

Full wwPDB X-ray Structure Validation Report (i)

Sep 5, 2023 – 02:22 AM EDT

PDB ID	:	3U69
Title	:	Unliganded wild-type human thrombin
Authors	:	Figueiredo, A.C.; Clement, C.C.; Philipp, M.; Barbosa Pereira, P.J.
Deposited on		
Resolution	:	1.55 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 (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:

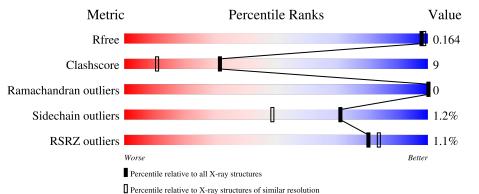
MolProbity	:	4.02b-467
Mogul	:	1.8.5 (274361), CSD as541be (2020)
Xtriage (Phenix)	:	1.13
EDS	:	2.35
Percentile statistics	:	20191225.v01 (using entries in the PDB archive December 25th 2019)
Refmac	:	5.8.0158
CCP4	:	7.0.044 (Gargrove)
Ideal geometry (proteins)	:	Engh & Huber (2001)
Ideal geometry (DNA, 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: $X\text{-}RAY\;DIFFRACTION$

The reported resolution of this entry is 1.55 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	$egin{array}{c} { m Whole \ archive} \ (\#{ m Entries}) \end{array}$	${f Similar\ resolution}\ (\#{ m Entries,\ resolution\ range}({ m \AA}))$
R_{free}	130704	1483 (1.56-1.56)
Clashscore	141614	1529 (1.56-1.56)
Ramachandran outliers	138981	1498 (1.56-1.56)
Sidechain outliers	138945	1495 (1.56-1.56)
RSRZ outliers	127900	1465 (1.56-1.56)

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								
1	L	30	80%	17%	·					
2	Н	259	% 	12%	·					



2 Entry composition (i)

There are 10 unique types of molecules in this entry. The entry contains 5272 atoms, of which 2477 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.

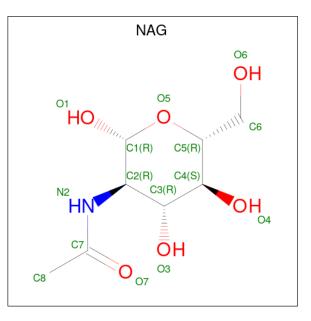
• Molecule 1 is a protein called Prothrombin.

Mol	Chain	Residues	Atoms					ZeroOcc	AltConf	Trace	
1	L	30	Total 533	C 165	Н 267	N 45	O 55	S 1	0	3	0

• Molecule 2 is a protein called Prothrombin.

Mol	Chain	Residues	Atoms					ZeroOcc	AltConf	Trace	
2	Н	251	Total 4269	C 1360	Н 2156	N 371	O 366	S 16	0	14	0

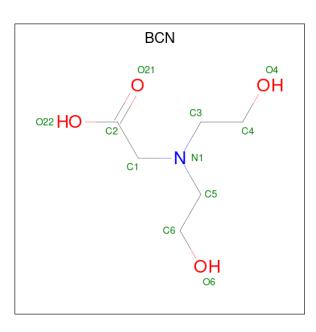
• Molecule 3 is 2-acetamido-2-deoxy-beta-D-glucopyranose (three-letter code: NAG) (formula: $C_8H_{15}NO_6$).



Mol	Chain	Residues	Atoms				ZeroOcc	AltConf
3	Н	1	Total 14	C 8	N 1	O 5	0	0

• Molecule 4 is BICINE (three-letter code: BCN) (formula: $C_6H_{13}NO_4$).





Mol	Chain	Residues	Atoms					ZeroOcc	AltConf
4	4 H	1	Total	С	Η	Ν	Ο	0	0
4		1	23	6	12	1	4		U

• Molecule 5 is SODIUM ION (three-letter code: NA) (formula: Na).

Mol	Chain	Residues	Atoms	ZeroOcc	AltConf
5	Н	1	Total Na 1 1	0	0

• Molecule 6 is IODIDE ION (three-letter code: IOD) (formula: I).

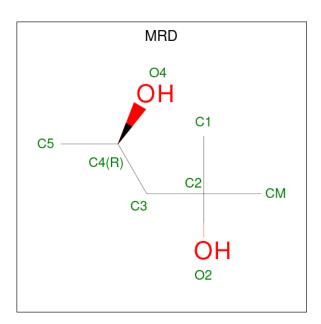
M	ol	Chain	Residues	Atoms	ZeroOcc	AltConf
6		Н	1	Total I 1 1	0	0

• Molecule 7 is CHLORIDE ION (three-letter code: CL) (formula: Cl).

Mol	Chain	Residues	Atoms	ZeroOcc	AltConf
7	Н	1	Total Cl 1 1	0	0

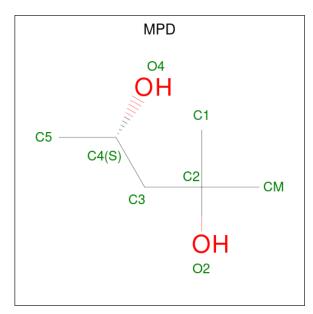
• Molecule 8 is (4R)-2-METHYLPENTANE-2,4-DIOL (three-letter code: MRD) (formula: $C_6H_{14}O_2$).





Mol	Chain	Residues					ZeroOcc	AltConf
8	Н	1	Total 22				0	0
8	Н	1	Total 22				0	0

• Molecule 9 is (4S)-2-METHYL-2,4-PENTANEDIOL (three-letter code: MPD) (formula: $C_6H_{14}O_2$).



Mol	Chain	Residues	Atoms				ZeroOcc	AltConf
9	Н	1	Total 22	С 6	H 14	O 2	0	0



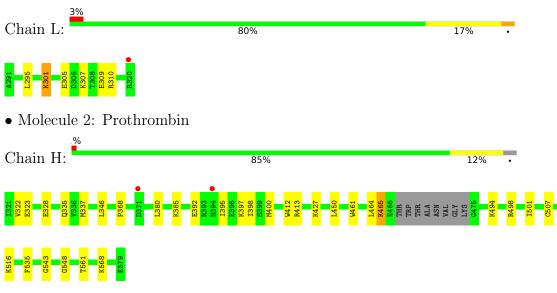
• Molecule 10 is water.

Mol	Chain	Residues	Atoms	ZeroOcc	AltConf
10	L	39	Total O 39 39	0	0
10	Н	325	Total O 325 325	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: Prothrombin



4 Data and refinement statistics (i)

Property	Value	Source
Space group	P 21 21 21	Depositor
Cell constants	57.55Å 73.00Å 83.04Å	Depositor
a, b, c, α , β , γ	90.00° 90.00° 90.00°	Depositor
Resolution (Å)	47.30 - 1.55	Depositor
Resolution (A)	54.83 - 1.55	EDS
% Data completeness	96.5 (47.30-1.55)	Depositor
(in resolution range)	97.8 (54.83-1.55)	EDS
R _{merge}	(Not available)	Depositor
R _{sym}	0.07	Depositor
$< I/\sigma(I) > 1$	$4.25 (at 1.55 \text{\AA})$	Xtriage
Refinement program	PHENIX 1.7_650	Depositor
B B.	0.133 , 0.167	Depositor
R, R_{free}	0.130 , 0.164	DCC
R_{free} test set	2542 reflections $(5.05%)$	wwPDB-VP
Wilson B-factor $(Å^2)$	15.1	Xtriage
Anisotropy	0.267	Xtriage
Bulk solvent $k_{sol}(e/Å^3), B_{sol}(Å^2)$	0.38 , 49.5	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	0.97	EDS
Total number of atoms	5272	wwPDB-VP
Average B, all atoms $(Å^2)$	22.0	wwPDB-VP

Xtriage's analysis on translational NCS is as follows: The largest off-origin peak in the Patterson function is 6.36% of the height of the origin peak. No significant pseudotranslation is detected.

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.



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: NAG, MRD, IOD, CL, MPD, NA, BCN

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	
	Chain	RMSZ	# Z > 5	RMSZ	# Z > 5
1	L	0.39	0/277	0.62	0/366
2	Н	0.45	0/2207	0.64	0/2975
All	All	0.44	0/2484	0.64	0/3341

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	L	266	267	267	4	0
2	Н	2113	2156	2152	35	0
3	Н	14	0	13	0	0
4	Н	11	12	11	1	0
5	Н	1	0	0	0	0
6	Н	1	0	0	1	0
7	Н	1	0	0	0	0
8	Н	16	28	28	7	0
9	Н	8	14	14	0	0
10	Н	325	0	0	10	0
10	L	39	0	0	1	0
All	All	2795	2477	2485	44	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 9.

