



wwPDB NMR Structure Validation Summary Report ⓘ

Apr 26, 2016 – 02:10 PM BST

PDB ID : 1BUQ
Title : SOLUTION STRUCTURE OF DELTA-5-3-KETOSTEROID ISOMERASE
COMPLEXED WITH THE STEROID 19-NORTESTOSTERONE-HEMISU
CCINATE
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Deposited on : 1998-09-04

This is a wwPDB NMR Structure Validation Summary Report for a publicly released PDB entry.
We welcome your comments at validation@mail.wwpdb.org
A user guide is available at
<http://wwpdb.org/validation/2016/NMRValidationReportHelp>
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:

Cyrange : Kirchner and Güntert (2011)
NmrClust : Kelley et al. (1996)
MolProbity : 4.02b-467
Mogul : 1.7.1 (RC1), CSD as537be (2016)
Percentile statistics : 20151230.v01 (using entries in the PDB archive December 30th 2015)
RCI : v_1n_11_5_13_A (Berjanski et al., 2005)
PANAV : Wang et al. (2010)
ShiftChecker : rb-20027457
Ideal geometry (proteins) : Engh & Huber (2001)
Ideal geometry (DNA, RNA) : Parkinson et al. (1996)
Validation Pipeline (wwPDB-VP) : rb-20027457

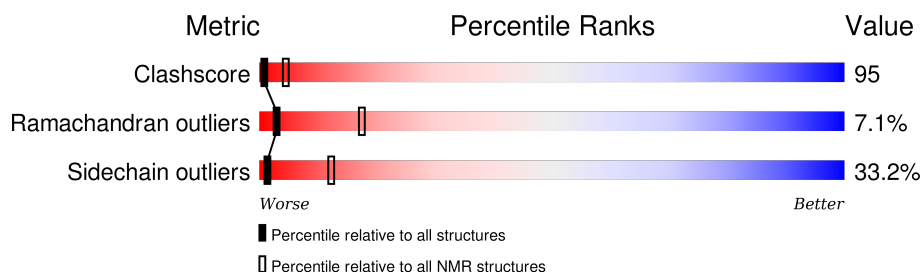
1 Overall quality at a glance

The following experimental techniques were used to determine the structure:

SOLUTION NMR

The overall completeness of chemical shifts assignment is 81%.

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)	NMR archive (#Entries)
Clashscore	114402	11133
Ramachandran outliers	111179	9975
Sidechain outliers	111093	9958

The table below summarises the geometric issues observed across the polymeric chains and their fit to the experimental data. The red, orange, yellow and green segments indicate the fraction of residues that contain outliers for ≥ 3 , 2, 1 and 0 types of geometric quality criteria. A cyan segment indicates the fraction of residues that are not part of the well-defined cores, and 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 $\leq 5\%$.

Mol	Chain	Length	Quality of chain
1	A	125	 9% 64% 24% • •
1	B	125	 11% 62% 23% • •

2 Ensemble composition and analysis ⓘ

This entry contains 15 models. Model 5 is the overall representative, medoid model (most similar to other models). The authors have identified model 1 as representative.

The following residues are included in the computation of the global validation metrics.

Well-defined (core) protein residues			
Well-defined core	Residue range (total)	Backbone RMSD (Å)	Medoid model
1	A:3-A:125, B:201-B:323 (246)	0.83	5

Ill-defined regions of proteins are excluded from the global statistics.

Ligands and non-protein polymers are included in the analysis.

The models can be grouped into 2 clusters and 1 single-model cluster was found.

Cluster number	Models
1	1, 2, 4, 5, 6, 7, 9, 10, 11, 12, 14
2	3, 8, 13
Single-model clusters	15

3 Entry composition [i](#)

There are 2 unique types of molecules in this entry. The entry contains 3858 atoms, of which 1918 are hydrogens and 0 are deuteriums.

