



# wwPDB X-ray Structure Validation Summary Report ⓘ

Mar 7, 2022 – 02:16 AM EST

PDB ID : 6X8K  
Title : Caspase-3 in complex with elongated ketomethylene inhibitor  
Authors : Solania, A.; Xu, J.H.; Wolan, D.W.  
Deposited on : 2020-06-01  
Resolution : 2.17 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](mailto: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 : 2.27  
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) : 2.27

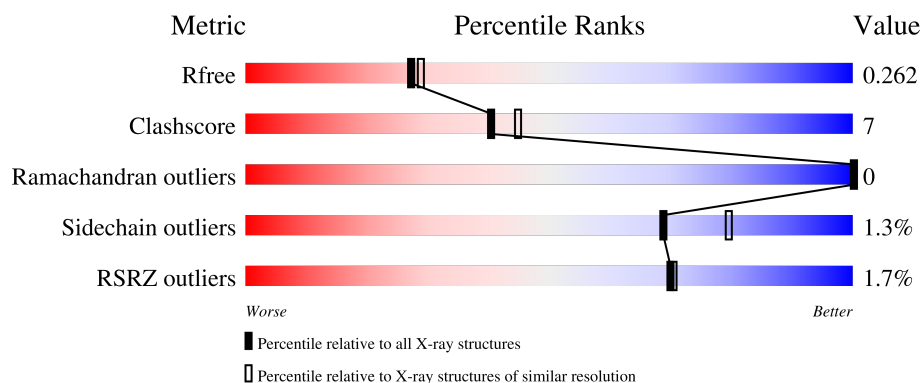
# 1 Overall quality at a glance

The following experimental techniques were used to determine the structure:

## *X-RAY DIFFRACTION*

The reported resolution of this entry is 2.17 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	6864 (2.20-2.16)
Clashscore	141614	7689 (2.20-2.16)
Ramachandran outliers	138981	7564 (2.20-2.16)
Sidechain outliers	138945	7564 (2.20-2.16)
RSRZ outliers	127900	6738 (2.20-2.16)

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  $\geq 3$ , 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	175	<div> <div>2%</div> <div>62% 18% 20%</div> </div>
1	B	175	<div> <div>%</div> <div>69% 11% 20%</div> </div>
2	C	110	<div> <div>2%</div> <div>65% 16% 18%</div> </div>
2	D	110	<div> <div>78% 5% 16%</div> </div>
3	E	8	<div> <div>12%</div> <div>38% 50% 12%</div> </div>

*Continued on next page...*

Continued from previous page...

Mol	Chain	Length	Quality of chain
3	F	8	<div> <div>12%</div> <div>75%</div> <div>25%</div> </div>

## 2 Entry composition

There are 4 unique types of molecules in this entry. The entry contains 3916 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 Caspase-3.

Mol	Chain	Residues	Atoms					ZeroOcc	AltConf	Trace
1	A	140	Total	C	N	O	S	0	1	0
			1081	668	197	206	10			
1	B	140	Total	C	N	O	S	0	3	0
			1108	684	199	215	10			

- Molecule 2 is a protein called Caspase-3.

Mol	Chain	Residues	Atoms					ZeroOcc	AltConf	Trace
2	C	90	Total	C	N	O	S	0	1	0
			740	487	116	132	5			
2	D	92	Total	C	N	O	S	0	0	0
			748	493	115	135	5			

There are 16 discrepancies between the modelled and reference sequences:

Chain	Residue	Modelled	Actual	Comment	Reference
C	278	LEU	-	expression tag	UNP P42574
C	279	GLU	-	expression tag	UNP P42574
C	280	HIS	-	expression tag	UNP P42574
C	281	HIS	-	expression tag	UNP P42574
C	282	HIS	-	expression tag	UNP P42574
C	283	HIS	-	expression tag	UNP P42574
C	284	HIS	-	expression tag	UNP P42574
C	285	HIS	-	expression tag	UNP P42574
D	278	LEU	-	expression tag	UNP P42574
D	279	GLU	-	expression tag	UNP P42574
D	280	HIS	-	expression tag	UNP P42574
D	281	HIS	-	expression tag	UNP P42574
D	282	HIS	-	expression tag	UNP P42574
D	283	HIS	-	expression tag	UNP P42574
D	284	HIS	-	expression tag	UNP P42574
D	285	HIS	-	expression tag	UNP P42574

- Molecule 3 is a protein called ketomethylene inhibitor.

