wwPDB NMR Structure Validation Summary Report (i)

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PDB ID	:	2KMX
BMRB ID	:	16441
Title	:	Solution structure of the nucleotide binding domain of the human Menkes protein in the ATP-bound form
Authors	:	Banci, L.; Bertini, I.; Cantini, F.; Inagaki, S.; Migliardi, M.; Rosato, A.
Deposited on	:	2009-08-05

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
https://www.wwpdb.org/validation/2017/NMRValidationReportHelp
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 (i)) were used in the production of this report:

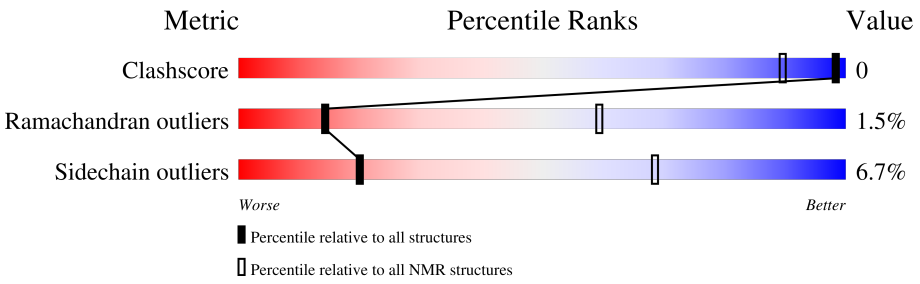
MolProbity	:	4.02b-467
Mogul	:	2022.3.0, CSD as543be (2022)
buster-report	:	1.1.7 (2018)
Percentile statistics	:	20191225.v01 (using entries in the PDB archive December 25th 2019)
wwPDB-RCI	:	v_1n_11_5_13_A (Berjanski et al., 2005)
PANAV	:	Wang et al. (2010)
wwPDB-ShiftChecker	:	v1.2
Ideal geometry (proteins)	:	Engh & Huber (2001)
Ideal geometry (DNA, RNA)	:	Parkinson et al. (1996)
Validation Pipeline (wwPDB-VP)	:	2.36.2

1 Overall quality at a glance (i)

The following experimental techniques were used to determine the structure:
SOLUTION NMR

The overall completeness of chemical shifts assignment is 86%.

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	158937	12864
Ramachandran outliers	154571	11451
Sidechain outliers	154315	11428

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 <=5%

Mol	Chain	Length	Quality of chain
1	A	185	64% • 32%

2 Ensemble composition and analysis (i)

This entry contains 20 models. Model 15 is the overall representative, medoid model (most similar to other models). The authors have identified model 1 as representative, based on the following criterion: *fewest violations*.

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:1053-A:1125, A:1177-A:1229 (126)	0.84	15

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 3 clusters and 3 single-model clusters were found.

Cluster number	Models
1	1, 2, 3, 4, 7, 9, 10, 13, 15, 16, 18, 19, 20
2	6, 17
3	5, 8
Single-model clusters	11; 12; 14

3 Entry composition (i)

There are 2 unique types of molecules in this entry. The entry contains 2863 atoms, of which 1417 are hydrogens and 0 are deuteriums.

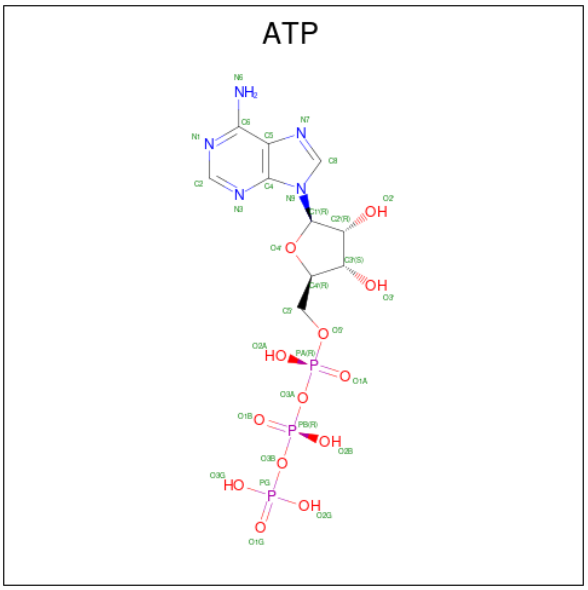
- Molecule 1 is a protein called Copper-transporting ATPase 1.

