter code: 31J) (formula: $C_{17}H_{22}O_6$).



Mol	Chain	Residues	Atoms			ZeroOcc	AltConf
			Total	C	O		
2	A	1	23	17	6	0	0
			Total	C	O		
2	B	1	23	17	6	0	0

- Molecule 3 is ADENOSINE-5'-DIPHOSPHATE (three-letter code: ADP) (formula: $C_{10}H_{15}N_5O_{10}P_2$).



Mol	Chain	Residues	Atoms						ZeroOcc	AltConf
			Total	C	H	N	O	P		
3	A	1	39	10	12	5	10	2	0	0

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Mol	Chain	Residues	Atoms						ZeroOcc	AltConf
			Total	C	H	N	O	P		
3	B	1	39	10	12	5	10	2	0	0

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

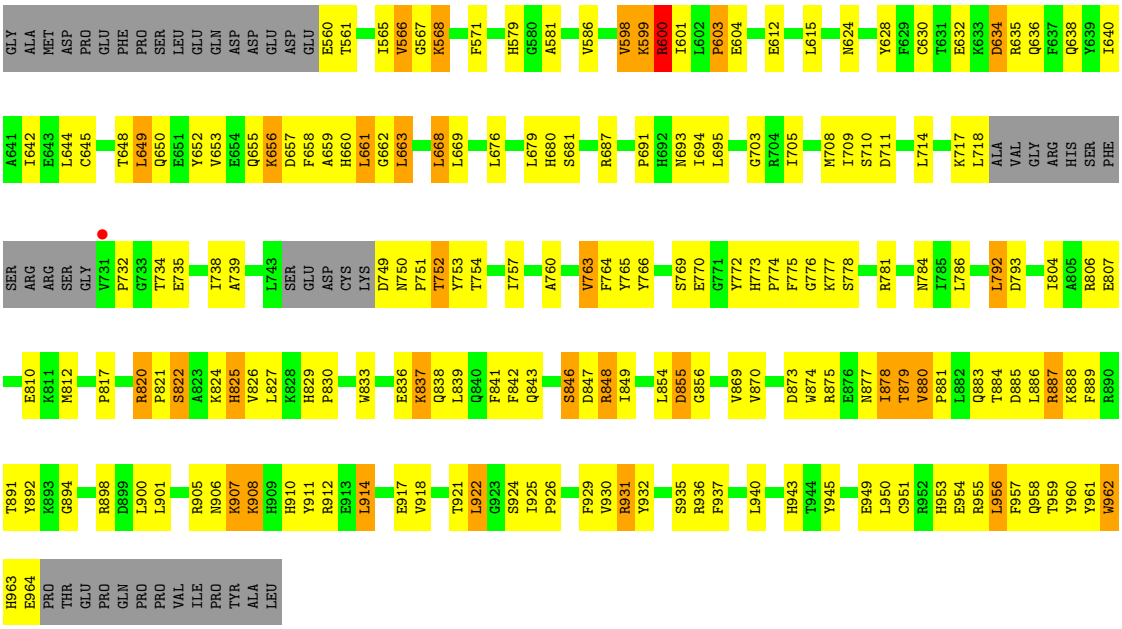
Mol	Chain	Residues	Atoms		ZeroOcc	AltConf
			Total	Mg		
4	A	1	1	1	0	0
			Total	Mg		
4	B	1	1	1	0	0

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.

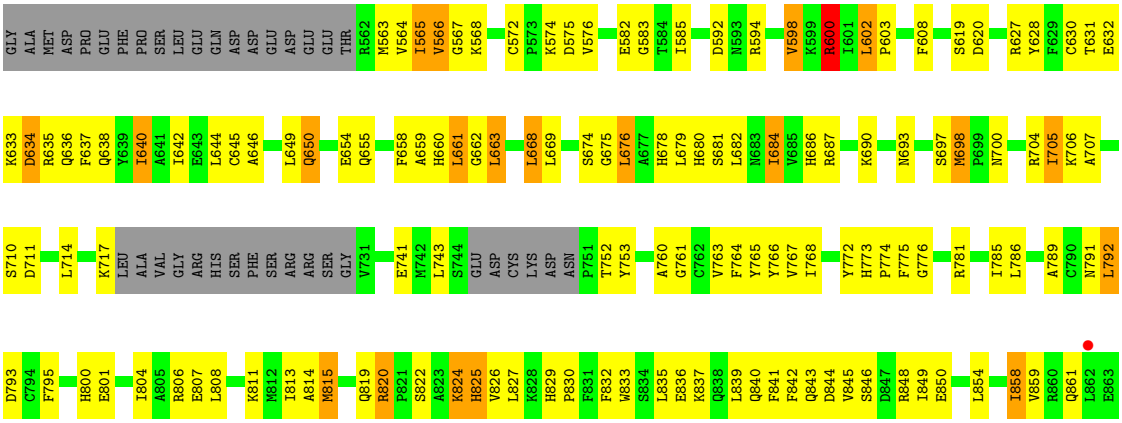
• Molecule 1: Serine/threonine-protein kinase/endoribonuclease IRE1

