

Full wwPDB X-ray Structure Validation Report (i)

Aug 19, 2023 – 11:15 PM EDT

PDB ID : 2HAL

Title: An episulfide cation (thiiranium ring) trapped in the active site of HAV 3C

proteinase inactivated by peptide-based ketone inhibitors

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Deposited on : 2006-06-13

Resolution : 1.35 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.orgA 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:

 $Mol Probity \quad : \quad 4.02b\text{--}467$

Mogul: 1.8.5 (274361), CSD as541be (2020)

Xtriage (Phenix) : 1.13 EDS : 2.35

Percentile statistics : 20191225.v01 (using entries in the PDB archive December 25th 2019)

 $Refmac \quad : \quad 5.8.0158$

CCP4 : 7.0.044 (Gargrove)

Ideal geometry (proteins) : Engh & Huber (2001) Ideal geometry (DNA, RNA) : Parkinson et al. (1996)

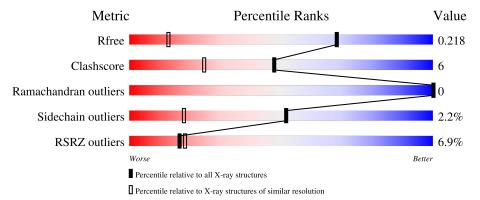
Validation Pipeline (wwPDB-VP) : 2.35

1 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 1.35 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 $(\# \mathrm{Entries})$	Similar resolution $(\# \text{Entries, resolution range}(\text{\AA}))$
R_{free}	130704	1509 (1.38-1.34)
Clashscore	141614	1551 (1.38-1.34)
Ramachandran outliers	138981	1530 (1.38-1.34)
Sidechain outliers	138945	1530 (1.38-1.34)
RSRZ outliers	127900	1487 (1.38-1.34)

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	212	92%	8% •
2	I	7	71% 29%	



2 Entry composition (i)

There are 4 unique types of molecules in this entry. The entry contains 2140 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 Hepatitis A Protease 3C.

Mol	Chain	Residues	Atoms			ZeroOcc	AltConf	Trace		
1	Λ	212	Total	С	N	О	S	0	1	0
1	A	212	1636	1043	280	303	10	0	1	

There is a discrepancy between the modelled and reference sequences:

Chain	Residue	Modelled	Actual	Comment	Reference
A	24	SER	CYS	engineered mutation	UNP Q81090

• Molecule 2 is a protein called N-ACETYL-LEUCYL-PHENYLALANYL-PHENYLALANY L-GLUTAMATE-FLUOROMETHYLKETONE INHIBITOR.

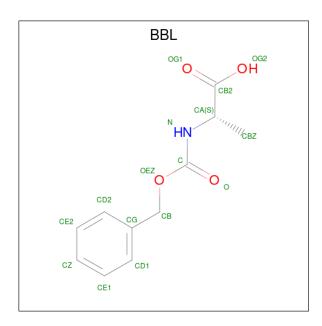
Mol	Chain	Residues	Atoms		ZeroOcc	AltConf	Trace		
2	I	6	Total 53	C 38	N 5	O 10	0	2	1

There is a discrepancy between the modelled and reference sequences:

Chain	Residue	Modelled	Actual	Comment	Reference
I	5	GLU	GLK	microheterogeneity	PDB 2HAL

• Molecule 3 is N-[(BENZYLOXY)CARBONYL]-L-ALANINE (three-letter code: BBL) (formula: C₁₁H₁₃NO₄).





Mol	Chain	Residues	Atoms		ZeroOcc	AltConf		
3	A	1	Total 16	C 11	N 1	O 4	0	0

• Molecule 4 is water.

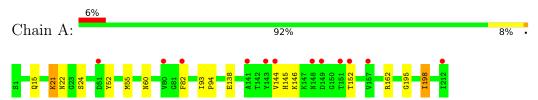
ľ	Mol	Chain	Residues	Atoms	ZeroOcc	AltConf
	4	A	433	Total O 433 433	0	0
	4	I	2	Total O 2 2	0	0



