Full wwPDB NMR Structure Validation Report (i)

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PDB ID	:	1XYK
Title	:	NMR Structure of the canine prion protein
Authors	:	Lysek, D.A.; Schorn, C.; Esteve-Moya, V.; Herrmann, T.; Wuthrich, K.
Deposited on	:	2004-11-10

The following versions of software and data (see references (i)) were used in the production of this report:

|		
Cyrange	:	Kirchner and Güntert (2011)
NmrClust	:	Kelley et al. (1996)
MolProbity	:	4.02b-467
Mogul	:	unknown
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 80%.

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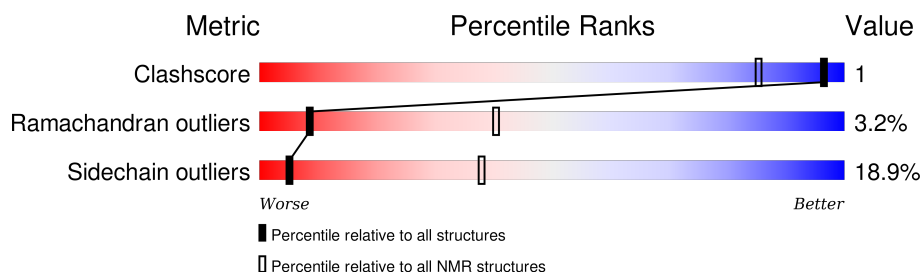
Metric	Whole archive (#Entries)	NMR archive (#Entries)
Clashscore	114402	11133
Ramachandran outliers	111179	9975
Sidechain outliers	111093	9958

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 <=5%

Mol	Chain	Length	Quality of chain
1	A	111	74% 18% 8%

2 Ensemble composition and analysis (i)

This entry contains 20 models. Model 2 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:126-A:227 (102)	0.65	2

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

3 Entry composition (i)

There is only 1 type of molecule in this entry. The entry contains 1773 atoms, of which 858 are hydrogens and 0 are deuteriums.

- Molecule 1 is a protein called prion protein.

Mol	Chain	Residues	Atoms						Trace
			Total	C	H	N	O	S	
1	A	111	1773	569	858	158	180	8	0

4 Residue-property plots (i)

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.
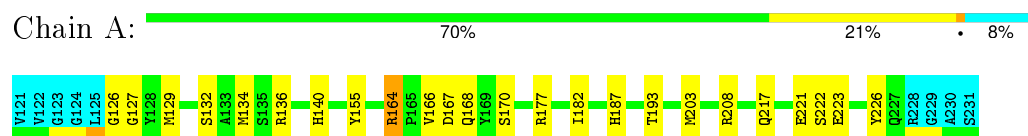
- Molecule 1: prion protein

Chain A:
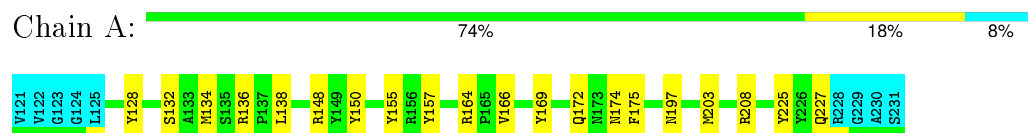
4.2 Scores per residue for each member of the ensemble

Colouring as in section 4.1 above.

4.2.1 Score per residue for model 1

- Molecule 1: prion protein

Chain A:

4.2.2 Score per residue for model 2 (medoid)

- Molecule 1: prion protein

Chain A:

4.2.3 Score per residue for model 3

● Molecule 1: prion protein

Chain A:

4.2.4 Score per residue for model 4

● Molecule 1: prion protein

Chain A:

4.2.5 Score per residue for model 5

● Molecule 1: prion protein

Chain A:
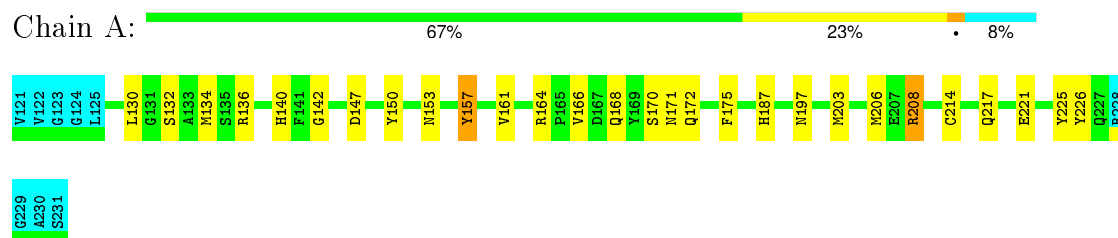
4.2.6 Score per residue for model 6

● Molecule 1: prion protein

Chain A:
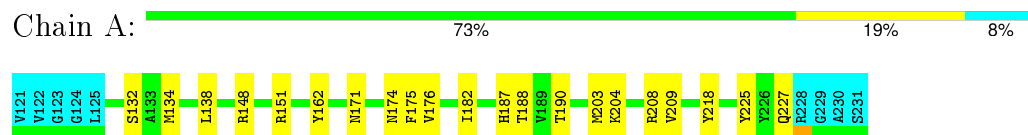
4.2.7 Score per residue for model 7

● Molecule 1: prion protein

Chain A:
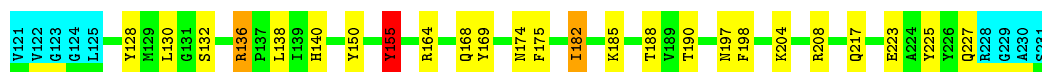
4.2.8 Score per residue for model 8

• Molecule 1: prion protein
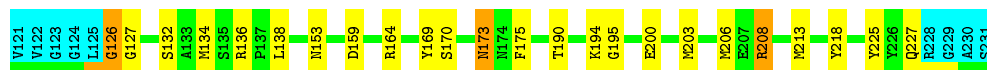
4.2.9 Score per residue for model 9

• Molecule 1: prion protein

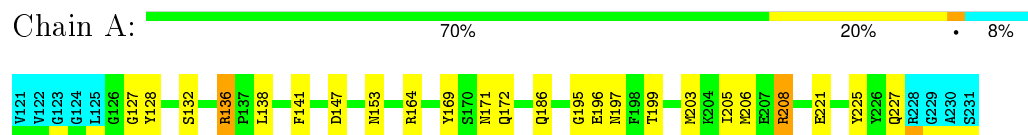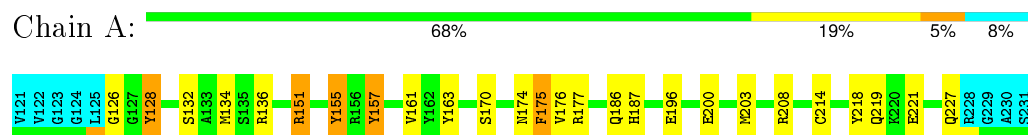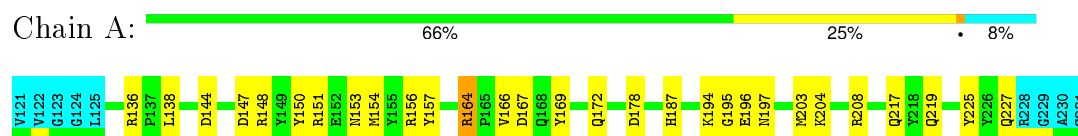
4.2.10 Score per residue for model 10

• Molecule 1: prion protein

4.2.11 Score per residue for model 11

• Molecule 1: prion protein

4.2.12 Score per residue for model 12

• Molecule 1: prion protein

Chain A:

4.2.13 Score per residue for model 13

• Molecule 1: prion protein

Chain A:
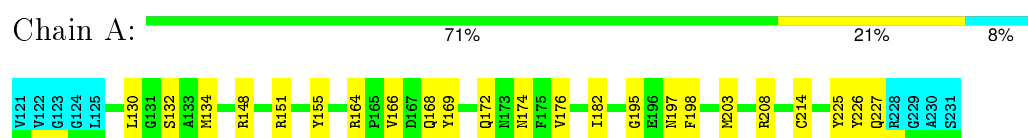
4.2.14 Score per residue for model 14

• Molecule 1: prion protein

Chain A:
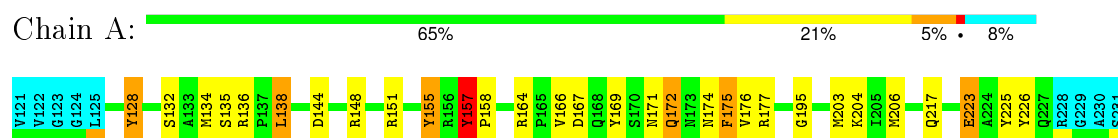
4.2.15 Score per residue for model 15

• Molecule 1: prion protein

Chain A:
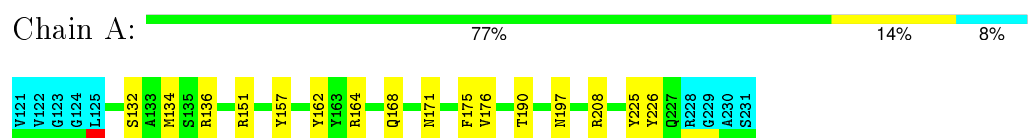
4.2.16 Score per residue for model 16

• Molecule 1: prion protein

Chain A:
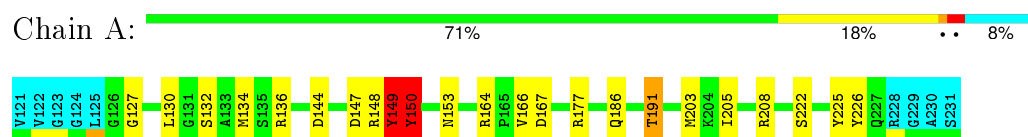
4.2.17 Score per residue for model 17

• Molecule 1: prion protein

Chain A:
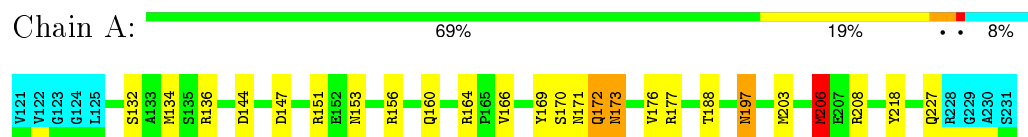
4.2.18 Score per residue for model 18

• Molecule 1: prion protein

Chain A:

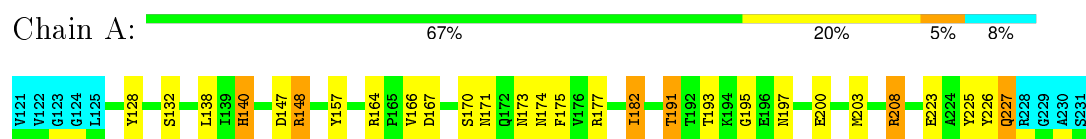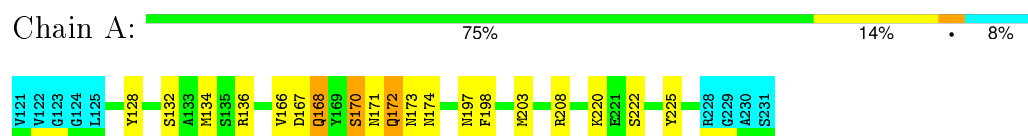#### 4.2.19 Score per residue for model 19