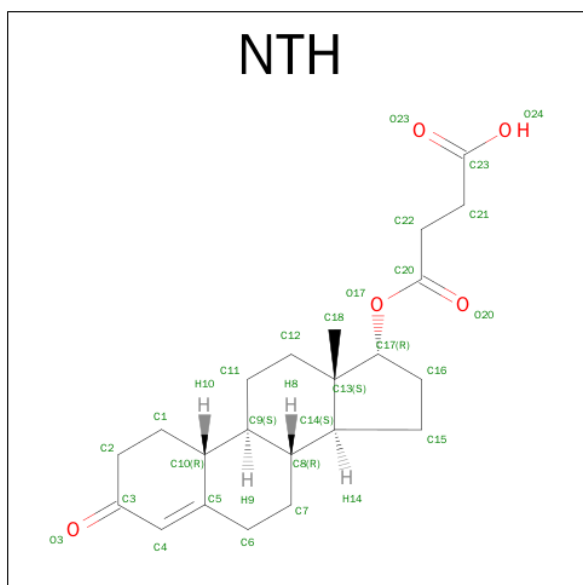
- Molecule 1 is a protein called PROTEIN (3-KETOSTEROID ISOMERASE-19-NORTESTOSTERONE-HEMISUCCINATE).

Mol	Chain	Residues	Atoms						Trace
1	A	125	Total	C	H	N	O	S	0
			1873	597	930	165	178	3	
1	B	125	Total	C	H	N	O	S	0
			1873	597	930	165	178	3	

There are 4 discrepancies between the modelled and reference sequences:

Chain	Residue	Modelled	Actual	Comment	Reference
A	55	PHE	TYR	ENGINEERED	UNP P00947
A	88	PHE	TYR	ENGINEERED	UNP P00947
B	255	PHE	TYR	ENGINEERED	UNP P00947
B	288	PHE	TYR	ENGINEERED	UNP P00947

- Molecule 2 is SUCCINIC ACID MONO-(13-METHYL-3-OXO-2,3,6,7,8,9,10,11,12,13,14,15,16,17-TETRADECAHYDRO-1H-CYCLOPENTA[A]PHENANTHREN-17-YL) ESTER (three-letter code: NTH) (formula: $C_{22}H_{30}O_5$).



Mol	Chain	Residues	Atoms			
2	A	1	Total	C	H	O
			56	22	29	5

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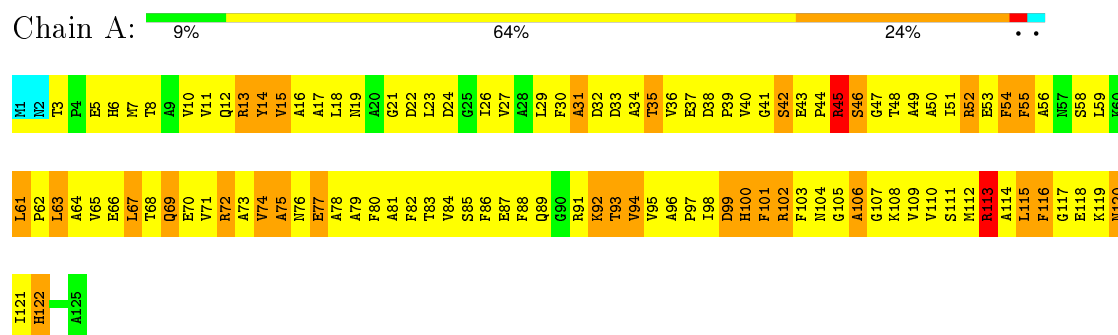
Mol	Chain	Residues	Atoms			
			Total	C	H	O
2	B	1	56	22	29	5

4 Residue-property plots

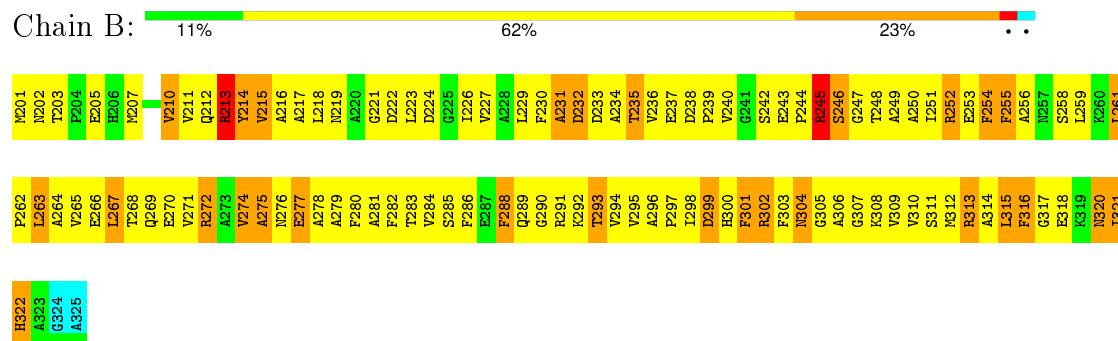
4.1 Average score per residue in the NMR ensemble

