

Full wwPDB X-ray Structure Validation Report (i)

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PDB ID	:	5TRU
Title	:	Structure of the first-in-class checkpoint inhibitor Ipilimumab bound to human
		CTLA-4
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Deposited on	:	2016-10-27
Resolution	:	3.00 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 Xtriage (Phenix) EDS Percentile statistics Refmac CCP4 Ideal geometry (proteins) Ideal geometry (DNA_BNA)	:::::::::::::::::::::::::::::::::::::::	4.02b-467 1.13 2.35.1 20191225.v01 (using entries in the PDB archive December 25th 2019) 5.8.0158 7.0.044 (Gargrove) Engh & Huber (2001) Parkingon et al. (1996)
Ideal geometry (DNA, RNA)	:	Parkinson et al. (1996)
valuation r ipenne (wwPDD-VP)	•	2.39.1

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 3.00 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	2092 (3.00-3.00)
Ramachandran outliers	138981	2333 (3.00-3.00)
Sidechain outliers	138945	2336 (3.00-3.00)
RSRZ outliers	127900	1990 (3.00-3.00)

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	L	215	91%	8% •
1	1	215	94%	
2	Н	225	91%	
2	h	225	84%	10% 6%
3	С	118	% 90%	7% •
3	с	118	94%	



5TRU

2 Entry composition (i)

There are 3 unique types of molecules in this entry. The entry contains 8036 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 Ipilimumab Fab light chain.

Mol	Chain	Residues	Atoms				ZeroOcc	AltConf	Trace	
1	т	212	Total	С	Ν	0	\mathbf{S}	0	0	0
1		210	1627	1023	275	325	4	0	0	0
1	1	919	Total	С	Ν	0	S	0	0	0
	1	212	1579	987	271	317	4	0	0	U

• Molecule 2 is a protein called Ipilimumab Fab heavy chain.

Mol	Chain	Residues	Atoms				ZeroOcc	AltConf	Trace	
2	Ц	215	Total	С	Ν	Ο	S	0	0	0
	11	210	1619	1030	270	313	6	0	0	0
0	h	919	Total	С	Ν	0	S	0	0	0
	11	212	1589	1011	266	306	6	0	0	0

• Molecule 3 is a protein called Cytotoxic T-lymphocyte protein 4.

Mol	Chain	Residues	Atoms			ZeroOcc	AltConf	Trace		
2	0	115	Total	С	Ν	0	S	0	0	0
0	C	110	816	515	134	158	9	0	0	0
2	C	114	Total	С	Ν	0	S	0	0	0
0		114	806	510	132	155	9	0		U



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.

- Chain L: 8% 91% • Molecule 1: Ipilimumab Fab light chain Chain l: 94% • Molecule 2: Ipilimumab Fab heavy chain Chain H: 91% SER SER GLY GLY • Molecule 2: Ipilimumab Fab heavy chain Chain h: 84% 10% 6% LYS SER SER SER GLY GLY • Molecule 3: Cytotoxic T-lymphocyte protein 4 Chain c: 94% • Molecule 3: Cytotoxic T-lymphocyte protein 4 Chain C: 90% 7%
- Molecule 1: Ipilimumab Fab light chain





4 Data and refinement statistics (i)

Property	Value	Source
Space group	C 2 2 21	Depositor
Cell constants	95.84Å 197.50Å 148.12Å	Depositor
a, b, c, α , β , γ	90.00° 90.00° 90.00°	Depositor
Bosolution (Å)	34.91 - 3.00	Depositor
Resolution (A)	34.91 - 3.00	EDS
% Data completeness	99.2 (34.91-3.00)	Depositor
(in resolution range)	99.3 (34.91-3.00)	EDS
R_{merge}	0.14	Depositor
R_{sym}	(Not available)	Depositor
$< I/\sigma(I) > 1$	$2.67 (at 3.00 \text{\AA})$	Xtriage
Refinement program	REFMAC 5	Depositor
B B.	0.203 , 0.268	Depositor
n, n_{free}	0.206 , 0.270	DCC
R_{free} test set	1447 reflections (5.10%)	wwPDB-VP
Wilson B-factor $(Å^2)$	62.1	Xtriage
Anisotropy	0.607	Xtriage
Bulk solvent $k_{sol}(e/Å^3), B_{sol}(Å^2)$	0.27, 13.4	EDS
L-test for $twinning^2$	$ < L >=0.47, < L^2>=0.30$	Xtriage
Estimated twinning fraction	No twinning to report.	Xtriage
F_o, F_c correlation	0.93	EDS
Total number of atoms	8036	wwPDB-VP
Average B, all atoms $(Å^2)$	71.0	wwPDB-VP

Xtriage's analysis on translational NCS is as follows: The largest off-origin peak in the Patterson function is 3.45% 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)

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).