Mol	Chain	Residues	Atoms						Trace
			Total	C	H	N	O	S	
1	A	185	2820	872	1405	249	285	9	0

There are 4 discrepancies between the modelled and reference sequences:

Chain	Residue	Modelled	Actual	Comment	Reference
A	1047	SER	-	expression tag	UNP Q04656
A	1048	PHE	-	expression tag	UNP Q04656
A	1049	THR	-	expression tag	UNP Q04656
A	1050	MET	-	expression tag	UNP Q04656

- Molecule 2 is ADENOSINE-5'-TRIPHOSPHATE (three-letter code: ATP) (formula: $C_{10}H_{16}N_5O_{13}P_3$).



Mol	Chain	Residues	Atoms					
			Total	C	H	N	O	P
2	A	1	43	10	12	5	13	3

4 Residue-property plots (i)

4.1 Average score per residue in the NMR ensemble

These plots are provided for all protein, RNA, DNA and oligosaccharide 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: Copper-transporting ATPase 1

Chain A: 64% • 32%

S1047 F1048 T1049 M1050 H1051 G1052 R1067 F1111 M1125 I1126 E1127 G1128 L1129 L1130 H1131 K1132 N1133 N1134 W1135 N1136 I1137 E1138 D1139 N1140 N1141 I1142 K1143 N1144 A1145 S1146 L1147 V1148 Q1149 I1150 D1151 A1152 S1153 N1154 E1155 Q1156 S1157 S1158 T1159 S1160 S1161 S1162 M1163 I1164 I1165 D1166 A1167 Q1168 I1169 S1170 N1171 A1172 L1173

N1174 A1175 Q1176 R1185 R1190 R1208 R1211 L1222 D1230 T1231

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

The representative model is number 15. Colouring as in section 4.1 above.

• Molecule 1: Copper-transporting ATPase 1

Chain A: 64% • 32%

S1047 F1048 T1049 M1050 H1051 G1052 R1067 I1119 M1125 I1126 E1127 G1128 L1129 L1130 H1131 K1132 N1133 N1134 W1135 N1136 I1137 E1138 D1139 N1140 N1141 I1142 K1143 N1144 A1145 S1146 L1147 V1148 Q1149 I1150 D1151 A1152 S1153 N1154 E1155 Q1156 S1157 S1158 T1159 S1160 S1161 S1162 M1163 I1164 I1165 D1166 A1167 Q1168 I1169 S1170 N1171 A1172 L1173

N1174 A1175 Q1176 R1185 M1188 I1189 R1190 R1208 L1222 D1230 T1231

5 Refinement protocol and experimental data overview (i)

The models were refined using the following method: *simulated annealing*.

Of the 500 calculated structures, 20 were deposited, based on the following criterion: *target function*.

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

Software name	Classification	Version
CYANA	structure solution	
Amber	refinement	10

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)	working_cs.cif
Number of chemical shift lists	1
Total number of shifts	2054
Number of shifts mapped to atoms	2046
Number of unparsed shifts	0
Number of shifts with mapping errors	8
Number of shifts with mapping warnings	0
Assignment completeness (well-defined parts)	86%

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: ATP

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	0.65±0.01	0±0/979 (0.0± 0.0%)	1.13±0.03	4±1/1330 (0.3± 0.1%)
All	All	0.65	0/19580 (0.0%)	1.13	86/26600 (0.3%)

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	0.1±0.3
All	All	0	2

There are no bond-length outliers.

5 of 9 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(^{o})	Ideal(^{o})	Models	
								Worst	Total
1	A	1211	ARG	NE-CZ-NH1	10.14	125.37	120.30	14	15
1	A	1067	ARG	NE-CZ-NH1	9.93	125.26	120.30	2	16
1	A	1185	ARG	NE-CZ-NH1	9.63	125.12	120.30	7	18
1	A	1208	ARG	NE-CZ-NH1	9.54	125.07	120.30	20	13
1	A	1190	ARG	NE-CZ-NH1	9.04	124.82	120.30	10	17

There are no chirality outliers.

All 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	A	1111	PHE	Sidechain	1

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Mol	Chain	Res	Type	Group	Models (Total)
1	A	1185	ARG	Sidechain	1

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	966	979	979	0±1
All	All	19940	19820	19820	6

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 0.

