



Full wwPDB X-ray Structure Validation Report ⓘ

May 16, 2020 – 02:04 am BST

PDB ID : 5A2S
Title : Potent, selective and CNS-penetrant tetrasubstituted cyclopropane class IIa histone deacetylase (HDAC) inhibitors
Authors : Luckhurst, C.A.; Breccia, P.; Stott, A.J.; Aziz, O.; Birch, H.; Burli, R.W.; Hughes, S.; Jarvis, R.E.; Lamers, M.; Leonard, P.; Matthews, K.L.; McAllister, G.; Pollack, S.; Saville-Stones, E.; Wishart, G.; Yates, D.; Dominguez, C.
Deposited on : 2015-05-22
Resolution : 2.65 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

A user guide is available at

<https://www.wwpdb.org/validation/2017/XrayValidationReportHelp>

with specific help available everywhere you see the ⓘ symbol.

The following versions of software and data (see [references ⓘ](#)) 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	:	2.11
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)	:	2.11

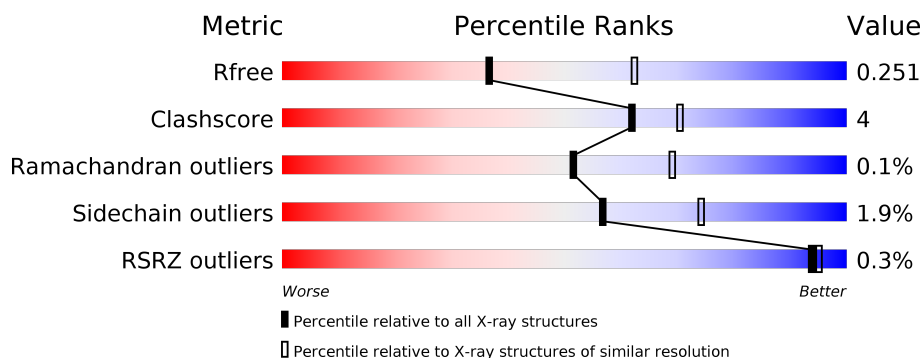
1 Overall quality at a glance

The following experimental techniques were used to determine the structure:

X-RAY DIFFRACTION

The reported resolution of this entry is 2.65 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	1332 (2.68-2.64)
Clashscore	141614	1374 (2.68-2.64)
Ramachandran outliers	138981	1349 (2.68-2.64)
Sidechain outliers	138945	1349 (2.68-2.64)
RSRZ outliers	127900	1318 (2.68-2.64)

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 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 $\leq 5\%$. 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	395	
1	B	395	

2 Entry composition

There are 5 unique types of molecules in this entry. The entry contains 5783 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 HISTONE DEACETYLASE 4.

Mol	Chain	Residues	Atoms					ZeroOcc	AltConf	Trace
1	A	380	Total	C	N	O	S	0	0	0
			2820	1779	488	533	20			
1	B	380	Total	C	N	O	S	0	0	0
			2816	1775	488	533	20			

There are 20 discrepancies between the modelled and reference sequences:

Chain	Residue	Modelled	Actual	Comment	Reference
A	645	MET	-	expression tag	UNP P56524
A	646	GLY	-	expression tag	UNP P56524
A	647	SER	-	expression tag	UNP P56524
A	1034	HIS	-	expression tag	UNP P56524
A	1035	HIS	-	expression tag	UNP P56524
A	1036	HIS	-	expression tag	UNP P56524
A	1037	HIS	-	expression tag	UNP P56524
A	1038	HIS	-	expression tag	UNP P56524
A	1039	HIS	-	expression tag	UNP P56524
A	728	ALA	LEU	engineered mutation	UNP P56524
B	645	MET	-	expression tag	UNP P56524
B	646	GLY	-	expression tag	UNP P56524
B	647	SER	-	expression tag	UNP P56524
B	1034	HIS	-	expression tag	UNP P56524
B	1035	HIS	-	expression tag	UNP P56524
B	1036	HIS	-	expression tag	UNP P56524
B	1037	HIS	-	expression tag	UNP P56524
B	1038	HIS	-	expression tag	UNP P56524
B	1039	HIS	-	expression tag	UNP P56524
B	728	ALA	LEU	engineered mutation	UNP P56524

- Molecule 2 is (1S,2S,3S)-1-fluoranyl-2-[4-(5-fluoranylpuridin-2-yl)phenyl]-N-oxidanyl-3-p henyl-cyclopropane-1-carboxamide (three-letter code: OTF) (formula: C₂₀H₁₅F₂N₃O₂).