These plots are provided for all protein, RNA and DNA chains in the entry. The first graphic is the same as shown in the summary in section 1 of this report. The second graphic shows the sequence where residues are colour-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. Stretches of 2 or more consecutive residues without any outliers are shown as green connectors. Residues which are classified as ill-defined in the NMR ensemble, are shown in cyan with an underline colour-coded according to the previous scheme. Residues which were present in the experimental sample, but not modelled in the final structure are shown in grey.

- Molecule 1: PROTEIN (3-KETOSTEROID ISOMERASE-19-NORTESTOSTERONE-HEMIS UCCINATE)



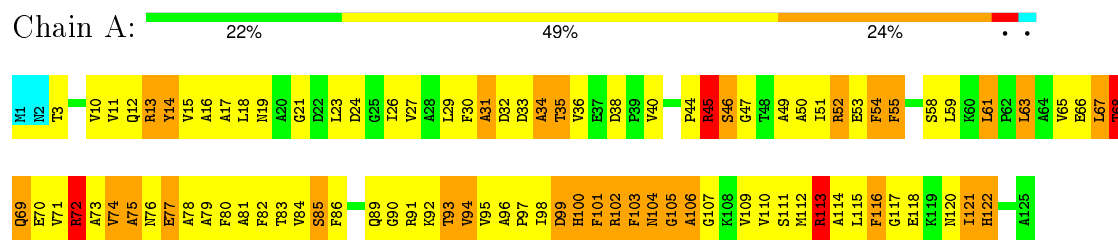
- Molecule 1: PROTEIN (3-KETOSTEROID ISOMERASE-19-NORTESTOSTERONE-HEMIS UCCINATE)



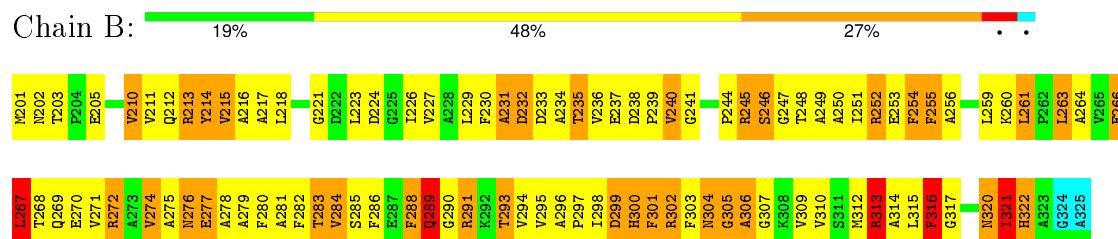
4.2 Residue scores for the representative (medoid) model from the NMR ensemble

The representative model is number 5. Colouring as in section 4.1 above.

- Molecule 1: PROTEIN (3-KETOSTEROID ISOMERASE-19-NORTESTOSTERONE-HEMIS UCCINATE)



• Molecule 1: PROTEIN (3-KETOSTEROID ISOMERASE-19-NORTESTOSTERONE-HEMIS UCCINATE)



5 Refinement protocol and experimental data overview

The models were refined using the following method: *DISTANCE GEOMETRY, SIMULATED ANNEALING AND REFINEMENT CALCULATIONS*.

Of the 120 calculated structures, 15 were deposited, based on the following criterion: *NOE VIOLATION = < 0.35Å AND DIHEDRAL VIOLATION < 5 DEG..*

The following table shows the software used for structure solution, optimisation and refinement.

Software name	Classification	Version
X-PLOR	refinement	3.8
X-PLOR	structure solution	3.8

The following table shows chemical shift validation statistics as aggregates over all chemical shift files. Detailed validation can be found in section 7 of this report.

Chemical shift file(s)	BMRB entry 4230
Number of chemical shift lists	1
Total number of shifts	2746
Number of shifts mapped to atoms	2730
Number of unparsed shifts	0
Number of shifts with mapping errors	16
Number of shifts with mapping warnings	0
Assignment completeness (well-defined parts)	81%

No validations of the models with respect to experimental NMR restraints is performed at this time.