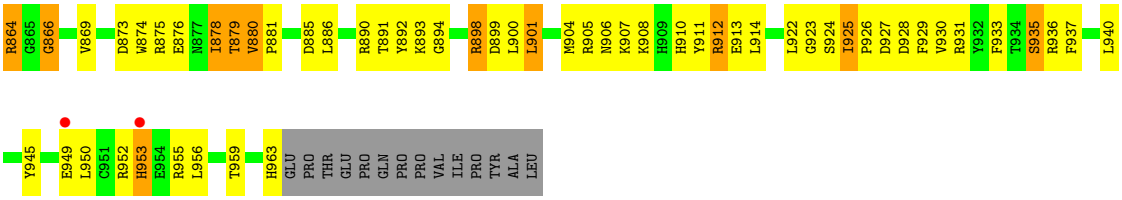
Chain A:



• Molecule 1: Serine/threonine-protein kinase/endoribonuclease IRE1

Chain B:




R864
G865
G866
V869
D873
W874
R875
E876
N877
I878
T879
V880
P881
D885
L886
R890
T891
Y892
K893
G894
R898
D899
L900
L901
M904
R905
N906
K907
K908
H909
H910
Y911
R912
E913
L914
L922
G923
S924
I925
P926
D927
D928
F929
V930
R931
Y932
F933
T934
S935
R936
F937
L940
Y945
E949
L950
C951
R952
H953
E954
R955
L956
T959
H963
GLU
PRO
THR
GLU
PRO
GLN
PRO
PRO
VAL
ILE
PRO
TYR
ALA
LEU

4 Data and refinement statistics (i)

Property	Value	Source
Space group	C 1 2 1	Depositor
Cell constants a, b, c, α, β, γ	246.08Å 90.50Å 72.13Å 90.00° 91.88° 90.00°	Depositor
Resolution (Å)	72.09 – 2.90 72.09 – 2.90	Depositor EDS
% Data completeness (in resolution range)	61.9 (72.09-2.90) 62.1 (72.09-2.90)	Depositor EDS
R_{merge}	0.13	Depositor
R_{sym}	(Not available)	Depositor
$< I/\sigma(I) >$ [1]	4.10 (at 2.91Å)	Xtriage
Refinement program	PHENIX (phenix.refine: dev_1175)	Depositor
R, R_{free}	0.201 , 0.234 0.206 , 0.239	Depositor DCC
R_{free} test set	458 reflections (2.09%)	wwPDB-VP
Wilson B-factor (Å^2)	38.8	Xtriage
Anisotropy	0.026	Xtriage
Bulk solvent k_{sol}(e/Å^3), B_{sol}(Å^2)	0.34 , 42.3	EDS
L-test for twinning[2]	$< \|L\| > = 0.47$, $< L^2 > = 0.29$	Xtriage
Estimated twinning fraction	0.028 for -h,-k,l	Xtriage
F_o,F_c correlation	0.86	EDS
Total number of atoms	12482	wwPDB-VP
Average B, all atoms (Å^2)	49.0	wwPDB-VP

Xtriage's analysis on translational NCS is as follows: *The largest off-origin peak in the Patterson function is 4.34% 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: 31J, ADP, MG

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	0.55	0/3206	0.70	1/4336 (0.0%)
1	B	0.51	0/3177	0.67	1/4294 (0.0%)
All	All	0.53	0/6383	0.69	2/8630 (0.0%)

There are no bond length outliers.

All (2) bond angle outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(^{o})	Ideal(^{o})
1	A	600	ARG	NE-CZ-NH1	7.42	124.01	120.30
1	B	600	ARG	NE-CZ-NH1	6.16	123.38	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	3129	3072	3054	154	0
1	B	3099	3056	3038	165	2
2	A	23	0	20	7	2
2	B	23	0	19	5	0
3	A	27	12	12	5	0
3	B	27	12	12	2	0
4	A	1	0	0	0	0

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Mol	Chain	Non-H	H(model)	H(added)	Clashes	Symm-Clashes
4	B	1	0	0	0	0
All	All	6330	6152	6155	324	2

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 26.

The worst 5 of 324 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:843:GLN:OE1	1:B:908:LYS:NZ	1.98	0.97
1:A:892:TYR:OH	2:A:1001:31J:O17	1.98	0.82
1:A:910:HIS:NE2	2:A:1001:31J:H13	1.95	0.82
1:A:932:TYR:O	1:A:936:ARG:NH1	2.15	0.80
1:A:829:HIS:CG	1:A:830:PRO:HD2	2.17	0.80

All (2) symmetry-related close contacts are listed below. The label for Atom-2 includes the symmetry operator and encoded unit-cell translations to be applied.

Atom-1	Atom-2	Interatomic distance (Å)	Clash overlap (Å)
1:B:864:ARG:HH12	2:A:1001:31J:O24[4_545]	1.43	0.17
1:B:864:ARG:NH1	2:A:1001:31J:O24[4_545]	2.05	0.15

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	382/435 (88%)	321 (84%)	54 (14%)	7 (2%)	8 29
1	B	377/435 (87%)	329 (87%)	44 (12%)	4 (1%)	14 42
All	All	759/870 (87%)	650 (86%)	98 (13%)	11 (1%)	11 36

5 of 11 Ramachandran outliers are listed below:

Mol	Chain	Res	Type
1	A	581	ALA
1	A	822	SER
1	B	925	ILE
1	B	592	ASP
1	A	656	LYS

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	337/387 (87%)	290 (86%)	47 (14%)	3 10
1	B	335/387 (87%)	282 (84%)	53 (16%)	2 8
All	All	672/774 (87%)	572 (85%)	100 (15%)	3 9

5 of 100 residues with a non-rotameric sidechain are listed below:

Mol	Chain	Res	Type
1	B	630	CYS
1	B	705	ILE
1	B	956	LEU
1	B	636	GLN
1	B	663	LEU

Sometimes sidechains can be flipped to improve hydrogen bonding and reduce clashes. All (4) such sidechains are listed below:

Mol	Chain	Res	Type
1	A	624	ASN
1	A	693	ASN
1	A	773	HIS
1	B	693	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)

There are no non-standard protein/DNA/RNA residues in this entry.

5.5 Carbohydrates (i)

There are no monosaccharides in this entry.

5.6 Ligand geometry (i)

Of 6 ligands modelled in this entry, 2 are monoatomic - leaving 4 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	ADP	A	1002	4	24,29,29	1.13	3 (12%)	29,45,45	1.45	5 (17%)
2	31J	B	1001	1	24,24,24	1.63	4 (16%)	28,33,33	2.94	12 (42%)
3	ADP	B	1002	4	24,29,29	0.99	1 (4%)	29,45,45	1.64	5 (17%)
2	31J	A	1001	1	24,24,24	1.56	1 (4%)	28,33,33	2.64	12 (42%)

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	ADP	A	1002	4	-	5/12/32/32	0/3/3/3
2	31J	B	1001	1	-	1/9/9/9	0/2/2/2
3	ADP	B	1002	4	-	4/12/32/32	0/3/3/3
2	31J	A	1001	1	-	2/9/9/9	0/2/2/2

The worst 5 of 9 bond length outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(Å)	Ideal(Å)
2	A	1001	31J	C08-C09	6.80	1.47	1.35

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Mol	Chain	Res	Type	Atoms	Z	Observed(Å)	Ideal(Å)
2	B	1001	31J	C08-C09	6.28	1.46	1.35
3	A	1002	ADP	O4'-C1'	2.56	1.44	1.41
3	B	1002	ADP	C5-C4	2.56	1.47	1.40
2	B	1001	31J	C13-C16	-2.41	1.37	1.40

The worst 5 of 34 bond angle outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(^{o})	Ideal(^{o})
2	A	1001	31J	O14-C13-C16	7.45	122.06	114.54
2	B	1001	31J	O14-C13-C16	6.27	120.86	114.54
2	B	1001	31J	C11-C21-C18	-6.14	118.86	123.22
2	A	1001	31J	C11-C21-C18	-6.08	118.90	123.22
2	B	1001	31J	O22-C21-C18	5.35	121.37	114.84

There are no chirality outliers.

5 of 12 torsion outliers are listed below:

Mol	Chain	Res	Type	Atoms
3	A	1002	ADP	C5'-O5'-PA-O3A
3	A	1002	ADP	O4'-C4'-C5'-O5'
3	B	1002	ADP	O4'-C4'-C5'-O5'
3	B	1002	ADP	C3'-C4'-C5'-O5'
3	A	1002	ADP	C3'-C4'-C5'-O5'

There are no ring outliers.