All (44) 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)
2:H:335[B]:GLN:NE2	10:H:108:HOH:O	2.04	0.87
2:H:398:ILE:O	8:H:700:MRD:H5C1	1.76	0.85
8:H:700:MRD:H1C1	8:H:700:MRD:H5C3	1.62	0.82
2:H:380[A]:LEU:HD22	2:H:400[A]:MET:HE1	1.61	0.81
2:H:498:ARG:HA	2:H:498:ARG:HE	1.47	0.80
2:H:427:LYS:HG2	6:H:2:IOD:I	2.54	0.78
2:H:380[A]:LEU:CD2	2:H:400[A]:MET:HE1	2.14	0.76
2:H:535:PHE:HB3	8:H:701:MRD:HMC1	1.74	0.70
2:H:397:LYS:HG3	10:H:815:HOH:O	1.93	0.69
8:H:701:MRD:H5C3	8:H:701:MRD:H1C1	1.74	0.69
2:H:498:ARG:HA	2:H:498:ARG:NE	2.09	0.68
2:H:380[A]:LEU:HD22	2:H:400[A]:MET:CE	2.23	0.67
2:H:494:LYS:CG	2:H:501[B]:ILE:HD11	2.24	0.67
8:H:700:MRD:H1C3	10:H:814:HOH:O	1.92	0.67
8:H:700:MRD:H5C3	8:H:700:MRD:C1	2.26	0.64
1:L:307:LYS:HG2	2:H:328:GLU:OE2	1.98	0.64
2:H:337[B]:MET:SD	2:H:385:LYS:HD3	2.41	0.61
2:H:337[B]:MET:HG3	2:H:461:TRP:CZ3	2.36	0.61
2:H:494:LYS:HG3	2:H:501[B]:ILE:HD11	1.83	0.60
2:H:395:ILE:HD12	10:H:803:HOH:O	2.02	0.60
2:H:494:LYS:HG2	2:H:501[B]:ILE:HD11	1.85	0.58
2:H:380[A]:LEU:CD2	2:H:400[A]:MET:CE	2.81	0.57
2:H:464:LEU:O	2:H:465:LYS:HD3	2.06	0.56
4:H:1500:BCN:H62	10:H:801:HOH:O	2.07	0.55
2:H:337[B]:MET:CG	2:H:461:TRP:CZ3	2.90	0.55
2:H:568:LYS:HB3	10:H:805:HOH:O	2.08	0.53
2:H:380[A]:LEU:CD2	2:H:400[A]:MET:SD	3.00	0.50
2:H:380[A]:LEU:HD21	2:H:400[A]:MET:HE1	1.94	0.49
2:H:548:GLY:HA3	10:H:818:HOH:O	2.13	0.47
2:H:322:VAL:HG12	2:H:323:GLU:HG2	1.98	0.46
2:H:337[B]:MET:HE3	2:H:346[B]:LEU:HD13	1.98	0.46
1:L:310[B]:ARG:HD2	10:L:151:HOH:O	2.15	0.46
2:H:494:LYS:HG2	2:H:501[B]:ILE:CD1	2.47	0.45
2:H:412:TRP:CZ3	2:H:413:ARG:HG2	2.52	0.45
2:H:516:LYS:HE3	10:H:659:HOH:O	2.16	0.45
1:L:295:LEU:HB2	1:L:301:LYS:HE3	1.99	0.43
8:H:701:MRD:HMC2	10:H:255:HOH:O	2.17	0.43

Continued on next page...



Atom-1	Atom-2	Interatomic distance (Å)	Clash overlap (Å)
1:L:305[B]:GLU:HG2	1:L:309:GLU:CG	2.49	0.43
2:H:543:GLY:HA2	2:H:561:THR:O	2.19	0.43
2:H:392:GLU:HB3	2:H:395:ILE:HG12	2.02	0.42
2:H:368:PRO:HG2	2:H:412:TRP:CZ2	2.55	0.42
2:H:450[B]:LEU:HA	2:H:450[B]:LEU:HD23	1.78	0.41
2:H:337[B]:MET:HG2	2:H:461:TRP:CZ3	2.57	0.40
2:H:568:LYS:CB	10:H:805:HOH:O	2.68	0.40

Continued from previous page...

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	ntiles
1	L	31/30~(103%)	30~(97%)	1 (3%)	0	100	100
2	Н	261/259~(101%)	252 (97%)	9(3%)	0	100	100
All	All	292/289~(101%)	282 (97%)	10 (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	Outliers	Percentiles
1	L	30/27~(111%)	29~(97%)	1 (3%)	38 10

Continued on next page...



Continued from previous page...