6 Model quality i

6.1 Standard geometry i

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

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 (average) 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	1.06±0.00	1±0/945 (0.1±0.0%)	0.86±0.00	0±0/1281 (0.0±0.0%)
1	B	1.06±0.00	1±0/952 (0.1±0.0%)	0.86±0.00	0±0/1293 (0.0±0.0%)
All	All	1.06	30/28455 (0.1%)	0.86	0/38610 (0.0%)

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	Planarity
1	A	0.0±0.0	6.8±0.5
1	B	0.0±0.0	6.7±0.4
All	All	0	203

All unique bond outliers are listed below. They are sorted according to the Z-score of the worst occurrence in the ensemble.

Mol	Chain	Res	Type	Atoms	Z	Observed(Å)	Ideal(Å)	Models	
								Worst	Total
1	A	99	ASP	CG-OD2	5.81	1.38	1.25	7	15
1	B	299	ASP	CG-OD2	5.79	1.38	1.25	10	15

There are no bond-angle outliers.

There are no chirality outliers.

5 of 14 unique planar outliers are listed below. They are sorted by the frequency of occurrence in the ensemble.

Mol	Chain	Res	Type	Group	Models (Total)
1	B	291	ARG	Sidechain	15
1	A	113	ARG	Sidechain	15

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Mol	Chain	Res	Type	Group	Models (Total)
1	A	52	ARG	Sidechain	15
1	A	91	ARG	Sidechain	15
1	B	213	ARG	Sidechain	15

6.2 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 each 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 averaged over the ensemble.

Mol	Chain	Non-H	H(model)	H(added)	Clashes
1	A	927	913	912	190±13
1	B	933	922	918	187±12
2	A	27	29	28	6±3
2	B	27	29	28	6±3
All	All	28710	28395	28290	5388

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 95.

5 of 1798 unique clashes are listed below, sorted by their clash magnitude.

Atom-1	Atom-2	Clash(Å)	Distance(Å)	Models	
				Worst	Total
1:B:261:LEU:HD23	1:B:263:LEU:HD21	1.12	1.15	13	1
1:B:295:VAL:HG22	1:B:321:ILE:HG23	1.11	1.18	3	10
1:B:261:LEU:HD21	1:B:263:LEU:HD21	1.03	1.21	8	1
1:A:63:LEU:HD13	1:A:84:VAL:HG13	1.03	1.23	14	1
1:A:74:VAL:HG11	1:B:240:VAL:HG21	1.03	1.28	5	3

6.3 Torsion angles [i](#)

6.3.1 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 PDB entries followed by that with respect to all NMR entries. 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	122/125 (98%)	92±3 (75±2%)	20±3 (17±3%)	10±2 (8±2%)	2	15
1	B	122/125 (98%)	92±3 (75±2%)	22±3 (18±2%)	8±2 (6±2%)	4	21
All	All	3660/3750 (98%)	2757 (75%)	644 (18%)	259 (7%)	3	17

5 of 47 unique Ramachandran outliers are listed below. They are sorted by the frequency of occurrence in the ensemble.

Mol	Chain	Res	Type	Models (Total)
1	B	316	PHE	15
1	A	116	PHE	15
1	B	267	LEU	15
1	A	31	ALA	15
1	A	67	LEU	14

6.3.2 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 PDB entries followed by that with respect to all NMR entries. 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	93/95 (98%)	61±4 (65±4%)	32±4 (35±4%)	1	10
1	B	95/95 (100%)	65±3 (68±3%)	30±3 (32±3%)	1	14
All	All	2820/2850 (99%)	1884 (67%)	936 (33%)	1	12

5 of 126 unique residues with a non-rotameric sidechain are listed below. They are sorted by the frequency of occurrence in the ensemble.

Mol	Chain	Res	Type	Models (Total)
1	A	35	THR	15
1	B	263	LEU	15
1	B	274	VAL	15
1	A	74	VAL	15
1	A	101	PHE	15

6.3.3 RNA ⓘ

There are no RNA molecules in this entry.

6.4 Non-standard residues in protein, DNA, RNA chains [i](#)

There are no non-standard protein/DNA/RNA residues in this entry.