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: Hepatitis A Protease 3C



• Molecule 2: N-ACETYL-LEUCYL-PHENYLALANYL-PHENYLALANYL-GLUTAMATE-FL UOROMETHYLKETONE INHIBITOR

Chain I: 71% 29%





4 Data and refinement statistics (i)

Property	Value	Source
Space group	P 21 21 21	Depositor
Cell constants	44.58Å 56.24Å 81.05Å	Donositor
a, b, c, α , β , γ	90.00° 90.00° 90.00°	Depositor
Resolution (Å)	20.00 - 1.35	Depositor
rtesolution (A)	39.06 - 1.35	EDS
% Data completeness	85.5 (20.00-1.35)	Depositor
(in resolution range)	85.4 (39.06-1.35)	EDS
R_{merge}	0.06	Depositor
R_{sym}	(Not available)	Depositor
$< I/\sigma(I) > 1$	1.41 (at 1.35Å)	Xtriage
Refinement program	REFMAC 5.2.0005	Depositor
D D.	0.184 , 0.204	Depositor
R, R_{free}	0.206 , 0.218	DCC
R_{free} test set	1950 reflections (5.02%)	wwPDB-VP
Wilson B-factor (Å ²)	13.5	Xtriage
Anisotropy	0.206	Xtriage
Bulk solvent $k_{sol}(e/Å^3)$, $B_{sol}(Å^2)$	0.30 , 31.0	EDS
L-test for twinning ²	$ < L > = 0.48, < L^2> = 0.31$	Xtriage
Estimated twinning fraction	No twinning to report.	Xtriage
F_o, F_c correlation	0.95	EDS
Total number of atoms	2140	wwPDB-VP
Average B, all atoms (Å ²)	17.0	wwPDB-VP

Xtriage's analysis on translational NCS is as follows: The largest off-origin peak in the Patterson function is 7.71% of the height of the origin peak. No significant pseudotranslation is detected.

²Theoretical 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.



¹Intensities estimated from amplitudes.

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: ACE, GLK, BBL, CF0

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		
MIOI	Chain	RMSZ	# Z > 5	RMSZ	# Z > 5	
1	A	0.41	0/1670	0.55	0/2258	
2	I	0.53	0/40	0.38	0/51	
All	All	0.41	0/1710	0.54	0/2309	

There are no bond length outliers.

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	1636	0	1661	20	0
2	I	53	0	43	7	0
3	A	16	0	11	0	0
4	A	433	0	0	2	0
4	I	2	0	0	0	0
All	All	2140	0	1715	21	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.

All (21) close contacts within the same asymmetric unit are listed below, sorted by their clash magnitude.



Atom-1	Atom-2	Interatomic	Clash
		distance (Å)	overlap (Å)
1:A:55:MET:HG3	4:A:1305:HOH:O	1.66	0.95
1:A:195:GLY:O	1:A:198:ILE:HG22	1.98	0.63
1:A:138:GLU:CG	1:A:162:ARG:HD3	2.30	0.62
1:A:146:LYS:HB3	2:I:4:PHE:CE2	2.37	0.60
1:A:144:VAL:HG22	1:A:152:THR:HB	1.89	0.55
1:A:138:GLU:HG3	1:A:162:ARG:HD3	1.91	0.53
1:A:21:LYS:HA	1:A:21:LYS:HZ2	1.75	0.52
1:A:21:LYS:NZ	1:A:22:ASN:H	2.07	0.52
1:A:138:GLU:HG2	1:A:162:ARG:HD3	1.90	0.52
1:A:138:GLU:HG3	1:A:162:ARG:CD	2.41	0.49
1:A:198:ILE:HG23	2:I:2:LEU:HD21	1.94	0.49
1:A:82:PHE:HE2	4:A:953:HOH:O	1.95	0.48
1:A:21:LYS:HZ1	1:A:22:ASN:H	1.62	0.47
1:A:146:LYS:HB3	2:I:4:PHE:HE2	1.79	0.46
2:I:4:PHE:CG	2:I:4:PHE:O	2.70	0.45
1:A:15:GLN:HB2	1:A:60:ASN:HB3	1.99	0.44
1:A:145:HIS:HD2	2:I:4:PHE:CG	2.38	0.42
1:A:145:HIS:HD2	2:I:4:PHE:CD1	2.38	0.41
1:A:93:ILE:HG13	1:A:94:PRO:HD2	2.02	0.40
1:A:144:VAL:CG2	1:A:152:THR:HB	2.50	0.40
1:A:145:HIS:CD2	2:I:4:PHE:CD1	3.09	0.40

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	Perce	entiles
1	A	211/212 (100%)	204 (97%)	7 (3%)	0	100	100
2	I	3/7 (43%)	3 (100%)	0	0	100	100
All	All	$214/219 \ (98\%)$	207 (97%)	7 (3%)	0	100	100

There are no Ramachandran outliers to report.