All unique clashes are listed below, sorted by their clash magnitude.

Atom-1	Atom-2	Clash(Å)	Distance(Å)	Models	
				Worst	Total
1:A:1107:THR:HG22	1:A:1108:CYS:H	0.56	1.60	7	2
1:A:1062:LEU:HD23	1:A:1222:LEU:HD22	0.51	1.82	6	3
1:A:1207:GLU:HG2	1:A:1213:ALA:HB3	0.45	1.89	14	1

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	126/185 (68%)	115±2 (91±1%)	9±2 (7±2%)	2±1 (2±1%)	14	59
All	All	2520/3700 (68%)	2294 (91%)	187 (7%)	39 (2%)	14	59

5 of 10 unique Ramachandran outliers are listed below. They are sorted by the frequency of occurrence in the ensemble.

Mol	Chain	Res	Type	Models (Total)
1	A	1125	ASN	15
1	A	1111	PHE	6
1	A	1177	GLN	5
1	A	1190	ARG	3
1	A	1067	ARG	2

6.3.2 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 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	110/162 (68%)	103±3 (93±3%)	7±3 (7±3%)	20 68
All	All	2200/3240 (68%)	2053 (93%)	147 (7%)	20 68

5 of 40 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	1222	LEU	16
1	A	1111	PHE	9
1	A	1112	GLN	8
1	A	1181	LEU	8
1	A	1191	ASN	7

6.3.3 RNA (i)

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 monosaccharides in this entry.

6.6 Ligand geometry (i)

1 ligand is 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	ATP	A	1232	-	28,33,33	2.12±0.19	5±0 (16±1%)

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	ATP	A	1232	-	34,52,52	1.53±0.31	5±1 (14±3%)

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	ATP	A	1232	-	-	0±0,18,38,38	0±0,3,3,3

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	1232	ATP	PB-O3B	7.39	1.51	1.59	19	20
2	A	1232	ATP	PA-O3A	6.80	1.52	1.59	2	20
2	A	1232	ATP	PB-O3A	6.55	1.52	1.59	19	20
2	A	1232	ATP	PG-O3G	2.39	1.45	1.54	9	18

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Mol	Chain	Res	Type	Atoms	Z	Observed(Å)	Ideal(Å)	Models	
								Worst	Total
2	A	1232	ATP	PG-O2G	2.31	1.46	1.54	18	17

5 of 8 unique angle outliers are listed below. They are sorted according to the Z-score of the worst occurrence in the ensemble.