6.5 Carbohydrates [i](#)

There are no carbohydrates in this entry.

6.6 Ligand geometry [i](#)

2 ligands are modelled in this entry.

In the following table, the Counts columns list the number of bonds for which Mogul statistics could be retrieved, the number of bonds that are observed in the model and the number of bonds 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 is the number of standard deviations the observed value is removed from the expected value. A bond length with $|Z| > 2$ is considered an outlier worth inspection. RMSZ is the average root-mean-square of all Z scores of the bond lengths.

Mol	Type	Chain	Res	Link	Bond lengths		
					Counts	RMSZ	#Z>2
2	NTH	A	126	-	27,30,30	2.97±0.02	1±0 (3±0%)
2	NTH	B	326	-	27,30,30	2.97±0.02	1±0 (3±0%)

In the following table, the Counts columns list the number of angles for which Mogul statistics could be retrieved, the number of angles that are observed in the model and the number of 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 angle is the number of standard deviations the observed value is removed from the expected value. A bond angle with $|Z| > 2$ is considered an outlier worth inspection. RMSZ is the average root-mean-square of all Z scores of the bond angles.

Mol	Type	Chain	Res	Link	Bond angles		
					Counts	RMSZ	#Z>2
2	NTH	A	126	-	42,45,45	2.39±0.01	3±0 (7±0%)
2	NTH	B	326	-	42,45,45	2.39±0.01	3±0 (7±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
2	NTH	A	126	-	-	0±0,7,62,62	0±0,4,4,4
2	NTH	B	326	-	-	0±0,7,62,62	0±0,4,4,4

All unique bond outliers are listed below. They are sorted according to the Z-score of the worst occurrence in the ensemble.

Mol	Chain	Res	Type	Atoms	Z	Observed(Å)	Ideal(Å)	Models	
								Worst	Total
2	A	126	NTH	O17-C17	11.91	1.25	1.46	2	15
2	B	326	NTH	O17-C17	11.89	1.25	1.46	1	15

5 of 6 unique angle outliers are listed below. They are sorted according to the Z-score of the worst occurrence in the ensemble.

Mol	Chain	Res	Type	Atoms	Z	Observed(°)	Ideal(°)	Models	
								Worst	Total
2	A	126	NTH	C18-C13-C14	8.35	127.61	111.76	7	15
2	B	326	NTH	C18-C13-C14	8.33	127.56	111.76	7	15
2	A	126	NTH	C16-C17-C13	6.56	112.39	105.15	8	15
2	B	326	NTH	C16-C17-C13	6.53	112.36	105.15	10	15
2	B	326	NTH	O17-C17-C16	5.31	124.47	111.00	8	15

There are no chirality outliers.

There are no torsion outliers.

There are no ring outliers.

6.7 Other polymers [i](#)

There are no such molecules in this entry.

6.8 Polymer linkage issues [i](#)

There are no chain breaks in this entry.

7 Chemical shift validation

The completeness of assignment taking into account all chemical shift lists is 81% for the well-defined parts and 81% for the entire structure.

7.1 Chemical shift list 1

File name: BMRB entry 4230

Chemical shift list name: *assigned_chem_shift_list_1*

7.1.1 Bookkeeping

The following table shows the results of parsing the chemical shift list and reports the number of nuclei with statistically unusual chemical shifts.

Total number of shifts	2746
Number of shifts mapped to atoms	2730
Number of unparsed shifts	0
Number of shifts with mapping errors	16
Number of shifts with mapping warnings	0
Number of shift outliers (ShiftChecker)	8

The following assigned chemical shifts were not mapped to the molecules present in the coordinate file.

- Residue not found in structure. First 5 (of 16) occurrences are reported below.

Chain	Res	Type	Atom	Shift Data		
				Value	Uncertainty	Ambiguity
A	99	ASH	HA	5.79	-1.0	1
A	99	ASH	HB3	1.32	-1.0	1
A	99	ASH	H	8.26	-1.0	1
B	99	ASH	HB3	1.32	-1.0	1
A	99	ASH	CA	50.4	-1.0	1

7.1.2 Chemical shift referencing

The following table shows the suggested chemical shift referencing corrections.