Mol	Chain	Residues	Atoms				ZeroOcc	AltConf	Trace
3	E	8	Total	C	N	O	0	0	0
			54	32	7	15			
3	F	8	Total	C	N	O	0	0	0
			54	32	7	15			

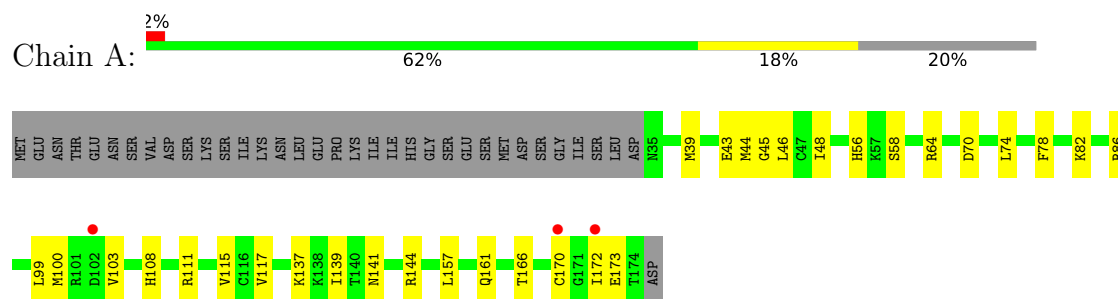
- Molecule 4 is water.

Mol	Chain	Residues	Atoms		ZeroOcc	AltConf
4	A	32	Total	O	0	0
			32	32		
4	B	43	Total	O	0	0
			43	43		
4	C	16	Total	O	0	0
			16	16		
4	D	32	Total	O	0	0
			32	32		
4	E	3	Total	O	0	0
			3	3		
4	F	5	Total	O	0	0
			5	5		

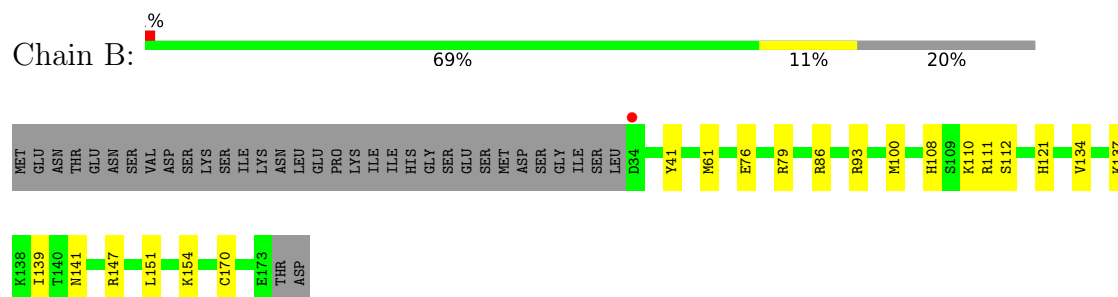
### 3 Residue-property plots [i](#)

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.