Mal	Mol Chain		lengths	Bond angles		
	Ullaili	RMSZ	# Z > 5	RMSZ	# Z > 5	
1	L	0.51	0/1665	0.73	0/2263	
1	1	0.47	0/1616	0.68	0/2200	
2	Н	0.54	0/1661	0.75	0/2268	
2	h	0.53	0/1631	0.70	0/2229	
3	С	0.49	0/819	0.67	0/1118	
3	с	0.47	0/828	0.65	0/1129	
All	All	0.51	0/8220	0.71	0/11207	

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)

Due to software issues we are unable to calculate clashes - this section is therefore empty.

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	Percentiles
1	L	211/215~(98%)	199 (94%)	11 (5%)	1 (0%)	29 68

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Mol	Chain	Analysed	Favoured	Allowed	Outliers	Percer	ntiles
1	1	210/215~(98%)	201~(96%)	7 (3%)	2(1%)	15	53
2	Н	211/225~(94%)	196~(93%)	14 (7%)	1 (0%)	29	68
2	h	208/225~(92%)	196~(94%)	9~(4%)	3~(1%)	11	43
3	\mathbf{C}	110/118~(93%)	104 (94%)	6~(6%)	0	100	100
3	с	111/118~(94%)	100 (90%)	11 (10%)	0	100	100
All	All	1061/1116~(95%)	996 (94%)	58 (6%)	7 (1%)	22	60

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All (7) Ramachandran outliers are listed below:

Mol	Chain	Res	Type
2	Н	101	TRP
2	h	101	TRP
1	1	139	ASN
2	h	41	PRO
1	L	139	ASN
1	1	69	GLY
2	h	52	SER

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	180/185~(97%)	164 (91%)	16 (9%)	9 35	
1	1	166/185~(90%)	159 (96%)	7 (4%)	30 66	
2	Н	174/191~(91%)	163~(94%)	11 (6%)	18 51	
2	h	173/191~(91%)	152 (88%)	21 (12%)	5 21	
3	С	78/97~(80%)	70~(90%)	8 (10%)	7 28	
3	с	82/97~(84%)	78~(95%)	4(5%)	25 61	
All	All	853/946~(90%)	786~(92%)	67~(8%)	12 41	

All (67) residues with a non-rotameric sidechain are listed below:



Mol	Chain	Res	Type
1	L	6	GLN
1	L	34	LEU
1	L	57	THR
1	L	61	ASP
1	L	66	SER
1	L	70	THR
1	L	109	ARG
1	L	115	SER
1	L	118	ILE
1	L	151	VAL
1	L	153	ASN
1	L	155	LEU
1	L	157	SER
1	L	188	GLU
1	L	196	GLU
1	L	204	SER
2	Н	31	SER
2	Н	62	ASP
2	Н	96	CYS
2	Н	101	TRP
2	Н	155	VAL
2	Н	156	THR
2	Н	174	VAL
2	Н	184	SER
2	Н	186	VAL
2	Н	188	THR
2	Н	202	ASN
1	1	7	SER
1	1	70	THR
1	1	90	GLN
1	1	101	GLN
1	1	110	THR
1	1	146	LYS
1	1	198	THR
2	h	1	GLN
2	h	5	VAL
2	h	7	SER
2	h	17	SER
2	h	50	PHE
2	h	51	ILE
2	h	69	THR
2	h	85	SER
2	h	87	ARG

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	<u> </u>	1	
Mol	Chain	Res	Type
2	h	89	GLU
2	h	101	TRP
2	h	156	THR
2	h	165	THR
2	h	166	SER
2	h	168	VAL
2	h	174	VAL
2	h	177	SER
2	h	182	SER
2	h	188	THR
2	h	190	PRO
2	h	214	LYS
3	с	34	VAL
3	с	37	THR
3	с	68	CYS
3	с	76	GLN
3	С	4	HIS
3	С	34	VAL
3	С	41	GLN
3	С	45	GLN
3	С	56	MET
3	С	67	ILE
3	С	87	MET
3	С	94	CYS

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Sometimes side chains can be flipped to improve hydrogen bonding and reduce clashes. All (9) such side chains are listed below:

Mol	Chain	Res	Type
1	L	139	ASN
2	Н	13	GLN
2	Н	82	GLN
2	Н	204	ASN
1	l	90	GLN
1	1	138	ASN
2	h	169	HIS
3	с	4	HIS
3	с	75	ASN

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^{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	$\mathbf{OWAB}(\mathrm{\AA}^2)$	Q<0.9
1	L	213/215~(99%)	-0.55	0 100 100	45, 59, 73, 81	0
1	1	212/215~(98%)	-0.28	0 100 100	45, 68, 132, 149	0
2	Н	215/225~(95%)	-0.43	0 100 100	48, 63, 91, 109	0
2	h	212/225~(94%)	-0.32	1 (0%) 91 75	46, 65, 108, 125	0
3	С	114/118~(96%)	-0.28	1 (0%) 84 63	60, 84, 108, 126	0
3	с	115/118~(97%)	-0.17	0 100 100	57, 84, 112, 134	0
All	All	1081/1116~(96%)	-0.36	2 (0%) 95 87	45, 66, 115, 149	0

All (2) RSRZ outliers are listed below:

Mol	Chain	Res	Type	RSRZ
2	h	133	SER	2.9
3	С	23	CYS	2.2

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.