Nucleus	# values	Correction \pm precision, ppm	Suggested action
$^{13}\text{C}_\alpha$	250	-0.51 ± 0.15	Should be applied
$^{13}\text{C}_\beta$	226	-0.40 ± 0.07	None needed (< 0.5 ppm)
$^{13}\text{C}'$	232	-0.26 ± 0.13	None needed (< 0.5 ppm)

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Nucleus	# values	Correction \pm precision, ppm	Suggested action
^{15}N	232	-1.81 \pm 0.23	Should be applied

7.1.3 Completeness of resonance assignments [i](#)

The following table shows the completeness of the chemical shift assignments for the well-defined regions of the structure. The overall completeness is 81%, i.e. 2333 atoms were assigned a chemical shift out of a possible 2881. 44 out of 44 assigned methyl groups (LEU and VAL) were assigned stereospecifically.

	Total	^1H	^{13}C	^{15}N
Backbone	1173/1210 (97%)	474/482 (98%)	471/492 (96%)	228/236 (97%)
Sidechain	1098/1433 (77%)	646/828 (78%)	433/538 (80%)	19/67 (28%)
Aromatic	62/238 (26%)	62/132 (47%)	0/100 (0%)	0/6 (0%)
Overall	2333/2881 (81%)	1182/1442 (82%)	904/1130 (80%)	247/309 (80%)

7.1.4 Statistically unusual chemical shifts [i](#)

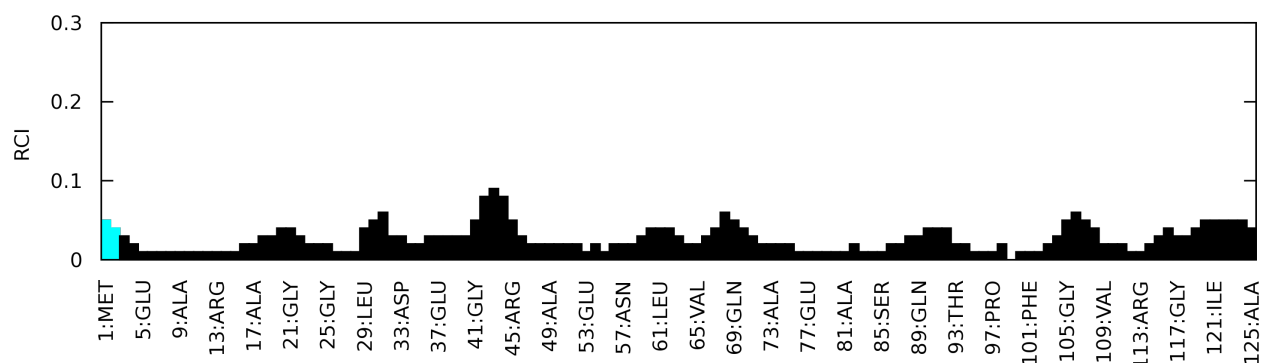
The following table lists the statistically unusual chemical shifts. These are statistical measures, and large deviations from the mean do not necessarily imply incorrect assignments. Molecules containing paramagnetic centres or hemes are expected to give rise to anomalous chemical shifts.

Mol	Chain	Res	Type	Atom	Shift, ppm	Expected range, ppm	Z-score
1	B	252	ARG	NH2	123.30	91.01 – 55.81	14.2
1	A	52	ARG	NH2	123.30	91.01 – 55.81	14.2
1	A	86	PHE	HZ	1.09	9.11 – 4.91	-14.1
1	B	286	PHE	HZ	1.09	9.11 – 4.91	-14.1
1	B	228	ALA	CB	32.00	28.03 – 9.93	7.2
1	A	28	ALA	CB	32.00	28.03 – 9.93	7.2
1	B	228	ALA	CA	67.20	63.07 – 43.27	7.1
1	A	28	ALA	CA	67.20	63.07 – 43.27	7.1

7.1.5 Random Coil Index (RCI) plots [i](#)

The images below report *random coil index* values for the protein chains in the structure. The height of each bar gives a probability of a given residue to be disordered, as predicted from the available chemical shifts and the amino acid sequence. A value above 0.2 is an indication of significant predicted disorder. The colour of the bar shows whether the residue is in the well-defined core (black) or in the ill-defined residue ranges (cyan), as described in section 2 on ensemble composition.

Random coil index (RCI) for chain A:



Random coil index (RCI) for chain B:

