

# Full wwPDB X-ray Structure Validation Report (i)

#### Aug 27, 2020 - 03:24 PM BST

PDB ID	:	6ZUX
Title	:	Crystal Structure of Thrombin in complex with compound42a
Authors	:	Schafer, M.
Deposited on		
Resolution	:	1.94 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 following versions of software and data (see references (1)) were used in the production of this report:

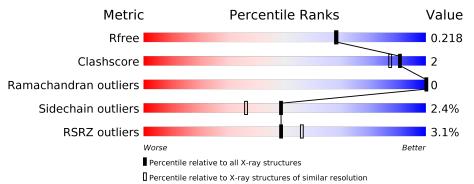
MolProbity	:	4.02b-467
Mogul	:	1.8.5 (274361), CSD as541be (2020)
Xtriage (Phenix)	:	1.13
$\mathrm{EDS}$	:	2.13
buster-report	:	1.1.7 (2018)
Percentile statistics	:	20191225.v01 (using entries in the PDB archive December 25th 2019)
$\operatorname{Refmac}$	:	5.8.0158
$\operatorname{CCP4}$	:	$7.0.044 (\mathrm{Gargrove})$
Ideal geometry (proteins)	:	Engh & Huber (2001)
Ideal geometry (DNA, RNA)	:	Parkinson et al. (1996)
Validation Pipeline (wwPDB-VP)	:	2.13

# 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 1.94 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	$egin{array}{c} { m Whole \ archive} \ (\#{ m Entries}) \end{array}$	${f Similar\ resolution}\ (\#{ m Entries},{ m resolution\ range}({ m \AA}))$
$R_{free}$	130704	4310 (1.96-1.92)
Clashscore	141614	1023 (1.94-1.94)
Ramachandran outliers	138981	1007 (1.94-1.94)
Sidechain outliers	138945	1007 (1.94-1.94)
RSRZ outliers	127900	4250 (1.96-1.92)

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 <=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	L	36	75%	25%
2	Н	259	2% 88%	7% ••
3	Ι	11	91%	9%



# 2 Entry composition (i)

There are 6 unique types of molecules in this entry. The entry contains 2685 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 Prothrombin.

Mol	Chain	Residues	Atoms			ZeroOcc	AltConf	Trace		
1	L	27	Total 222	C 140	N 36	O 45	${ m S}$	0	0	0

• Molecule 2 is a protein called Prothrombin.

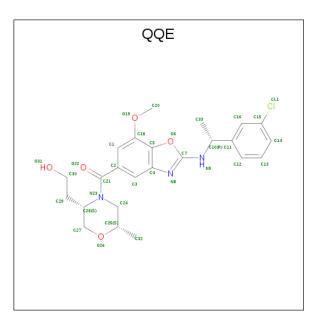
M	ol Chair	n Residues	Atoms			ZeroOcc	AltConf	Trace		
2	Н	250	Total 2025	C 1292	N 358	O 361	S 14	0	0	0

• Molecule 3 is a protein called Hirudin-2.

Mol	Chain	Residues	Atoms			ZeroOcc	AltConf	Trace		
3	Ι	11	Total 99	С 61	N 11	O 26	S 1	0	0	0

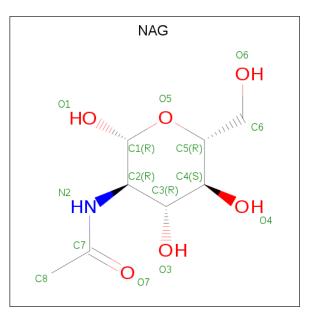
• Molecule 4 is [2-[[(1 {R})-1-(3-chlorophenyl)ethyl]amino]-7-methoxy-1,3-benzoxazol-5-yl]-[ (2 {S},5 {S})-5-(2-hydroxyethyl)-2-methyl-morpholin-4-yl]methanone (three-letter code: QQE) (formula: C<sub>24</sub>H<sub>28</sub>ClN<sub>3</sub>O<sub>5</sub>) (labeled as "Ligand of Interest" by author).