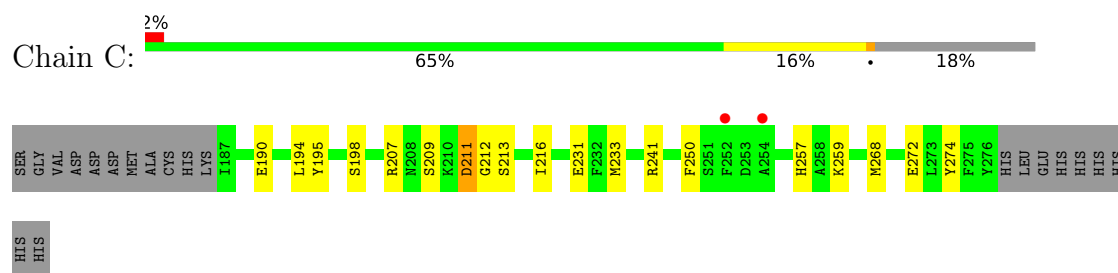
#### • Molecule 1: Caspase-3



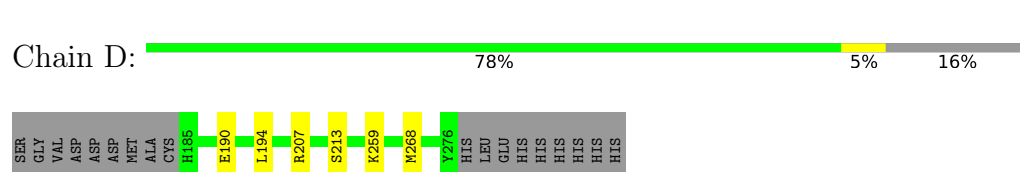
#### • Molecule 1: Caspase-3



#### • Molecule 2: Caspase-3

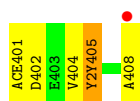


#### • Molecule 2: Caspase-3




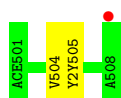
- Molecule 3: ketomethylene inhibitor

Chain E: 



- Molecule 3: ketomethylene inhibitor

Chain F: 



## 4 Data and refinement statistics

Property	Value	Source
Space group	P 21 21 2	Depositor
Cell constants a, b, c, $\alpha$ , $\beta$ , $\gamma$	69.86Å 96.91Å 100.46Å 90.00° 90.00° 90.00°	Depositor
Resolution (Å)	39.81 – 2.17 39.82 – 2.09	Depositor EDS
% Data completeness (in resolution range)	97.4 (39.81-2.17) 92.0 (39.82-2.09)	Depositor EDS
$R_{merge}$	0.20	Depositor
$R_{sym}$	(Not available)	Depositor
$\langle I/\sigma(I) \rangle$ <sup>1</sup>	2.57 (at 2.10Å)	Xtriage
Refinement program	PHENIX 1.16_3549	Depositor
R, $R_{free}$	0.221 , 0.263 0.222 , 0.262	Depositor DCC
$R_{free}$ test set	1929 reflections (5.12%)	wwPDB-VP
Wilson B-factor (Å <sup>2</sup> )	22.3	Xtriage
Anisotropy	0.268	Xtriage
Bulk solvent $k_{sol}$ (e/Å <sup>3</sup> ), $B_{sol}$ (Å <sup>2</sup> )	0.34 , 53.7	EDS
L-test for twinning <sup>2</sup>	$\langle  L  \rangle = 0.42$ , $\langle L^2 \rangle = 0.24$	Xtriage
Estimated twinning fraction	0.047 for -h,l,k	Xtriage
$F_o, F_c$ correlation	0.92	EDS
Total number of atoms	3916	wwPDB-VP
Average B, all atoms (Å <sup>2</sup> )	28.0	wwPDB-VP

Xtriage's analysis on translational NCS is as follows: *The analyses of the Patterson function reveals a significant off-origin peak that is 51.18 % of the origin peak, indicating pseudo-translational symmetry. The chance of finding a peak of this or larger height randomly in a structure without pseudo-translational symmetry is equal to 5.8175e-05. The detected translational NCS is most likely also responsible for the elevated intensity ratio.*

<sup>1</sup>Intensities estimated from amplitudes.

<sup>2</sup>Theoretical 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

### 5.1 Standard geometry

Bond lengths and bond angles in the following residue types are not validated in this section: ACE, Y2Y

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	0.41	0/1099	0.59	0/1478
1	B	0.41	0/1132	0.57	0/1518
2	C	0.42	0/766	0.57	0/1039
2	D	0.45	0/771	0.57	0/1045
3	E	0.54	0/38	0.59	0/50
3	F	0.65	0/38	0.52	0/50
All	All	0.42	0/3844	0.58	0/5180

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
3	E	0	2
3	F	0	2
All	All	0	4

There are no bond length outliers.