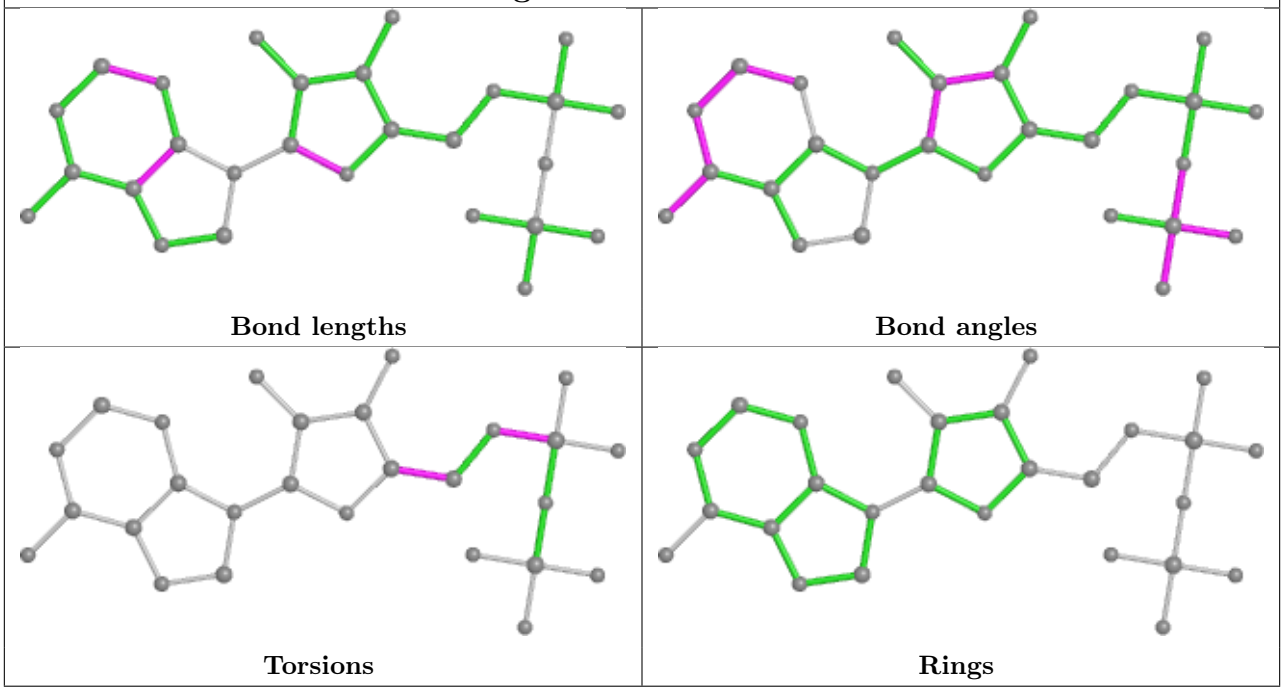
4 monomers are involved in 21 short contacts:

Mol	Chain	Res	Type	Clashes	Symm-Clashes
3	A	1002	ADP	5	0
2	B	1001	31J	5	0
3	B	1002	ADP	2	0
2	A	1001	31J	7	2

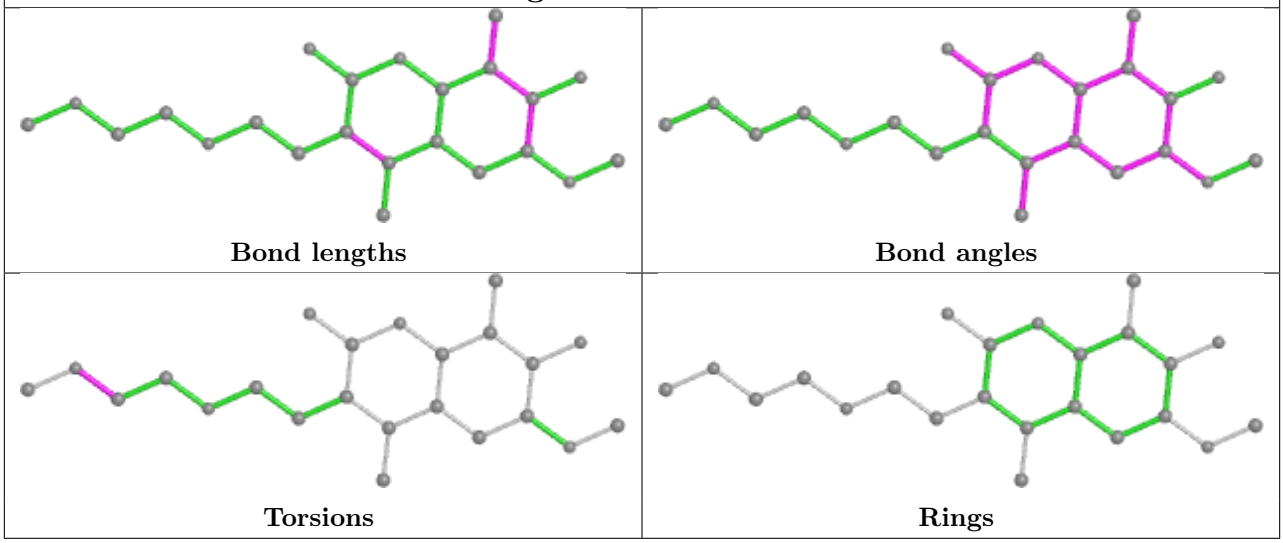
The following is a two-dimensional graphical depiction of Mogul quality analysis of bond lengths, bond angles, torsion angles, and ring geometry for all instances of the Ligand of Interest. In addition, ligands with molecular weight > 250 and outliers as shown on the validation Tables will also be included. 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.