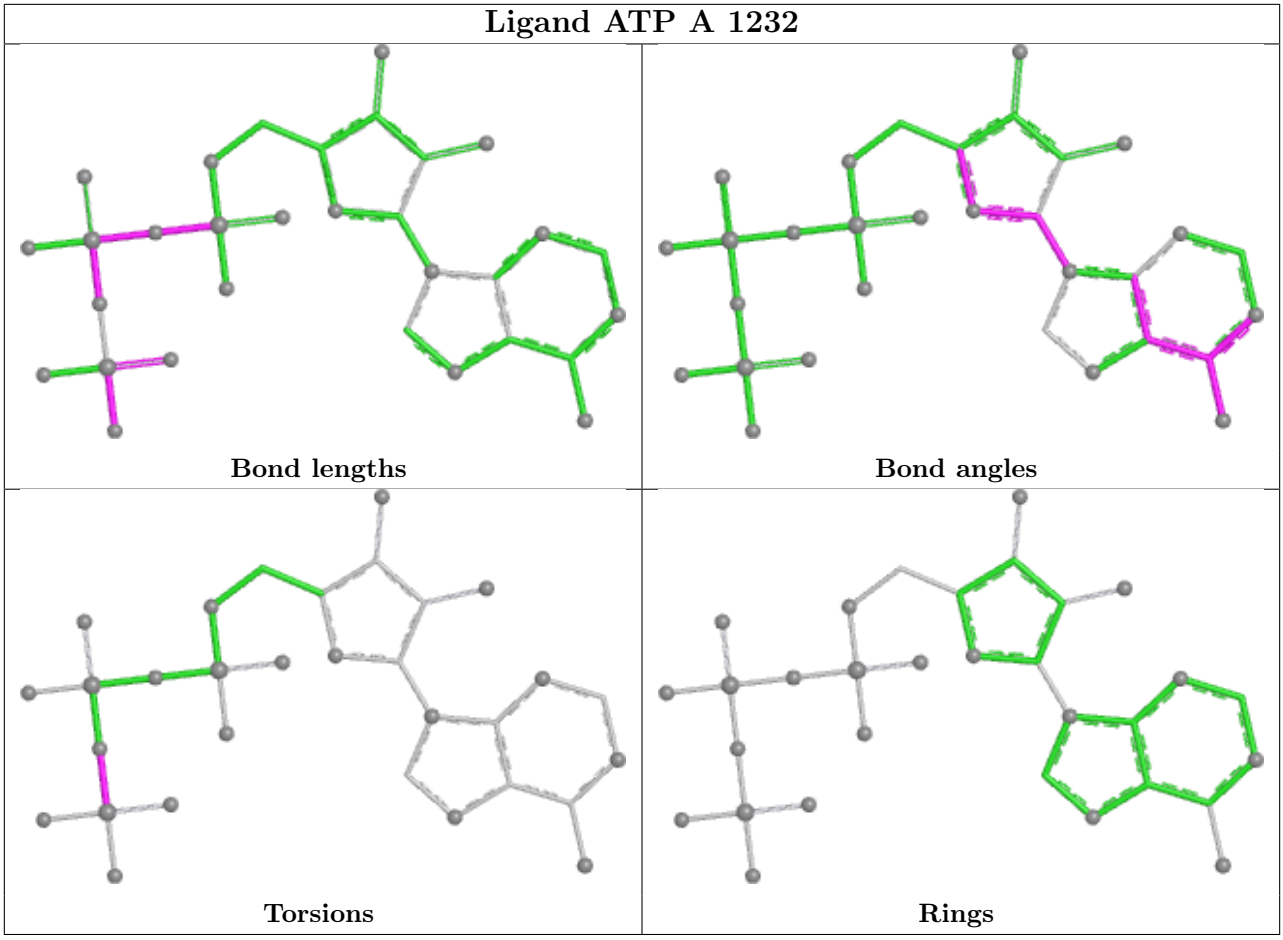
Mol	Chain	Res	Type	Atoms	Z	Observed(^{o})	Ideal(^{o})	Models	
								Worst	Total
2	A	1232	ATP	O4'-C1'-N9	8.49	120.00	108.75	13	17
2	A	1232	ATP	C4'-O4'-C1'	6.37	104.09	109.92	5	14
2	A	1232	ATP	C5-C6-N6	5.61	128.86	120.31	13	20
2	A	1232	ATP	N6-C6-N1	3.89	110.01	118.33	13	19
2	A	1232	ATP	C1'-N9-C4	3.14	121.12	126.64	13	5

There are no chirality outliers.

There are no torsion outliers.

There are no ring outliers.

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.



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 (i)

The completeness of assignment taking into account all chemical shift lists is 86% for the well-defined parts and 84% for the entire structure.

7.1 Chemical shift list 1

File name: working_cs.cif

Chemical shift list name: *assigned_chem_shift_list_1*

7.1.1 Bookkeeping (i)

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	2054
Number of shifts mapped to atoms	2046
Number of unparsed shifts	0
Number of shifts with mapping errors	8
Number of shifts with mapping warnings	0
Number of shift outliers (ShiftChecker)	1

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

- No matching atom found in the structure. First 5 (of 8) occurrences are reported below.

List ID	Chain	Res	Type	Atom	Shift Data		
					Value	Uncertainty	Ambiguity
1	UNMAPPED	1	ATP	H5'1	3.475	0.030	2
1	UNMAPPED	1	ATP	H5'2	3.586	0.030	2
1	UNMAPPED	1	ATP	H4'	4.082	0.030	1
1	UNMAPPED	1	ATP	H3'	3.091	0.030	1
1	UNMAPPED	1	ATP	H2'	3.543	0.030	1
1	UNMAPPED	1	ATP	H1'	5.93	0.030	1
1	UNMAPPED	1	ATP	H2	8.189	0.030	1
1	UNMAPPED	1	ATP	H8	7.175	0.030	1

7.1.2 Chemical shift referencing (i)

The following table shows the suggested chemical shift referencing corrections.

Nucleus	# values	Correction ± precision, *ppm*	Suggested action
$^{13}C_{\alpha}$	175	-0.69 ± 0.10	Should be checked
$^{13}C_{\beta}$	164	-0.16 ± 0.09	None needed (< 0.5 ppm)
$^{13}C'$	157	0.03 ± 0.09	None needed (< 0.5 ppm)
^{15}N	172	-0.27 ± 0.25	None needed (< 0.5 ppm)

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 86%, i.e. 1453 atoms were assigned a chemical shift out of a possible 1693. 0 out of 24 assigned methyl groups (LEU and VAL) were assigned stereospecifically.

	Total	^{1}H	^{13}C	^{15}N
Backbone	606/633 (96%)	254/258 (98%)	231/252 (92%)	121/123 (98%)
Sidechain	827/982 (84%)	567/641 (88%)	250/306 (82%)	10/35 (29%)
Aromatic	20/78 (26%)	20/40 (50%)	0/33 (0%)	0/5 (0%)
Overall	1453/1693 (86%)	841/939 (90%)	481/591 (81%)	131/163 (80%)

7.1.4 Statistically unusual chemical shifts (i)

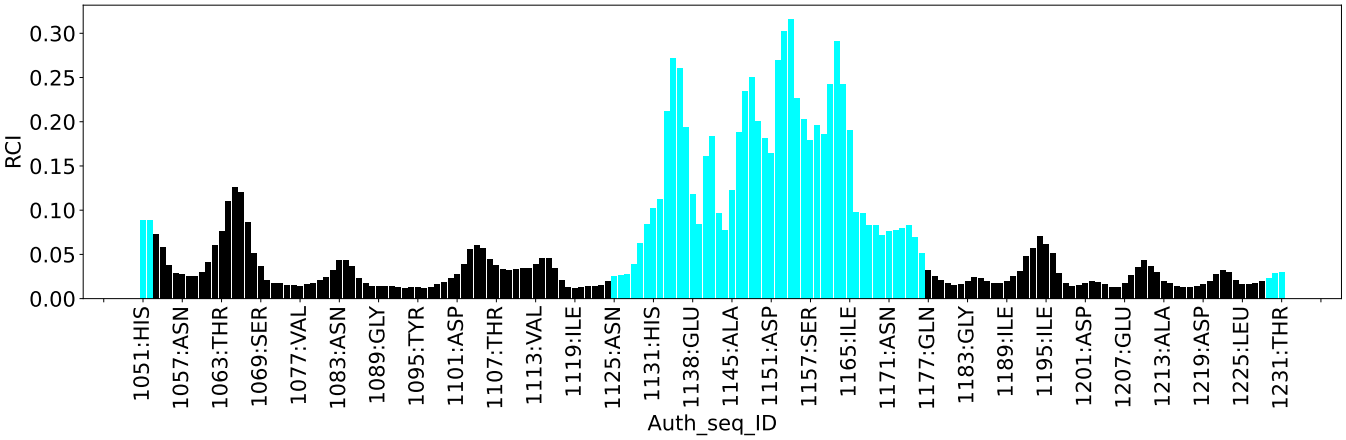
The following table lists the statistically unusual chemical shifts. These are statistical measures, and large deviations from the mean do not necessarily imply incorrect assignments. Molecules containing paramagnetic centres or hemes are expected to give rise to anomalous chemical shifts.

List Id	Chain	Res	Type	Atom	Shift, *ppm*	Expected range, *ppm*	Z-score
1	A	1079	THR	HG1	5.44	0.08 – 2.19	20.4

7.1.5 Random Coil Index (RCI) plots (i)

The image below reports *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.If well-defined core and ill-defined regions are not identified then it is shown as gray bars.

Random coil index (RCI) for chain A:


RCI
0.00
0.05
0.10
0.15
0.20
0.25
0.30
1051:HIS
1057:ASN
1063:THR
1069:SER
1077:VAL
1083:ASN
1089:GLY
1095:TYR
1101:ASP
1107:THR
1113:VAL
1119:ILE
1125:ASN
1131:HIS
1138:GLU
1145:ALA
1151:ASP
1157:SER
1165:ILE
1171:ASN
1177:GLN
1183:GLY
1189:ILE
1195:ILE
1201:ASP
1207:GLU
1213:ALA
1219:ASP
1225:LEU
1231:THR
Auth_seq_ID