Mol	Chain	Analysed	Rotameric	Outliers	Percentiles
2	Н	233/225~(104%)	231~(99%)	2(1%)	78 61
All	All	263/252~(104%)	260~(99%)	3 (1%)	71 53

All (3) residues with a non-rotameric sidechain are listed below:

Mol	Chain	Res	Type
1	L	301	LYS
2	Н	465	LYS
2	Н	507	CYS

Sometimes side chains can be flipped to improve hydrogen bonding and reduce clashes. All (1) such side chains are listed below:

Mol	Chain	Res	Type
2	Н	481	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 monosaccharides in this entry.

5.6 Ligand geometry (i)

Of 8 ligands modelled in this entry, 3 are monoatomic - leaving 5 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	Mol Type Chain R		Res Link		Link Bond lengths			Bond angles		
10101	Mol Type Chain	Chain	nes	LIIIK	Counts	RMSZ	# Z >2	Counts	RMSZ	# Z >2
3	NAG	Н	1373	2	$14,\!14,\!15$	0.52	0	17,19,21	0.73	1 (5%)
8	MRD	Н	700	-	7,7,7	0.36	0	9,10,10	0.37	0
4	BCN	Н	1500	-	10,10,10	0.82	0	11,11,11	1.71	4 (36%)
8	MRD	Н	701	-	7,7,7	0.21	0	9,10,10	0.55	0
9	MPD	Н	702	-	7,7,7	0.35	0	9,10,10	0.20	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
3	NAG	Н	1373	2	-	0/6/23/26	0/1/1/1
8	MRD	Н	700	-	-	1/5/5/5	-
4	BCN	Н	1500	-	-	2/10/10/10	-
8	MRD	Н	701	-	-	1/5/5/5	-
9	MPD	Н	702	-	-	0/5/5/5	-

There are no bond length outliers.

All (5) bond angle outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	$Observed(^{o})$	$\mathbf{Ideal}(^{o})$
4	Н	1500	BCN	O4-C4-C3	2.82	122.88	111.19
4	Н	1500	BCN	C4-C3-N1	2.35	121.84	113.40
4	Н	1500	BCN	C5-N1-C3	2.32	116.96	111.44
4	Н	1500	BCN	C1-N1-C5	-2.22	106.55	111.94
3	Н	1373	NAG	O5-C5-C6	2.09	110.48	107.20

There are no chirality outliers.

All (4) torsion outliers are listed below:

Mol	Chain	Res	Type	Atoms
8	Н	700	MRD	C2-C3-C4-O4
8	Н	701	MRD	C2-C3-C4-O4
4	Н	1500	BCN	C6-C5-N1-C3
4	Н	1500	BCN	C6-C5-N1-C1

There are no ring outliers.

3 monomers are involved in 8 short contacts:



Mol	Chain	Res	Type	Clashes	Symm-Clashes
8	Н	700	MRD	4	0
4	Н	1500	BCN	1	0
8	Н	701	MRD	3	0

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	$\langle RSRZ \rangle$	#RSRZ>2	$OWAB(Å^2)$	Q<0.9
1	L	30/30~(100%)	0.00	1 (3%) 46 54	12, 20, 34, 38	0
2	Н	251/259~(96%)	-0.35	2 (0%) 86 89	7, 16, 33, 50	0
All	All	281/289~(97%)	-0.32	3 (1%) 80 84	7, 16, 33, 50	0

All (3) RSRZ outliers are listed below:

Mol	Chain	Res	Type	RSRZ
2	Н	394	ASN	2.6
2	Н	371	ASP	2.6
1	L	320	ARG	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, 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	$\mathbf{B} ext{-factors}(\mathrm{\AA}^2)$	Q<0.9
3	NAG	Н	1373	14/15	0.71	0.16	49,56,61,61	0
9	MPD	Н	702	8/8	0.79	0.21	40,56,69,69	0

Continued on next page...



Mol	Type	Chain	Res	Atoms	RSCC	RSR	$B-factors(Å^2)$	Q<0.9
4	BCN	Н	1500	11/11	0.86	0.16	12,24,40,42	23
8	MRD	Н	701	8/8	0.89	0.14	$25,\!41,\!59,\!65$	0
8	MRD	Н	700	8/8	0.92	0.20	24,34,69,81	0
6	IOD	Н	2	1/1	0.98	0.08	27,27,27,27	1
5	NA	Н	1	1/1	0.99	0.08	13,13,13,13	0
7	CL	Н	3	1/1	1.00	0.07	20,20,20,20	0

Continued from previous page...

6.5 Other polymers (i)

There are no such residues in this entry.