There are no bond angle outliers.

There are no chirality outliers.

All (4) planarity outliers are listed below:

Mol	Chain	Res	Type	Group
3	E	404	VAL	Peptide
3	E	405	Y2Y	Mainchain
3	F	504	VAL	Peptide
3	F	505	Y2Y	Mainchain

## 5.2 Too-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 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	1081	0	1047	25	0
1	B	1108	0	1088	12	0
2	C	740	0	701	19	0
2	D	748	0	709	5	0
3	E	54	0	37	4	0
3	F	54	0	37	0	0
4	A	32	0	0	0	0
4	B	43	0	0	2	0
4	C	16	0	0	2	0
4	D	32	0	0	0	0
4	E	3	0	0	0	0
4	F	5	0	0	0	0
All	All	3916	0	3619	53	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 7.

The worst 5 of 53 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:100:MET:HG3	1:A:139:ILE:HG23	1.68	0.75
1:A:48:ILE:HD11	1:A:103:VAL:HG11	1.68	0.74
1:A:170[A]:CYS:SG	2:C:259:LYS:NZ	2.64	0.71
2:D:207:ARG:HA	2:D:213:SER:HA	1.74	0.70
1:A:74:LEU:HD11	2:C:216:ILE:HD12	1.77	0.67

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	139/175 (79%)	134 (96%)	5 (4%)	0	100	100
1	B	141/175 (81%)	138 (98%)	3 (2%)	0	100	100
2	C	89/110 (81%)	88 (99%)	1 (1%)	0	100	100
2	D	90/110 (82%)	89 (99%)	1 (1%)	0	100	100
3	E	5/8 (62%)	4 (80%)	1 (20%)	0	100	100
3	F	5/8 (62%)	5 (100%)	0	0	100	100
All	All	469/586 (80%)	458 (98%)	11 (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	118/159 (74%)	116 (98%)	2 (2%)	60	72
1	B	125/159 (79%)	125 (100%)	0	100	100
2	C	77/98 (79%)	75 (97%)	2 (3%)	46	55
2	D	78/98 (80%)	77 (99%)	1 (1%)	69	79
3	E	3/3 (100%)	3 (100%)	0	100	100
3	F	3/3 (100%)	3 (100%)	0	100	100
All	All	404/520 (78%)	399 (99%)	5 (1%)	69	81

All (5) residues with a non-rotameric sidechain are listed below:

Mol	Chain	Res	Type
1	A	56	HIS
1	A	58	SER
2	C	211	ASP
2	C	268	MET
2	D	268	MET

Sometimes sidechains can be flipped to improve hydrogen bonding and reduce clashes. There are no such sidechains identified.

### 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](#)

There are no ligands in this entry.

## 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<sup>th</sup> 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 <sup>2</sup> )	Q<0.9
1	A	140/175 (80%)	0.28	3 (2%) 63 64	15, 34, 47, 65	0
1	B	140/175 (80%)	-0.17	1 (0%) 87 88	12, 26, 41, 60	0
2	C	90/110 (81%)	0.32	2 (2%) 62 62	12, 28, 44, 51	0
2	D	92/110 (83%)	-0.06	0 100 100	8, 19, 35, 44	0
3	E	6/8 (75%)	0.79	1 (16%) 1 1	29, 41, 45, 47	0
3	F	6/8 (75%)	0.51	1 (16%) 1 1	19, 30, 39, 40	0
All	All	474/586 (80%)	0.10	8 (1%) 70 70	8, 28, 44, 65	0

The worst 5 of 8 RSRZ outliers are listed below:

Mol	Chain	Res	Type	RSRZ
3	E	408	ALA	4.4
3	F	508	ALA	3.8
2	C	254	ALA	3.4
2	C	252	PHE	3.2
1	A	170[A]	CYS	2.5

### 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](#)

There are no ligands in this entry.

## 6.5 Other polymers [i](#)

There are no such residues in this entry.