

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

#### Nov 11, 2023 – 08:08 am GMT

PDB ID : 4Y10

Title: Trypsin in complex with with BPTI mutant (2S)-2-amino-4,4-difluorobutanoi

c acid

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Deposited on : 2015-02-06

Resolution : 1.37 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 : 4.02b-467

Mogul : 1.8.4, CSD as541be (2020)

Xtriage (Phenix) : 1.13 EDS : 2.36

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)

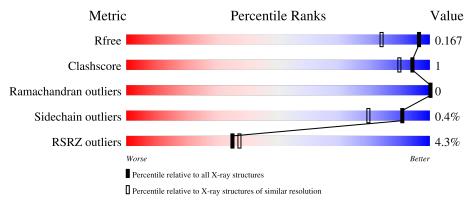
Validation Pipeline (wwPDB-VP) : 2.36

## 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 1.37 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 $(\# \text{Entries})$	Similar resolution $(\#\text{Entries, resolution range}(\mathring{\mathbf{A}}))$
$R_{free}$	130704	2907 (1.40-1.36)
Clashscore	141614	3037 (1.40-1.36)
Ramachandran outliers	138981	2970 (1.40-1.36)
Sidechain outliers	138945	2969 (1.40-1.36)
RSRZ outliers	127900	2846 (1.40-1.36)

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	Е	223	96%			
2	I	58	90%	7% • •		



## 2 Entry composition (i)

There are 6 unique types of molecules in this entry. The entry contains 4809 atoms, of which 2208 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 Cationic trypsin.

Mol	Chain	Residues	Atoms				ZeroOcc	AltConf	Trace		
1	E	223	Total 3440	C 1077	H 1721	N 286	O 342	S 14	0	21	0

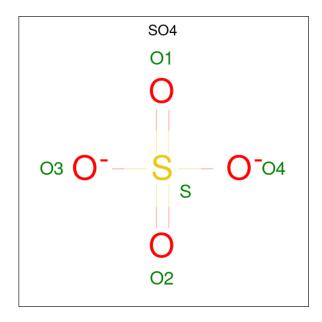
• Molecule 2 is a protein called Pancreatic trypsin inhibitor.

Mol	Chain	Residues	Atoms				ZeroOcc	AltConf	Trace			
2	Т	57	Total	С	F	Н	N	О	S	0	7	0
2	1	31	963	307	2	479	84	83	8	0	'	

There is a discrepancy between the modelled and reference sequences:

Chain	Residue	Modelled	Actual	Comment	Reference
I	15	OBF	LYS	engineered mutation	UNP P00974

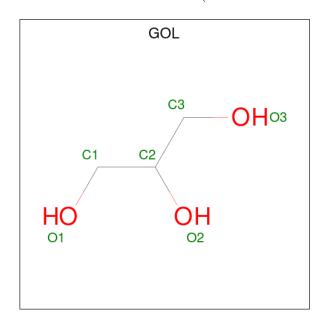
• Molecule 3 is SULFATE ION (three-letter code: SO4) (formula: O<sub>4</sub>S).





Mol	Chain	Residues	Atoms	ZeroOcc	AltConf
3	Е	1	Total O S	0	0
	П	1	5 4 1	0	U
3	E	1	Total O S	0	0
	П	1	5 4 1	· ·	Ŭ
3	E	1	Total O S	0	0
	L	1	5 4 1	0	Ŭ
3	E	1	Total O S	0	0
	L	1	5 4 1	Ŭ	Ü
3	E	1	Total O S	0	0
		1	5 4 1	Ŭ	Ü
3	E	1	Total O S	0	0
		_	5 4 1	Ü	Ü
3	I	1	Total O S	0	0
	-	_	5 4 1	Ü	Ü
3	I	1	Total O S	0	0
	*	_	5 4 1	ļ ,	
3	I	1	Total O S	0	0
	1	1	5 4 1		

• Molecule 4 is GLYCEROL (three-letter code: GOL) (formula: C<sub>3</sub>H<sub>8</sub>O<sub>3</sub>).