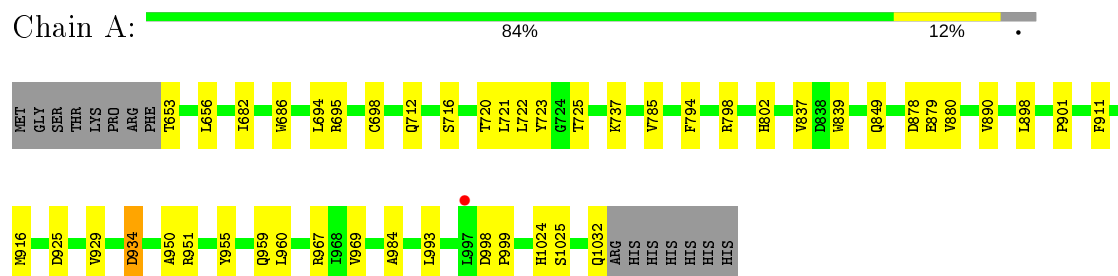
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Mol	Chain	Residues	Atoms		ZeroOcc	AltConf
5	B	44	Total	O	0	0
			44	44		

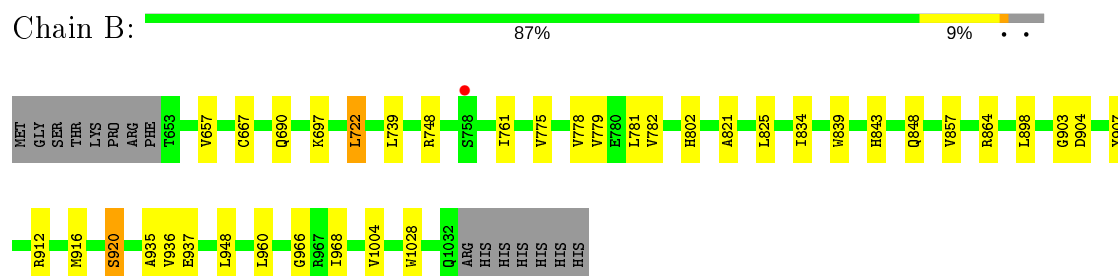
3 Residue-property plots [i](#)

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: HISTONE DEACETYLASE 4



• Molecule 1: HISTONE DEACETYLASE 4



4 Data and refinement statistics

Property	Value	Source
Space group	P 2 21 21	Depositor
Cell constants a, b, c, α , β , γ	52.95Å 82.14Å 161.63Å 90.00° 90.00° 90.00°	Depositor
Resolution (Å)	161.63 – 2.65 28.80 – 2.65	Depositor EDS
% Data completeness (in resolution range)	99.2 (161.63-2.65) 99.4 (28.80-2.65)	Depositor EDS
R_{merge}	0.18	Depositor
R_{sym}	(Not available)	Depositor
$\langle I/\sigma(I) \rangle$ ¹	1.49 (at 2.64Å)	Xtriage
Refinement program	REFMAC 5.6.0117	Depositor
R, R_{free}	0.185 , 0.256 0.188 , 0.251	Depositor DCC
R_{free} test set	1079 reflections (5.14%)	wwPDB-VP
Wilson B-factor (Å ²)	33.4	Xtriage
Anisotropy	0.155	Xtriage
Bulk solvent k_{sol} (e/Å ³), B_{sol} (Å ²)	0.34 , 28.8	EDS
L-test for twinning ²	$\langle L \rangle = 0.45$, $\langle L^2 \rangle = 0.27$	Xtriage
Estimated twinning fraction	No twinning to report.	Xtriage
F_o, F_c correlation	0.93	EDS
Total number of atoms	5783	wwPDB-VP
Average B, all atoms (Å ²)	26.0	wwPDB-VP

Xtriage's analysis on translational NCS is as follows: *The largest off-origin peak in the Patterson function is 6.48% of the height of the origin peak. No significant pseudotranslation is detected.*

¹Intensities estimated from amplitudes.