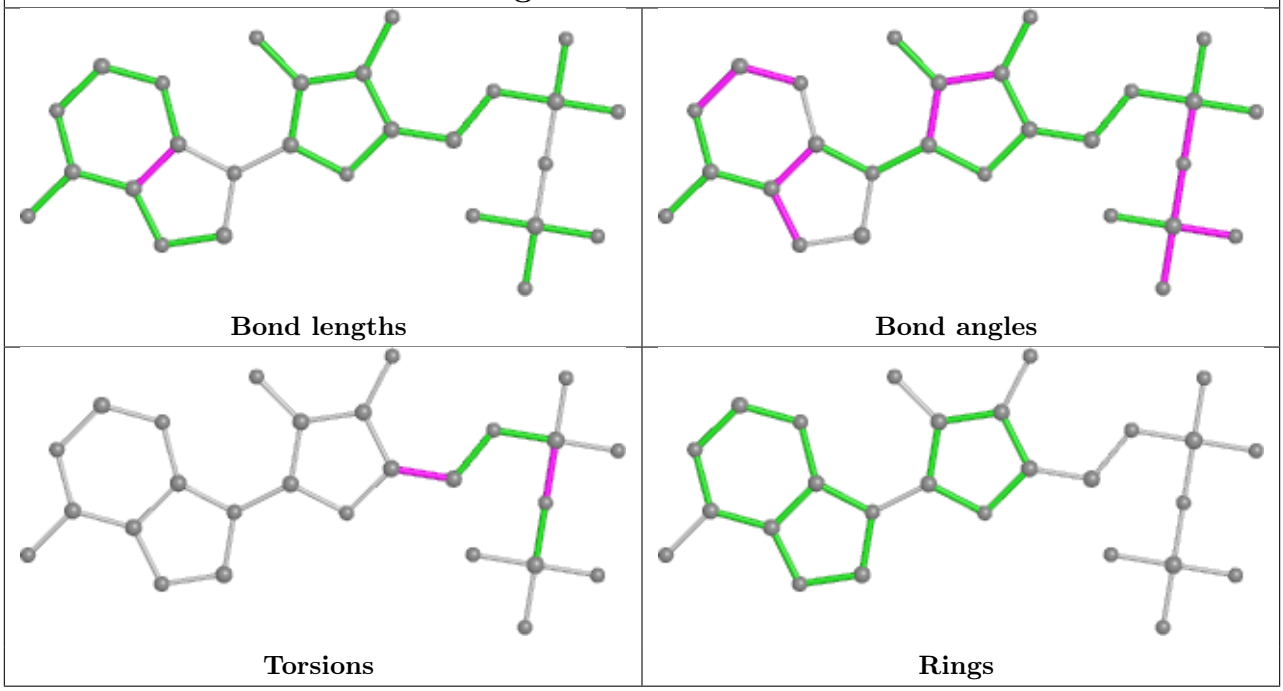
Ligand ADP A 1002



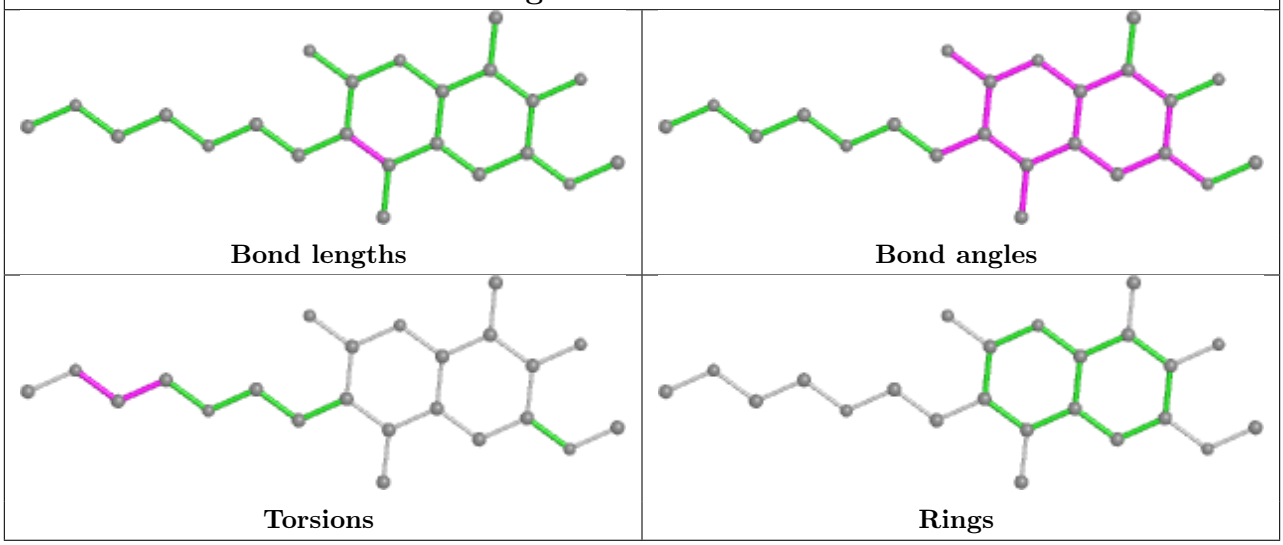
Ligand 31J B 1001



Ligand ADP B 1002



Ligand 31J A 1001



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	388/435 (89%)	-0.19	1 (0%)	94	94	16, 40, 84, 105	0
1	B	383/435 (88%)	-0.18	3 (0%)	86	86	18, 42, 78, 112	0
All	All	771/870 (88%)	-0.18	4 (0%)	91	91	16, 41, 82, 112	0

All (4) RSRZ outliers are listed below:

Mol	Chain	Res	Type	RSRZ
1	B	953	HIS	2.6
1	A	731	VAL	2.4
1	B	862	LEU	2.3
1	B	949	GLU	2.3