Mol	Chain	Residues	Atoms				ZeroOcc	AltConf
1	E	1	Total	С	Н	О	0	0
4	12	1	14	3	8	3	0	0

• Molecule 5 is CALCIUM ION (three-letter code: CA) (formula: Ca).



Mol	Chain	Residues	Atoms	ZeroOcc	AltConf
5	E	1	Total Ca 1 1	0	0

## $\bullet\,$ Molecule 6 is water.

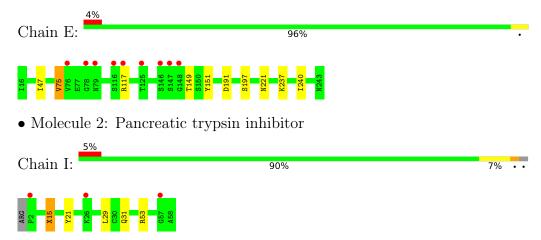
Mol	Chain	Residues	Atoms	ZeroOcc	AltConf
6	Е	263	Total O 263 263	0	5
6	I	83	Total O 83 83	0	1



# 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: Cationic trypsin





# 4 Data and refinement statistics (i)

Property	Value	Source
Space group	I 2 2 2	Depositor
Cell constants	74.69Å 81.85Å 123.61Å	Donositor
a, b, c, $\alpha$ , $\beta$ , $\gamma$	90.00° 90.00° 90.00°	Depositor
Resolution (Å)	34.12 - 1.37	Depositor
Resolution (A)	34.47 - 1.37	EDS
% Data completeness	99.6 (34.12-1.37)	Depositor
(in resolution range)	99.6 (34.47-1.37)	EDS
$R_{merge}$	0.07	Depositor
$R_{sym}$	(Not available)	Depositor
$< I/\sigma(I) > 1$	1.46 (at 1.37Å)	Xtriage
Refinement program	PHENIX (phenix.refine: 1.9_1692)	Depositor
D D	0.138 , 0.165	Depositor
$R, R_{free}$	0.140 , 0.167	DCC
$R_{free}$ test set	3992 reflections $(5.00\%)$	wwPDB-VP
Wilson B-factor (Å <sup>2</sup> )	13.5	Xtriage
Anisotropy	0.448	Xtriage
Bulk solvent $k_{sol}(e/Å^3)$ , $B_{sol}(Å^2)$	0.45 , 48.7	EDS
L-test for twinning <sup>2</sup>	$< L >=0.49, < L^2>=0.33$	Xtriage
Estimated twinning fraction	No twinning to report.	Xtriage
$F_o, F_c$ correlation	0.98	EDS
Total number of atoms	4809	wwPDB-VP
Average B, all atoms (Å <sup>2</sup> )	20.0	wwPDB-VP

Xtriage's analysis on translational NCS is as follows: The largest off-origin peak in the Patterson function is 5.18% of the height of the origin peak. No significant pseudotranslation is detected.

<sup>&</sup>lt;sup>2</sup>Theoretical 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.



<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: SO4, GOL, CA, OBF

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		nd lengths	Bond angles		
IVIOI	Chain	RMSZ	# Z  > 5	RMSZ	# Z  > 5	
1	Е	0.77	1/1819 (0.1%)	0.79	3/2467 (0.1%)	
2	I	0.84	0/507	0.94	1/676 (0.1%)	
All	All	0.78	1/2326 (0.0%)	0.83	4/3143 (0.1%)	

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 maintain group or atoms of a sidechain that are expected to be planar.

Mol	Chain	#Chirality outliers	#Planarity outliers
1	Ε	0	1

#### All (1) bond length outliers are listed below:

Mol	Chain	Res	Type	Atoms	$\mathbf{Z}$	$\operatorname{Observed}(\text{\AA})$	$\operatorname{Ideal}( ext{\AA})$
1	Е	75	VAL	C-O	-10.53	1.03	1.23

#### All (4) bond angle outliers are listed below:

Mol	Chain	Res	Type	Atoms	$\mathbf{Z}$	$Observed(^o)$	$\operatorname{Ideal}(^{o})$
2	I	53	ARG	NE-CZ-NH2	-7.37	116.62	120.30
1	Е	117	ARG	NE-CZ-NH2	-7.25	116.67	120.30
1	Е	75	VAL	CA-C-N	-7.03	101.75	117.20
1	Е	191	ASP	CB-CG-OD1	6.07	123.76	118.30

There are no chirality outliers.