²Theoretical 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 [i](#)

5.1 Standard geometry [i](#)

Bond lengths and bond angles in the following residue types are not validated in this section: NA, ZN, OTF

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	1/2884 (0.0%)	0.56	0/3923
1	B	0.56	1/2880 (0.0%)	0.58	0/3919
All	All	0.55	2/5764 (0.0%)	0.57	0/7842

All (2) bond length outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(Å)	Ideal(Å)
1	B	1028	TRP	CD2-CE2	5.19	1.47	1.41
1	A	849	GLN	CD-OE1	5.10	1.35	1.24

There are no bond angle outliers.

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	2820	0	2731	25	1
1	B	2816	0	2720	24	1
2	A	27	0	13	0	0
2	B	27	0	13	0	0
3	A	2	0	0	0	0
3	B	2	0	0	0	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
5	A	43	0	0	0	0
5	B	44	0	0	1	0
All	All	5783	0	5477	49	1

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 (49) 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:697:LYS:CB	5:B:2011:HOH:O	2.29	0.79
1:B:916:MET:CE	1:B:960:LEU:HD23	2.19	0.73
1:B:916:MET:HE1	1:B:960:LEU:HD23	1.72	0.70
1:A:916:MET:CE	1:A:960:LEU:HD23	2.23	0.69
1:A:916:MET:HE2	1:A:960:LEU:HD23	1.75	0.69
1:A:785:VAL:HG21	1:A:969:VAL:HG22	1.76	0.67
1:A:1025:SER:O	1:A:1032:GLN:NE2	2.33	0.62
1:B:864:ARG:CZ	1:B:898:LEU:HD21	2.30	0.61
1:A:925:ASP:O	1:A:967:ARG:NH1	2.34	0.61
1:B:657:VAL:HG12	1:B:781:LEU:HD22	1.83	0.60
1:B:778:VAL:O	1:B:782:VAL:HG23	2.03	0.58
1:B:904:ASP:HB2	1:B:1004:VAL:HG11	1.87	0.57
1:B:935:ALA:HB1	1:B:948:LEU:HD22	1.87	0.56
1:B:907:TYR:CD1	1:B:948:LEU:HD11	2.42	0.54
1:A:686:TRP:CZ2	1:A:695:ARG:HD2	2.43	0.53
1:B:916:MET:HE2	1:B:960:LEU:HD23	1.90	0.52
1:A:656:LEU:HD12	1:A:698:CYS:HB2	1.92	0.51
1:A:712:GLN:HG3	1:A:720:THR:HG21	1.91	0.51
1:A:955:TYR:OH	1:A:999:PRO:HA	2.11	0.51
1:A:785:VAL:HG21	1:A:969:VAL:CG2	2.41	0.50
1:B:903:GLY:HA2	1:B:948:LEU:HD12	1.91	0.50
1:A:682:ILE:HD13	1:A:798:ARG:HG2	1.92	0.50
1:A:722:LEU:HD23	1:A:723:TYR:CE2	2.47	0.49
1:B:722:LEU:HD13	1:B:739:LEU:HD22	1.94	0.49
1:B:916:MET:O	1:B:920:SER:OG	2.28	0.48
1:B:935:ALA:CB	1:B:948:LEU:HD22	2.43	0.48
1:A:878:ASP:O	1:A:880:VAL:HG13	2.15	0.47
1:A:916:MET:HE1	1:A:960:LEU:HD23	1.95	0.47
1:B:782:VAL:CG1	1:B:825:LEU:HD11	2.44	0.47
1:B:834:ILE:HB	1:B:857:VAL:HG22	1.97	0.47