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	Rotameric Outliers		Percentiles		
1	A	179/178 (101%)	175 (98%)	4 (2%)	52	18		
2	I	4/4 (100%)	4 (100%)	0	100	100		
All	All	183/182 (100%)	179 (98%)	4 (2%)	52	18		

All (4) residues with a non-rotameric sidechain are listed below:

Mol	Chain	Res	Type
1	A	21	LYS
1	A	24	SER
1	A	52	TYR
1	A	198	ILE

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	15	GLN
1	A	145	HIS
1	A	196	ASN
1	A	206	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)

1 non-standard protein/DNA/RNA residue is 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			
MIOI	туре	Chain	nes	Lilik	Counts	RMSZ	# Z > 2	Counts	RMSZ	# Z > 2
2	GLK	I	5[A]	2,1	8,8,9	0.87	0	8,9,11	0.91	0

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	GLK	I	5[A]	2,1	-	2/7/7/9	-

There are no bond length outliers.

There are no bond angle outliers.

There are no chirality outliers.

All (2) torsion outliers are listed below:

Mol	Chain	Res	Type	Atoms
2	I	5[A]	GLK	O-C-CA-N
2	I	5[A]	GLK	O-C-CA-CB

There are no ring outliers.

No monomer is involved in short contacts.

5.5 Carbohydrates (i)

There are no monosaccharides in this entry.

5.6 Ligand geometry (i)

1 ligand is 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			В	ond ang	les
	WIOI	туре	Chain	rtes	Lilik	Counts	RMSZ	# Z > 2	Counts	RMSZ	# Z > 2
Ī	3	BBL	A	901	1	16,16,16	1.45	1 (6%)	20,20,20	1.14	1 (5%)

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	BBL	A	901	1	-	2/13/13/13	0/1/1/1

All (1) bond length outliers are listed below:

ľ	Mol	Chain	Res	Type	Atoms	\mathbf{Z}	$\operatorname{Observed}(\text{\AA})$	$\operatorname{Ideal}(ext{\AA})$
	3	A	901	BBL	OEZ-C	4.81	1.44	1.35

All (1) bond angle outliers are listed below:

Mo	l Chain	Res	Type	Atoms	\mathbf{Z}	$\mathbf{Observed}(^{o})$	$\operatorname{Ideal}({}^{o})$
3	A	901	BBL	CB-OEZ-C	2.85	122.28	115.93

There are no chirality outliers.

All (2) torsion outliers are listed below:

Mol	Chain	Res	Type	Atoms
3	A	901	BBL	CBZ-CA-CB2-OG2
3	A	901	BBL	CBZ-CA-CB2-OG1

There are no ring outliers.

No monomer is involved in short contacts.

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, 95^{th} 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 $>$	$\# \mathrm{RSRZ}{>}2$	$OWAB(Å^2)$	Q<0.9
1	A	212/212 (100%)	0.34	12 (5%) 23 26	4, 10, 28, 39	0
2	I	3/7 (42%)	6.90	3 (100%) 0 0	26, 26, 26, 34	0
All	All	215/219 (98%)	0.43	15 (6%) 16 18	4, 10, 28, 39	0

All (15) RSRZ outliers are listed below:

Mol	Chain	Res	Type	RSRZ	
2	I	4	PHE	9.5	
2	I	3	PHE	6.7	
1	A	82	PHE	4.9	
1	A	157	VAL	4.6	
2	I	2	LEU	4.5	
1	A	212	ILE	4.0	
1	A	144	VAL	3.4	
1	A	80	VAL	2.8	
1	A	149	ASP	2.6	
1	A	148	ASN	2.5	
1	A	152	THR	2.5	
1	A	143	TYR	2.4	
1	A	151	THR	2.4	
1	A	51	ASP	2.2	
1	1 A		ALA	2.2	

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, 95^{th} 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.



\mathbf{M}	ol	Type	Chain	Res	Atoms	RSCC	RSR	$\mathbf{B} ext{-}\mathbf{factors}(\mathbf{\mathring{A}}^2)$	Q<0.9
2	2	GLK	I	5[A]	9/10	0.74	0.21	22,24,32,37	9

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, 95^{th} 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	$\mathbf{B} ext{-}\mathbf{factors}(\mathbf{\mathring{A}}^2)$	Q < 0.9
3	BBL	A	901	16/16	0.86	0.13	12,16,20,24	0

6.5 Other polymers (i)

There are no such residues in this entry.