Mol	Chain	Residues		$\mathbf{At}$	$\mathbf{oms}$			ZeroOcc	AltConf
4	н	1	Total	С	$\operatorname{Cl}$	Ν	Ο	Ο	Ο
T	11	L	33	24	1	3	5	0	0

• Molecule 5 is 2-acetamido-2-deoxy-beta-D-glucopyranose (three-letter code: NAG) (formula:  $C_8H_{15}NO_6$ ).



Mol	Chain	Residues	A	ton	ns		ZeroOcc	AltConf
5	Н	1	Total 14	C 8	N 1	O 5	0	0

• Molecule 6 is water.

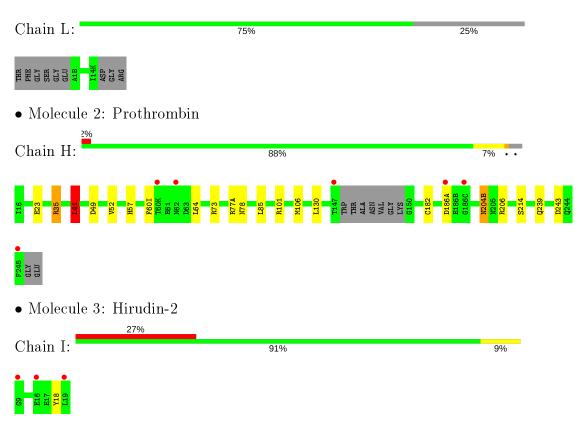


Mol	Chain	Residues	Atoms	ZeroOcc	AltConf
6	L	33	Total O 33 33	0	0
6	Н	248	Total         O           248         248	0	0
6	Ι	11	Total O 11 11	0	0



# 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	C 1 2 1	Depositor
Cell constants	70.26Å 71.30Å 72.45Å	Depositor
a, b, c, $\alpha$ , $\beta$ , $\gamma$	$90.00^{\circ}$ $100.43^{\circ}$ $90.00^{\circ}$	Depositor
Resolution (Å)	71.25 - 1.94	Depositor
Resolution (A)	38.44 - 1.94	EDS
% Data completeness	96.9 (71.25-1.94)	Depositor
(in resolution range)	96.9(38.44-1.94)	EDS
R <sub>merge</sub>	(Not available)	Depositor
R <sub>sym</sub>	0.06	Depositor
$< I/\sigma(I) > 1$	2.79 (at $1.94$ Å)	Xtriage
Refinement program	REFMAC 5.8.0158	Depositor
D D.	0.162 , $0.206$	Depositor
$R, R_{free}$	0.176 , $0.218$	DCC
$R_{free}$ test set	1295 reflections $(5.11%)$	wwPDB-VP
Wilson B-factor $(Å^2)$	15.6	Xtriage
Anisotropy	0.253	Xtriage
Bulk solvent $k_{sol}(e/Å^3), B_{sol}(Å^2)$	0.42, 58.0	EDS
L-test for twinning <sup>2</sup>	$ \langle L  \rangle = 0.49, \langle L^2 \rangle = 0.32$	Xtriage
Estimated twinning fraction	No twinning to report.	Xtriage
$F_o, F_c$ correlation	0.94	EDS
Total number of atoms	2685	wwPDB-VP
Average B, all atoms $(Å^2)$	28.0	wwPDB-VP

Xtriage's analysis on translational NCS is as follows: The largest off-origin peak in the Patterson function is 7.35% of the height of the origin peak. No significant pseudotranslation is detected.

<sup>&</sup>lt;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.



<sup>&</sup>lt;sup>1</sup>Intensities 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: QQE, NAG, TYS

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	Bo	nd lengths	Bond angles		
	Cham	RMSZ	# Z  > 5	RMSZ	# Z  > 5	
1	L	1.08	0/224	1.03	0/298	
2	Н	0.98	1/2077~(0.0%)	1.06	10/2806~(0.4%)	
3	Ι	0.95	0/83	1.03	0/108	
All	All	0.99	1/2384~(0.0%)	1.05	10/3212~(0.3%)	

All (1) bond length outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(Å)	Ideal(Å)
2	H	23	GLU	CD-OE1	-5.24	1.19	1.25

Mol	Chain	Res	Type	Atoms	Z	$\mathbf{Observed}(^{o})$	$Ideal(^{o})$
2	Н	35	ARG	NE-CZ-NH2	8.47	124.53	120.30
2	Н	35	ARG	NE-CZ-NH1	-7.57	116.52	120.30
2	Н	73	ARG	NE-CZ-NH1	6.37	123.48	120.30
2	Н	101	ARG	NE-CZ-NH2	-6.30	117.15	120.30
2	Н	186(A)	ASP	CB-CG-OD1	6.29	123.96	118.30
2	Н	243	ASP	CB-CG-OD2	-6.26	112.66	118.30
2	Н	243	ASP	CB-CG-OD1	5.65	123.39	118.30
2	Н	49	ASP	CB-CG-OD2	-5.40	113.44	118.30
2	Н	206	ARG	NE-CZ-NH2	-5.38	117.61	120.30
2	Н	41	LEU	CA-CB-CG	5.25	127.36	115.30

All (10) bond angle outliers are listed below:

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	222	0	225	0	0
2	Н	2025	0	2000	8	0
3	Ι	99	0	77	0	0
4	Н	33	0	0	0	0
5	Н	14	0	13	0	0
6	Н	248	0	0	1	0
6	Ι	11	0	0	0	0
6	L	33	0	0	0	0
All	All	2685	0	2315	8	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 2.

All (8) 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)
2:H:204(B):ASN:C	2:H:204(B):ASN:HD22	2.18	0.47
2:H:35:ARG:HB2	2:H:41:LEU:HD23	1.97	0.46
2:H:78:ASN:HD22	2:H:78:ASN:N	2.12	0.46
2:H:85:LEU:HD22	2:H:106:MET:HB3	1.98	0.45
2:H:41:LEU:HD21	2:H:60(I):PHE:CZ	2.53	0.43
2:H:52:VAL:HG12	2:H:106:MET:HE3	2.01	0.42
2:H:77(A):ARG:HD2	6:H:1254:HOH:O	2.19	0.42
2:H:57:HIS:HE1	2:H:214:SER:O	2.02	0.42

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.



Mol	Chain	Analysed	Favoured	Allowed	Outliers	rs   Percentile	
1	L	25/36~(69%)	24~(96%)	1 (4%)	0	100	100
2	Н	246/259~(95%)	237~(96%)	9~(4%)	0	100	100
3	Ι	8/11 (73%)	8 (100%)	0	0	100	100
All	All	279/306~(91%)	269~(96%)	10 (4%)	0	100	100

The Analysed column shows the number of residues for which the backbone conformation was analysed, and the total number of residues.

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	25/31~(81%)	25~(100%)	0	100 100
2	Η	219/225~(97%)	213~(97%)	6(3%)	44 31
3	Ι	9/9~(100%)	9~(100%)	0	100 100
All	All	253/265~(96%)	247 (98%)	6 (2%)	49 36

All (6) residues with a non-rotameric sidechain are listed below:

Mol	Chain	Res	Type
2	Н	41	LEU
2	Н	64	LEU
2	Н	130	LEU
2	Н	182	CYS
2	Н	204(B)	ASN
2	Н	239	GLN

Some sidechains can be flipped to improve hydrogen bonding and reduce clashes. All (3) such sidechains are listed below:

Mol	Chain	$\mathbf{Res}$	Type
2	Н	78	ASN

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Mol	Chain	Res	Type
2	Η	204(B)	ASN
2	Н	244	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	Dog	Link Bond lengths			Bond angles			
IVIOI	туре	Ullalli	nes		Counts	RMSZ	# Z  > 2	Counts	RMSZ	# Z >2
3	TYS	Ι	18	3	15, 16, 17	1.44	2 (13%)	18,22,24	1.23	2 (11%)

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	$\mathbf{Res}$	$\mathbf{Link}$	Chirals	Torsions	Rings
3	TYS	Ι	18	3	-	0/10/11/13	0/1/1/1

All (2) bond length outliers are listed below:

Mol	Chain	$\mathbf{Res}$	Type	Atoms	Z	$\operatorname{Observed}(\operatorname{\AA})$	Ideal(Å)
3	Ι	18	TYS	OH-CZ	-4.45	1.35	1.42
3	Ι	18	TYS	O3-S	2.08	1.63	1.50

All (2) bond angle outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	$Observed(^{o})$	$Ideal(^{o})$
3	Ι	18	TYS	O3-S-O1	2.66	117.75	108.49

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Mol	Chain	$\mathbf{Res}$	Type	Atoms	Z	$Observed(^{o})$	$Ideal(^{o})$
3	Ι	18	TYS	CB-CA-C	-2.40	106.98	111.47

There are no chirality outliers.

There are no torsion outliers.

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)

2 ligands are 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).

Mal	Mol Type Chain Re		Res	Link	Bond lengths			Bond angles		
	Moi Type Chain	nes		Counts	RMSZ	# Z  > 2	Counts	RMSZ	# Z  > 2	
4	QQE	Н	1001	-	33,36,36	1.67	6 (18%)	$33,\!51,\!51$	1.65	10 (30%)
5	NAG	Н	1002	2	14,14,15	0.57	0	17,19,21	1.68	<mark>5 (29%)</mark>

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	$\mathbf{Res}$	Link	Chirals	Torsions	Rings
4	QQE	Н	1001	-	-	2/19/34/34	0/4/4/4
5	NAG	Н	1002	2	-	4/6/23/26	0/1/1/1

All (6) bond length outliers are listed below:



Mol	Chain	Res	Type	Atoms	Z	$\operatorname{Observed}(\operatorname{\AA})$	Ideal(Å)
4	Н	1001	QQE	C7-N9	-4.11	1.28	1.35
4	Н	1001	QQE	O26-C27	-3.76	1.37	1.43
4	Н	1001	QQE	C3-C2	3.43	1.43	1.37
4	Н	1001	QQE	O19-C18	3.16	1.42	1.36
4	Н	1001	QQE	C24-N23	2.25	1.49	1.46
4	Н	1001	QQE	C2-C21	2.01	1.53	1.50

All (15) bond angle outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	$Observed(^{o})$	$Ideal(^{o})$
4	Н	1001	QQE	C33-C10-C11	-3.61	104.01	112.25
5	Н	1002	NAG	C8-C7-N2	3.35	121.78	116.10
4	Н	1001	QQE	O26-C25-C32	-2.77	103.76	107.64
4	Н	1001	QQE	C16-C15-CL1	2.75	122.58	119.15
4	Н	1001	QQE	C14-C15-C16	-2.49	118.22	121.53
4	Н	1001	QQE	O31-C30-C29	-2.42	102.84	111.31
5	Н	1002	NAG	O4-C4-C5	2.39	115.24	109.30
5	Н	1002	NAG	O5-C1-C2	-2.25	107.74	111.29
4	Н	1001	QQE	C3-C4-C5	-2.22	118.88	121.12
4	Н	1001	QQE	O26-C25-C24	-2.22	103.76	110.58
4	Н	1001	QQE	O22-C21-C2	-2.19	115.97	120.23
4	Н	1001	QQE	C12-C13-C14	2.14	123.29	120.25
5	Н	1002	NAG	C3-C4-C5	-2.13	106.44	110.24
4	Н	1001	QQE	C15-C16-C11	2.11	122.21	119.42
5	Н	1002	NAG	C1-O5-C5	2.03	114.94	112.19

There are no chirality outliers.