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Atom-1	Atom-2	Interatomic distance (Å)	Clash overlap (Å)
1:A:916:MET:HE3	1:A:959:GLN:HB3	1.96	0.46
1:A:898:LEU:HD22	1:A:901:PRO:HB3	1.97	0.46
1:A:950:ALA:HB1	1:A:984:ALA:N	2.30	0.46
1:A:721:LEU:O	1:A:725:THR:OG1	2.34	0.45
1:A:716:SER:O	1:A:720:THR:HG23	2.17	0.44
1:B:667:CYS:HB2	1:B:761:ILE:HG21	2.00	0.44
1:A:694:LEU:HD22	1:A:993:LEU:HD12	1.99	0.44
1:A:934:ASP:OD1	1:A:934:ASP:N	2.51	0.43
1:A:890:VAL:HG22	1:A:1024:HIS:CE1	2.54	0.42
1:B:775:VAL:O	1:B:779:VAL:HG23	2.19	0.42
1:B:936:VAL:HG12	1:B:937:GLU:O	2.20	0.41
1:A:656:LEU:HD23	1:A:794:PHE:HB3	2.02	0.41
1:B:782:VAL:HG21	1:B:821:ALA:HB1	2.02	0.41
1:B:966:GLY:O	1:B:968:ILE:HG22	2.20	0.41
1:B:864:ARG:NH1	1:B:898:LEU:HD21	2.36	0.41
1:A:837:VAL:HB	1:A:929:VAL:HG22	2.03	0.41
1:B:843:HIS:NE2	1:B:848:GLN:OE1	2.47	0.40
1:B:907:TYR:CE1	1:B:948:LEU:HD11	2.56	0.40
1:A:951:ARG:HE	1:A:998:ASP:CB	2.34	0.40

All (1) 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:A:737:LYS:O	1:B:748:ARG:NH2[4_445]	2.17	0.03

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	378/395 (96%)	357 (94%)	20 (5%)	1 (0%)	41 56

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Mol	Chain	Analysed	Favoured	Allowed	Outliers	Percentiles	
1	B	378/395 (96%)	362 (96%)	16 (4%)	0	100	100
All	All	756/790 (96%)	719 (95%)	36 (5%)	1 (0%)	51	69

All (1) Ramachandran outliers are listed below:

Mol	Chain	Res	Type
1	A	879	GLU

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	297/321 (92%)	292 (98%)	5 (2%)	60	77
1	B	296/321 (92%)	290 (98%)	6 (2%)	55	73
All	All	593/642 (92%)	582 (98%)	11 (2%)	57	74

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

Mol	Chain	Res	Type
1	A	653	THR
1	A	802	HIS
1	A	839	TRP
1	A	911	PHE
1	A	934	ASP
1	B	690	GLN
1	B	722	LEU
1	B	802	HIS
1	B	839	TRP
1	B	912	ARG
1	B	920	SER

Some sidechains can be flipped to improve hydrogen bonding and reduce clashes. All (5) such sidechains are listed below:

Mol	Chain	Res	Type
1	A	683	GLN
1	A	690	GLN
1	A	826	GLN
1	A	947	ASN
1	B	731	GLN

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 carbohydrates in this entry.

5.6 Ligand geometry [i](#)

Of 8 ligands modelled in this entry, 6 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$
2	OTF	B	1	3	30,30,30	1.29	3 (10%)	34,44,44	2.13	9 (26%)
2	OTF	A	1	3	30,30,30	1.20	3 (10%)	34,44,44	1.91	8 (23%)

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	OTF	B	1	3	-	0/16/33/33	0/4/4/4
2	OTF	A	1	3	-	0/16/33/33	0/4/4/4

All (6) bond length outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(Å)	Ideal(Å)
2	A	1	OTF	F5-C2	-4.02	1.31	1.39
2	B	1	OTF	F5-C2	-3.83	1.32	1.39
2	B	1	OTF	C25-C24	2.85	1.41	1.37
2	A	1	OTF	C9-C4	-2.61	1.47	1.51
2	B	1	OTF	C2-C3	2.25	1.55	1.52
2	A	1	OTF	C23-C24	2.02	1.40	1.37

All (17) bond angle outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(°)	Ideal(°)
2	B	1	OTF	C18-C21-N26	5.29	123.11	117.41
2	B	1	OTF	C9-C4-C3	-5.26	114.08	123.41
2	B	1	OTF	C23-N22-C21	4.75	122.50	116.22
2	A	1	OTF	C9-C4-C3	-4.46	115.50	123.41
2	A	1	OTF	C4-C2-C1	4.07	124.06	117.05
2	A	1	OTF	C24-C25-N26	-3.94	119.08	121.46
2	A	1	OTF	C25-N26-C21	3.64	121.03	116.22
2	A	1	OTF	C24-C23-N22	-3.55	119.32	121.46
2	B	1	OTF	C25-N26-C21	3.52	120.88	116.22
2	A	1	OTF	C23-N22-C21	3.23	120.50	116.22
2	B	1	OTF	N26-C21-N22	-3.20	118.65	125.57
2	B	1	OTF	C3-C2-C1	3.12	122.44	117.05
2	B	1	OTF	C4-C2-C1	2.95	122.13	117.05
2	A	1	OTF	C18-C21-N22	2.77	120.39	117.41
2	B	1	OTF	C24-C23-N22	-2.57	119.91	121.46
2	B	1	OTF	C15-C3-C4	-2.44	119.08	123.41
2	A	1	OTF	N26-C21-N22	-2.36	120.48	125.57

There are no chirality outliers.

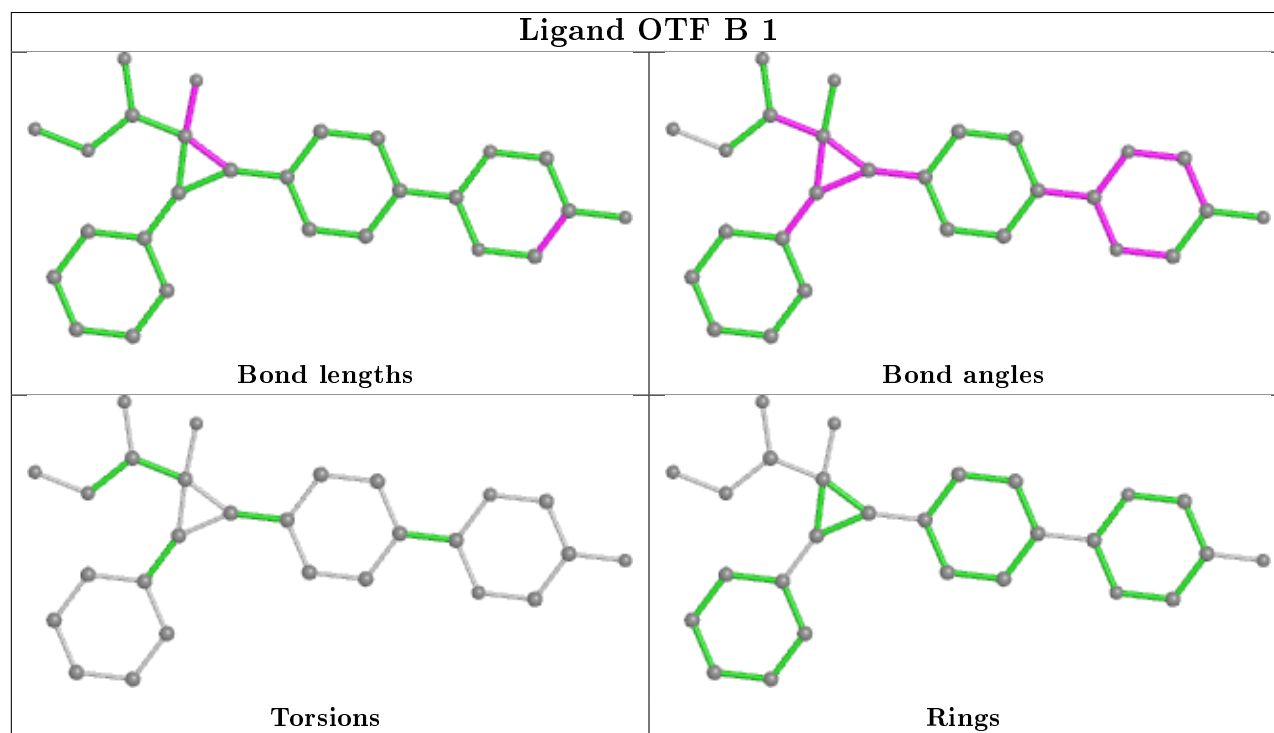
There are no torsion outliers.

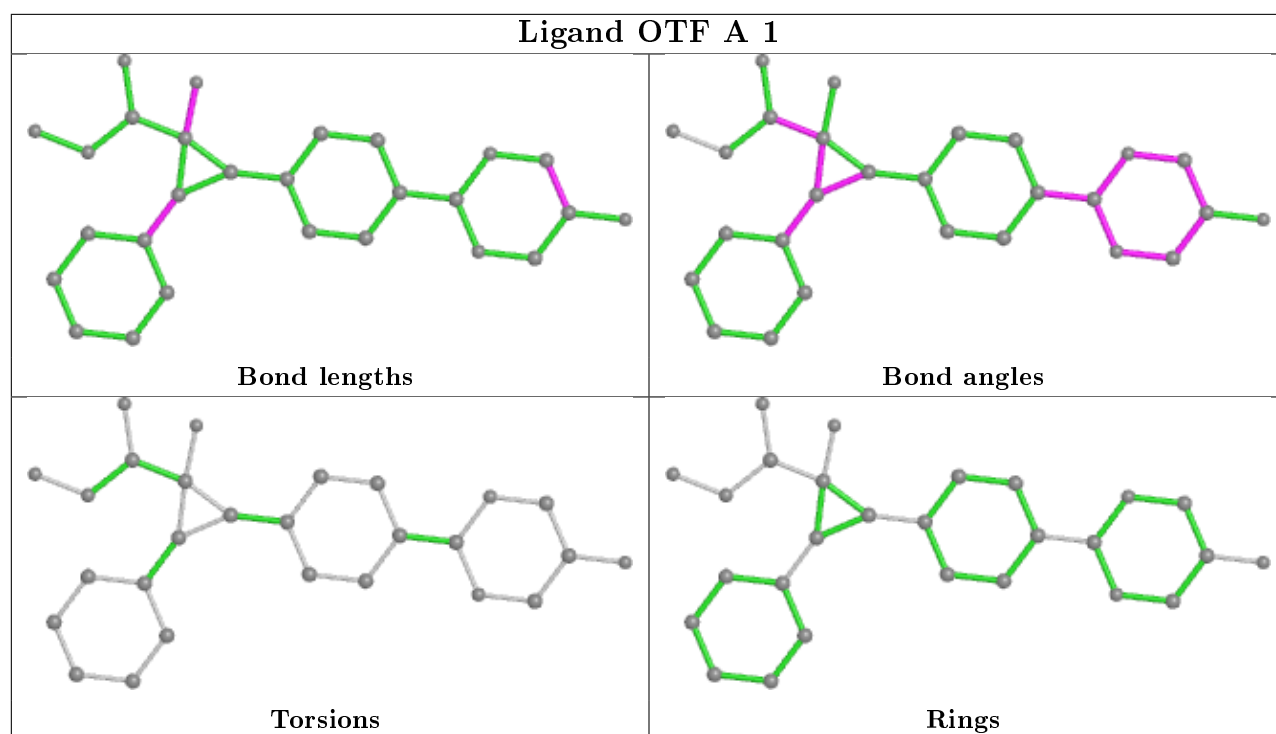
There are no ring outliers.

No monomer is involved in short contacts.

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 than 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.





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	380/395 (96%)	-0.39	1 (0%) 94 95	17, 24, 35, 46	0
1	B	380/395 (96%)	-0.35	1 (0%) 94 95	17, 26, 41, 66	0
All	All	760/790 (96%)	-0.37	2 (0%) 94 95	17, 25, 39, 66	0

All (2) RSRZ outliers are listed below:

Mol	Chain	Res	Type	RSRZ
1	B	758	SER	4.3
1	A	997	LEU	2.8

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. 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.

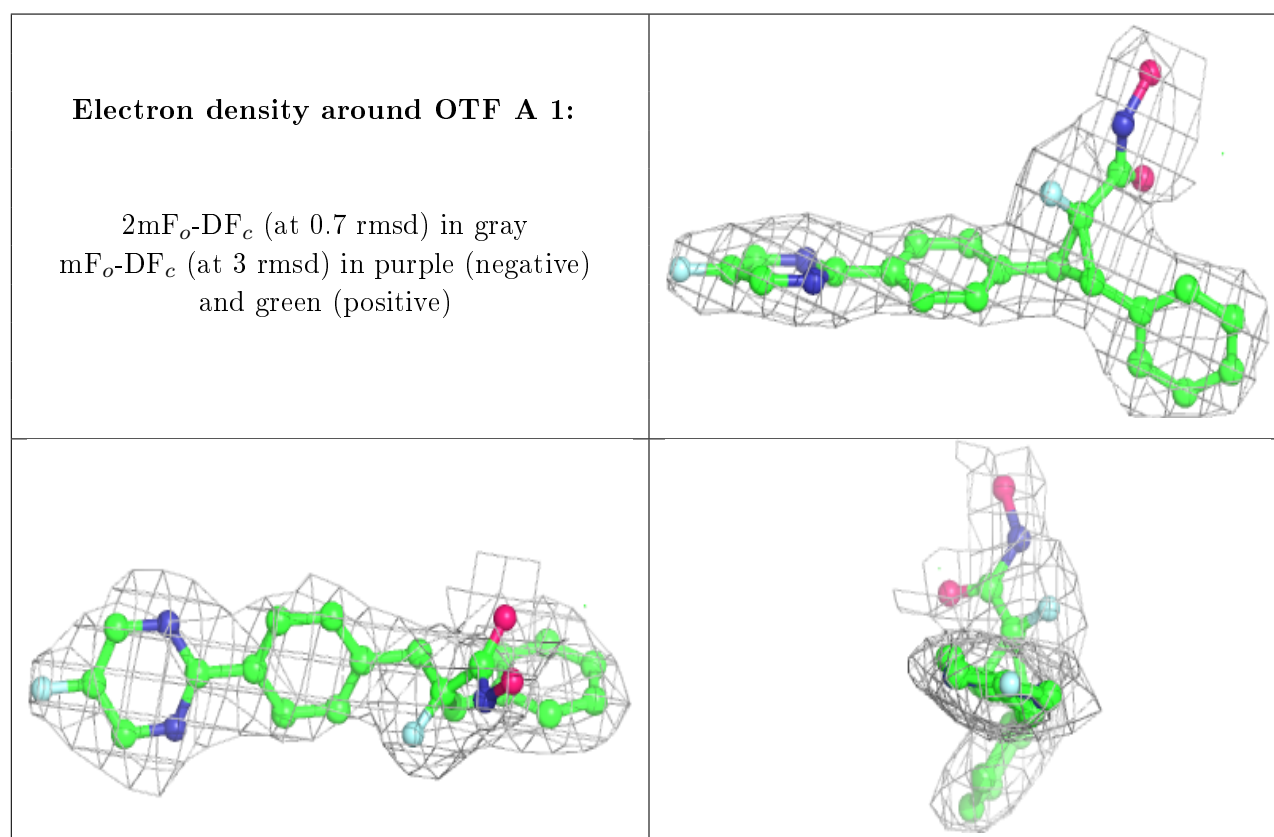
Mol	Type	Chain	Res	Atoms	RSCC	RSR	B-factors(Å ²)	Q<0.9
2	OTF	A	1	27/27	0.94	0.17	21,22,30,32	0
2	OTF	B	1	27/27	0.95	0.16	23,29,32,33	0
4	NA	B	4	1/1	0.98	0.16	22,22,22,22	0

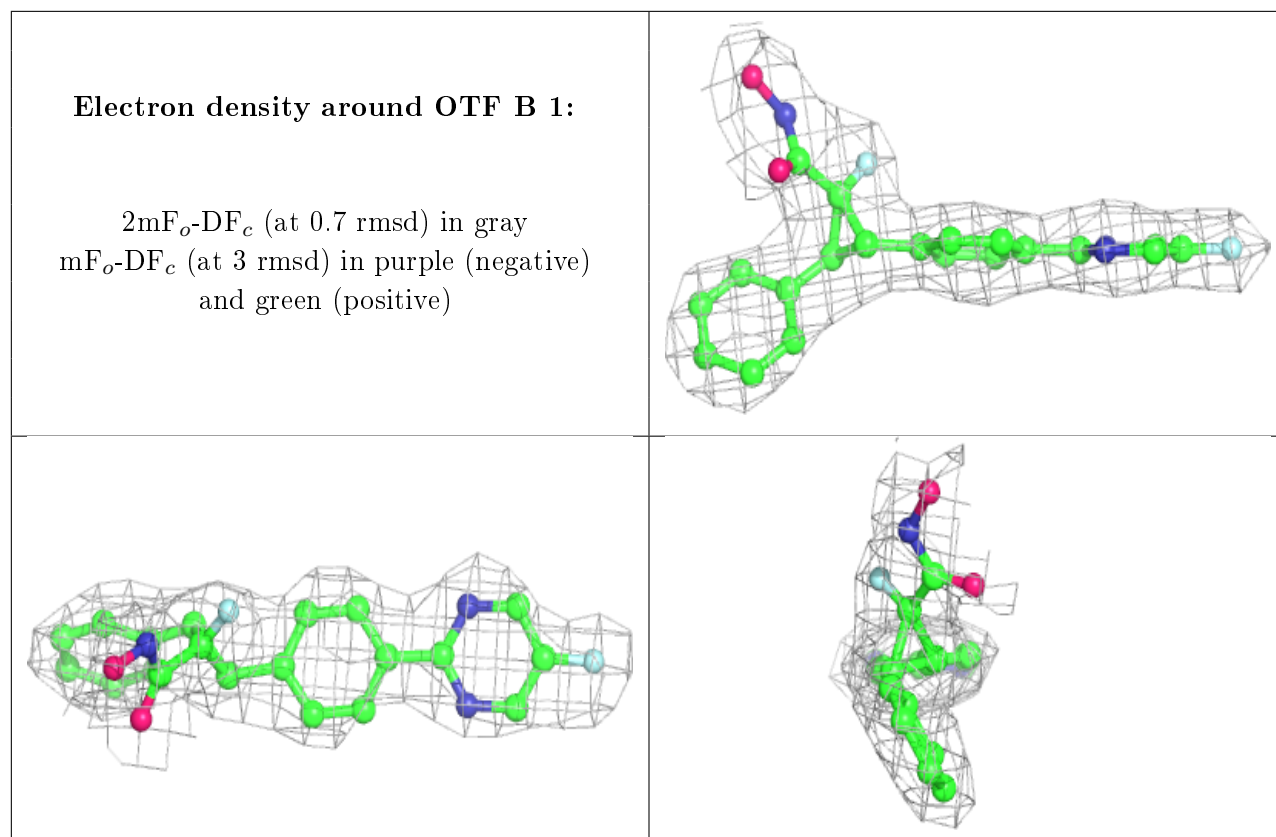
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Mol	Type	Chain	Res	Atoms	RSCC	RSR	B-factors(\AA^2)	Q<0.9
4	NA	A	4	1/1	0.99	0.19	22,22,22,22	0
3	ZN	B	3	1/1	0.99	0.04	34,34,34,34	0
3	ZN	B	2	1/1	0.99	0.08	22,22,22,22	0
3	ZN	A	3	1/1	0.99	0.05	27,27,27,27	0
3	ZN	A	2	1/1	1.00	0.07	20,20,20,20	0

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.





6.5 Other polymers [i](#)

There are no such residues in this entry.