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 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	A	1003	1/1	0.89	0.12	31,31,31,31	0

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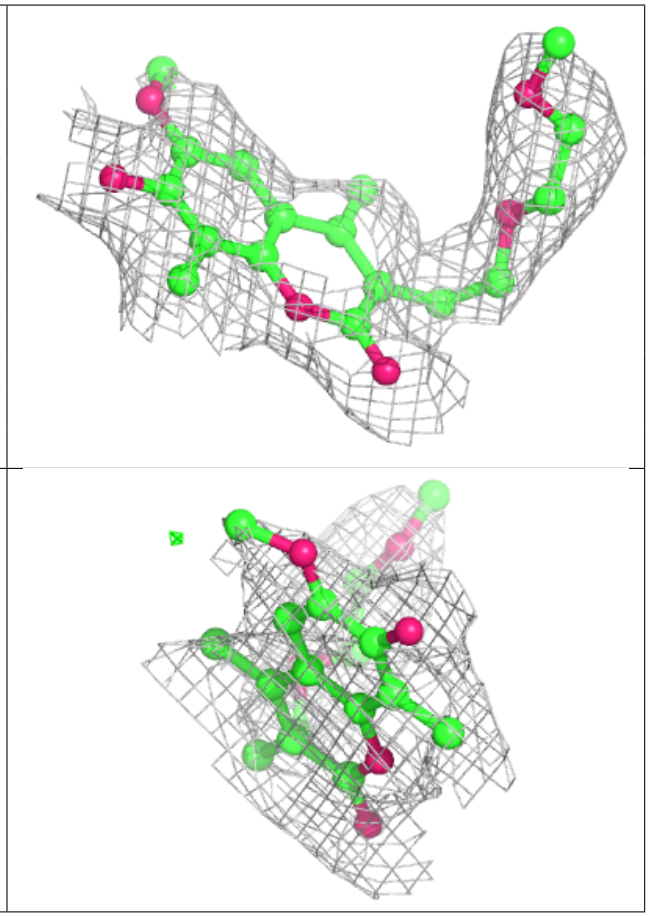
Mol	Type	Chain	Res	Atoms	RSCC	RSR	B-factors(Å^2)	Q<0.9
2	31J	A	1001	23/23	0.94	0.16	13,78,83,91	0
2	31J	B	1001	23/23	0.95	0.15	11,45,59,69	0
3	ADP	A	1002	27/27	0.97	0.16	20,20,24,24	0
4	MG	B	1003	1/1	0.97	0.09	59,59,59,59	0
3	ADP	B	1002	27/27	0.98	0.17	10,37,71,89	0

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.

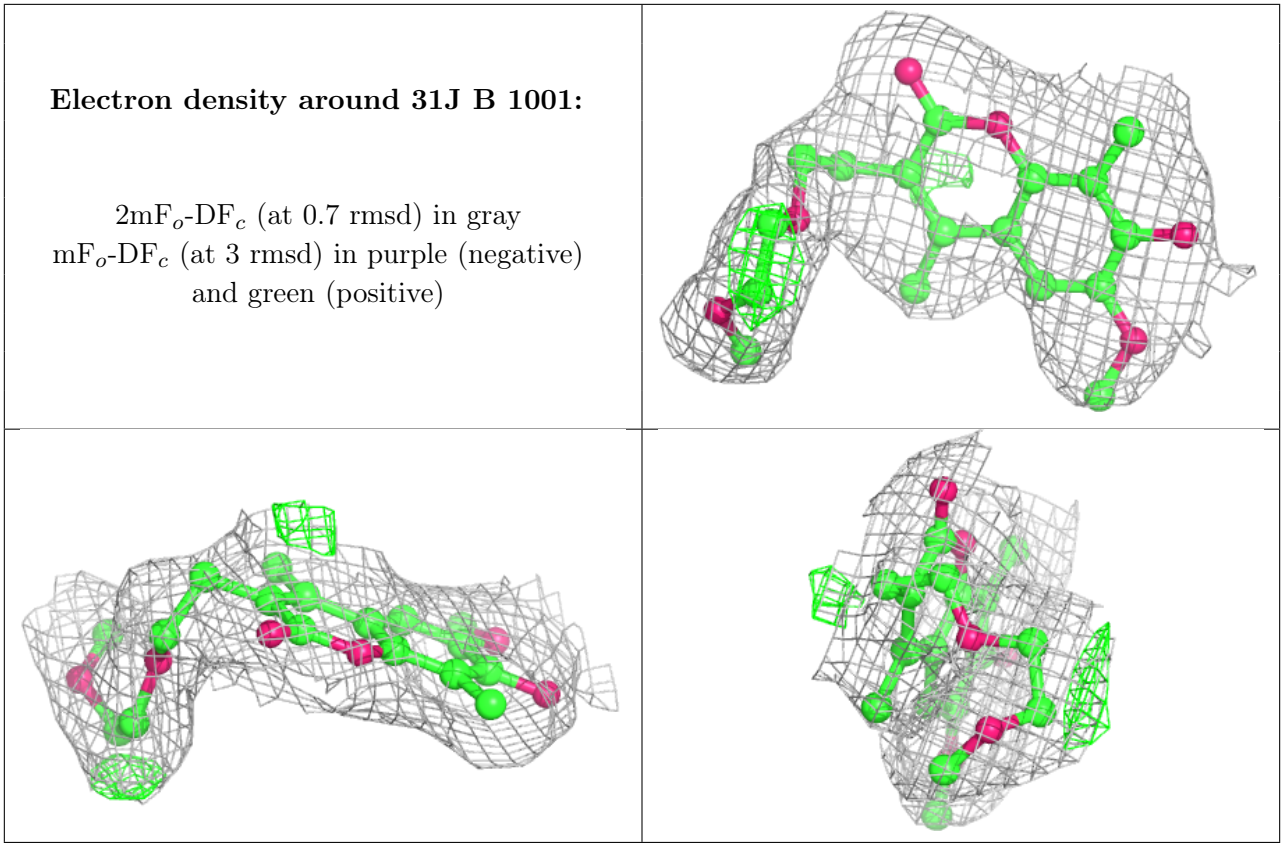
Electron density around 31J A 1001:

2mF$_o$-DF$_c$ (at 0.7 rmsd) in gray
mF$_o$-DF$_c$ (at 3 rmsd) in purple (negative)
and green (positive)



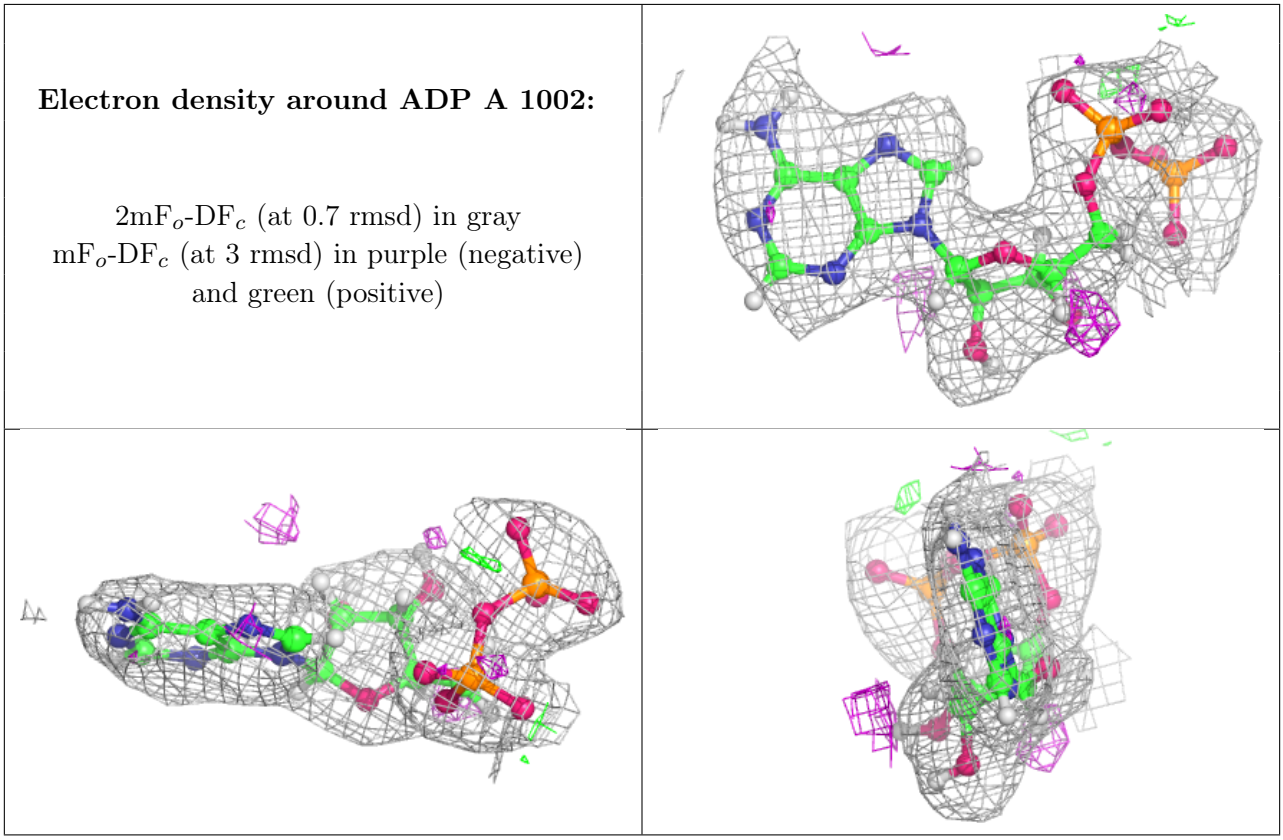
Electron density around 31J B 1001:

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



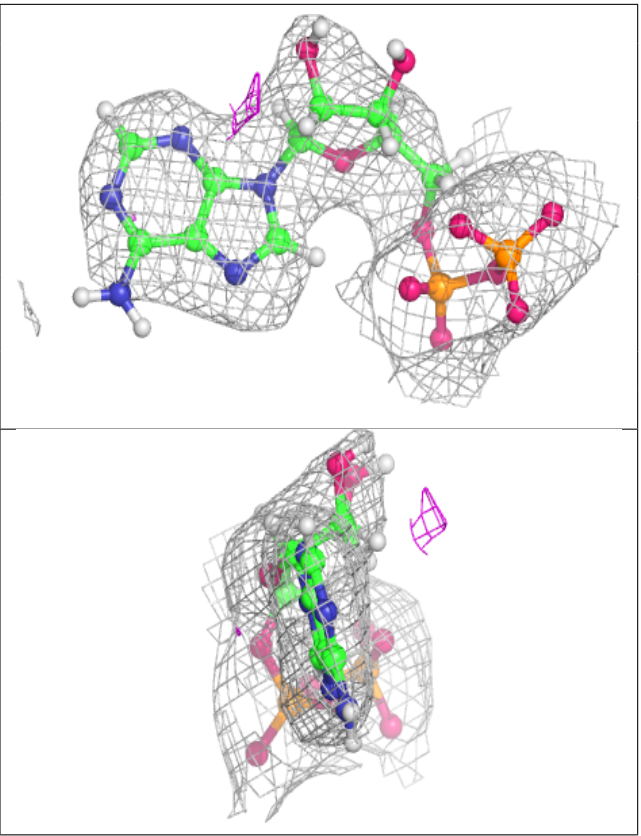
Electron density around ADP A 1002:

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



Electron density around ADP B 1002:

$2mF_o$-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.