All (6) torsion outliers are listed below:

Mol	Chain	Res	Type	Atoms
4	Н	1001	QQE	C28-C29-C30-O31
5	Н	1002	NAG	O5-C5-C6-O6
5	Н	1002	NAG	C4-C5-C6-O6
5	Н	1002	NAG	C8-C7-N2-C2
5	Н	1002	NAG	O7-C7-N2-C2
4	Н	1001	QQE	N23-C28-C29-C30

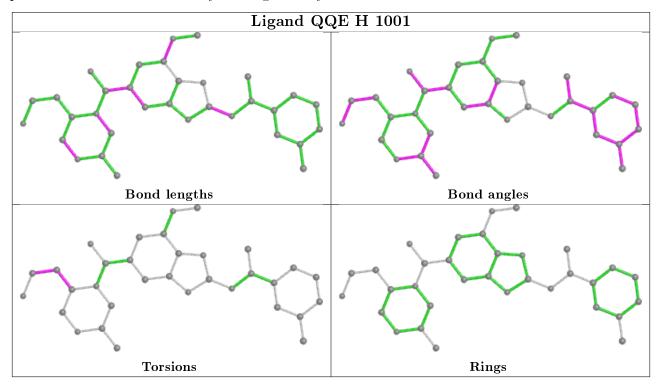
There are no ring outliers.

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 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.



#### 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	$\mathbf{OWAB}(\mathbf{\AA}^2)$	Q<0.9
1	L	27/36~(75%)	-0.26	0 100 100	19, 23, 35, 40	1 (3%)
2	Н	250/259~(96%)	-0.00	6 (2%) 59 66	17, 25, 40, 58	1 (0%)
3	Ι	$10/11 \ (90\%)$	1.64	3 (30%) 0 0	41, 51, 63, 66	0
All	All	287/306~(93%)	0.03	9 (3%) 49 56	17, 25, 44, 66	2(0%)

All (9) RSRZ outliers are listed below:

Mol	Chain	Res	Type	RSRZ
3	Ι	9	GLY	4.7
3	Ι	19	LEU	4.4
2	Н	147	THR	3.8
2	Н	62	ASN	3.4
2	Н	245	PHE	2.8
3	Ι	16	GLU	2.6
2	Н	60(K)	THR	2.6
2	Н	186(A)	ASP	2.3
2	Н	186(C)	GLY	2.1

## 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.

Mol	Type	Chain	Res	Atoms	RSCC	RSR	${f B} ext{-factors}({ m \AA}^2)$	Q<0.9
3	TYS	Ι	18	16/17	0.96	0.12	$38,\!39,\!44,\!45$	0



### 6.3 Carbohydrates (i)

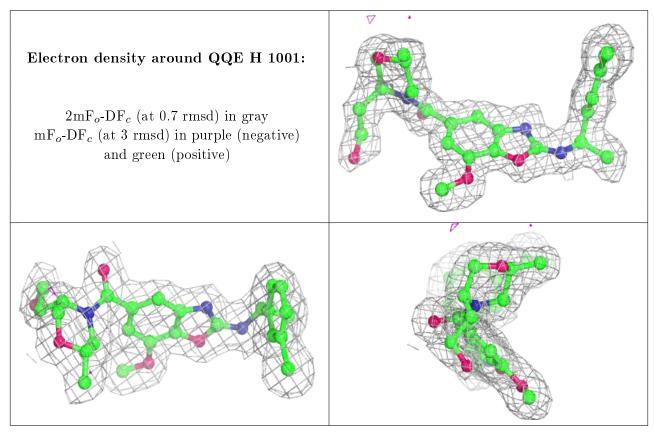
There are no monosaccharides in this entry.

#### 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	$\mathbf{B} ext{-factors}(\mathbf{A}^2)$	$\mathbf{Q}{<}0.9$
5	NAG	Н	1002	14/15	0.72	0.30	$47,\!54,\!57,\!63$	0
4	QQE	Н	1001	33/33	0.96	0.10	19,22,31,42	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.