All (1) planarity outliers are listed below:



Mol	Chain	Res	Type	Group
1	E	75	VAL	Mainchain

### 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	Ε	1719	1721	1699	4	1
2	I	484	479	480	3	1
3	Ε	30	0	0	0	0
3	I	15	0	0	0	0
4	Ε	6	8	8	0	0
5	Ε	1	0	0	0	0
6	Е	263	0	0	0	0
6	I	83	0	0	0	0
All	All	2601	2208	2187	6	2

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 1.

All (6) close contacts within the same asymmetric unit are listed below, sorted by their clash magnitude.

Atom-1	Atom-2	Interatomic	Clash
Atom-1	Atom-2	$\operatorname{distance}\ ( ext{\AA})$	overlap (Å)
1:E:47:ILE:HD11	1:E:240:ILE:HD11	1.75	0.67
1:E:149[A]:THR:HG23	1:E:151[A]:TYR:CE2	2.41	0.55
1:E:197:SER:OG	2:I:15:OBF:C	2.60	0.50
1:E:149[A]:THR:CG2	1:E:151[A]:TYR:CE2	2.98	0.46
2:I:31[B]:GLN:OE1	2:I:31[B]:GLN:HA	2.16	0.45
2:I:31[B]:GLN:OE1	2:I:31[B]:GLN:CA	2.68	0.41

All (2) 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	$\begin{array}{c} {\rm Interatomic} \\ {\rm distance} \ ({\rm \AA}) \end{array}$	Clash overlap (Å)	
2:I:21:TYR:HH	2:I:21:TYR:HH[4_559]	1.28	0.32	
1:E:221:ASN:HD22	1:E:237:LYS:HZ2[8_459]	1.35	0.25	



## 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	ysed Favoured Allowed		Outliers   Percent		entiles
1	E	243/223 (109%)	241 (99%)	2 (1%)	0	100	100
2	I	61/58 (105%)	59 (97%)	2 (3%)	0	100	100
All	All	304/281 (108%)	300 (99%)	4 (1%)	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	Perce	ntiles
1	$\mathbf{E}$	205/184 (111%)	205 (100%)	0	100	100
2	I	51/45~(113%)	50 (98%)	1 (2%)	55	23
All	All	256/229 (112%)	255 (100%)	1 (0%)	91	80

All (1) residues with a non-rotameric sidechain are listed below:

Mol	Chain	Res	Type
2	I	29	LEU

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)

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	LIIIK	Counts	RMSZ	# Z  > 2	Counts	RMSZ	# Z  > 2
2	OBF	I	15	2	5,7,8	0.89	0	2,8,10	1.49	1 (50%)

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	$\operatorname{Res}$	Link	Chirals	Torsions	Rings
2	OBF	I	15	2	-	0/5/6/8	-

There are no bond length outliers.

All (1) bond angle outliers are listed below:

Mol	Chain	Res	Type	Atoms	$\mathbf{Z}$	$\mathbf{Observed}(^{o})$	$\operatorname{Ideal}({}^{o})$
2	I	15	OBF	FG2-CG-FG1	-2.10	100.38	105.53

There are no chirality outliers.

There are no torsion outliers.

There are no ring outliers.

1 monomer is involved in 1 short contact:

$\mathbf{Mol}$	Chain	Res	Type	Clashes	Symm-Clashes
2	I	15	OBF	1	0

## 5.5 Carbohydrates (i)

There are no monosaccharides in this entry.



## 5.6 Ligand geometry (i)

Of 11 ligands modelled in this entry, 1 is monoatomic - leaving 10 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).

Mal	Trino	Chain	Dag	Link	В	ond leng	$_{ m gths}$	Bond angles		
Mol	Type	Chain	Res	es Lilik	Counts	RMSZ	# Z  > 2	Counts	RMSZ	# Z  > 2
3	SO4	I	101	-	4,4,4	0.16	0	6,6,6	0.36	0
3	SO4	Е	306	-	4,4,4	0.20	0	6,6,6	0.33	0
4	GOL	Е	307	-	5,5,5	0.55	0	5,5,5	1.00	0
3	SO4	Е	302	-	4,4,4	0.47	0	6,6,6	0.71	0
3	SO4	Е	301	-	4,4,4	0.24	0	6,6,6	0.42	0
3	SO4	I	103	-	4,4,4	0.30	0	6,6,6	0.37	0
3	SO4	I	102	-	4,4,4	0.53	0	6,6,6	0.44	0
3	SO4	Е	303	-	4,4,4	0.26	0	6,6,6	1.05	1 (16%)
3	SO4	Е	305	-	4,4,4	0.20	0	6,6,6	0.27	0
3	SO4	Е	304	-	4,4,4	0.81	0	6,6,6	0.66	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
4	GOL	E	307	_	-	2/4/4/4	-

There are no bond length outliers.

All (1) bond angle outliers are listed below:

Mol	Chain	Res	Type	Atoms	$\mathbf{Z}$	$Observed(^o)$	$\operatorname{Ideal}({}^{o})$
3	Ε	303	SO4	O4-S-O2	-2.20	97.82	109.31

There are no chirality outliers.

All (2) torsion outliers are listed below:

Mol	Chain	Res	Type	Atoms
4	${ m E}$	307	GOL	O1-C1-C2-C3

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Mol	Chain	Res	Type	Atoms
4	E	307	GOL	O1-C1-C2-O2

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	$\begin{array}{c c} \textbf{Analysed} &  <\!\!\text{RSRZ}\!\!>\!  & \#\!\!\text{RSRZ}\!\!>\!\!2 \end{array}$		$OWAB(Å^2)$	Q<0.9	
1	E	223/223 (100%)	-0.08	9 (4%) 38 40	10, 17, 26, 36	0
2	I	56/58 (96%)	-0.20	3 (5%) 25 26	10, 13, 27, 36	0
All	All	279/281 (99%)	-0.10	12 (4%) 35 37	10, 17, 27, 36	0

All (12) RSRZ outliers are listed below:

Mol	Chain	Res	Type	RSRZ
1	Е	147	SER	4.2
1	Е	146	SER	3.5
1	Е	117	ARG	3.3
1	Е	78	GLY	3.0
2	I	2	PRO	3.0
1	Е	79	ASN	3.0
1	Е	116	SER	2.9
1	Е	76	VAL	2.8
1	Е	125	THR	2.7
2	I	57	GLY	2.5
2	I	26	LYS	2.4
1	Е	148	GLY	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.

$\mathbf{Mol}$	$\mathbf{Type}$	Chain	$\operatorname{Res}$	Atoms	RSCC	RSR	$\mathbf{B} ext{-}\mathbf{factors}(\mathbf{ ilde{A}}^2)$	Q<0.9
2	OBF	I	15	8/9	0.98	0.07	10,13,16,18	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	$\mathbf{B} ext{-}\mathbf{factors}(\mathbf{\mathring{A}}^2)$	Q < 0.9
3	SO4	Е	304	5/5	0.79	0.17	31,32,33,34	5
4	GOL	E	307	6/6	0.79	0.18	37,44,48,49	14
3	SO4	Е	306	5/5	0.84	0.24	34,34,35,35	5
3	SO4	Е	305	5/5	0.91	0.25	33,34,34,34	5
3	SO4	I	102	5/5	0.97	0.08	18,19,24,24	5
3	SO4	Е	303	5/5	0.97	0.09	31,31,32,33	5
3	SO4	E	301	5/5	0.98	0.16	38,38,38,38	5
3	SO4	Е	302	5/5	0.99	0.10	19,19,21,21	5
3	SO4	I	103	5/5	0.99	0.05	14,14,16,17	0
3	SO4	I	101	5/5	0.99	0.07	16,17,21,21	0
5	CA	Е	308	1/1	0.99	0.04	19,19,19,19	0

## 6.5 Other polymers (i)

There are no such residues in this entry.