• Molecule 1: prion protein

Chain A:
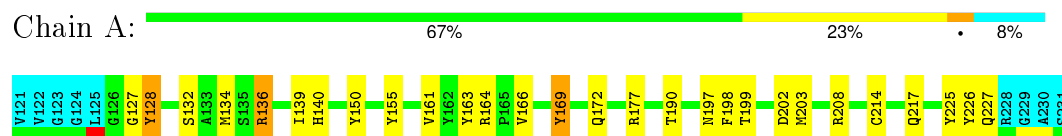
4.2.20 Score per residue for model 20

• Molecule 1: prion protein

Chain A:
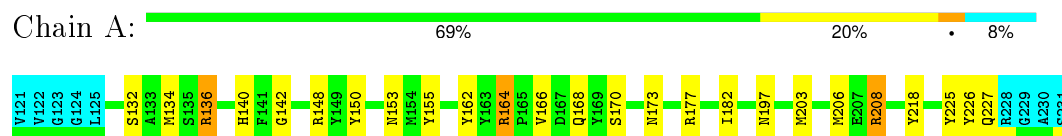
5 Refinement protocol and experimental data overview (i)

The models were refined using the following method: *torsion angle dynamics*.

Of the 100 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
DYANA	structure solution	6.2
CANDID	refinement	1.0
ATNOS	refinement	1.0

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 6378
Number of chemical shift lists	1
Total number of shifts	1259
Number of shifts mapped to atoms	1249
Number of unparsed shifts	0
Number of shifts with mapping errors	10
Number of shifts with mapping warnings	0
Assignment completeness (well-defined parts)	80%

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

6 Model quality ⓘ

6.1 Standard geometry ⓘ

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	0.70±0.01	0±0/879 (0.0±0.0%)	1.10±0.04	2±1/1189 (0.1±0.1%)
All	All	0.70	0/17580 (0.0%)	1.10	34/23780 (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 mainchain group or atoms of a sidechain that are expected to be planar.

Mol	Chain	Chirality	Planarity
1	A	0.1±0.2	2.2±1.4
All	All	1	44

There are no bond-length outliers.

All 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	Models Total
1	A	157	TYR	CB-CA-C	9.95	130.29	110.40	14	4
1	A	157	TYR	CB-CG-CD1	-8.20	116.08	121.00	10	3
1	A	151	ARG	NE-CZ-NH2	-7.74	116.43	120.30	17	3
1	A	155	TYR	CB-CG-CD2	-6.17	117.30	121.00	3	3
1	A	191	THR	CA-CB-CG2	-5.95	104.07	112.40	16	2
1	A	150	TYR	CB-CG-CD2	-5.81	117.51	121.00	16	3
1	A	162	TYR	CB-CG-CD2	-5.63	117.62	121.00	20	2
1	A	136	ARG	NE-CZ-NH2	-5.63	117.48	120.30	2	1
1	A	156	ARG	NE-CZ-NH2	-5.61	117.50	120.30	4	1
1	A	148	ARG	NE-CZ-NH2	-5.51	117.54	120.30	11	1
1	A	136	ARG	NE-CZ-NH1	5.41	123.00	120.30	10	2
1	A	184	VAL	CA-CB-CG1	5.41	119.01	110.90	10	1
1	A	208	ARG	NE-CZ-NH1	5.40	123.00	120.30	2	1
1	A	169	TYR	CB-CG-CD2	-5.40	117.76	121.00	7	1
1	A	149	TYR	CB-CG-CD1	-5.38	117.77	121.00	16	1

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Mol	Chain	Res	Type	Atoms	Z	Observed(^{o})	Ideal(^{o})	Models Worst	Models Total
1	A	206	MET	CB-CG-SD	5.21	128.03	112.40	17	1
1	A	226	TYR	CB-CG-CD2	-5.19	117.88	121.00	1	1
1	A	136	ARG	CD-NE-CZ	5.12	130.77	123.60	10	1
1	A	164	ARG	NE-CZ-NH2	-5.10	117.75	120.30	9	1
1	A	209	VAL	CG1-CB-CG2	-5.09	102.76	110.90	6	1

All unique chiral outliers are listed below.

Mol	Chain	Res	Type	Atoms	Models (Total)
1	A	157	TYR	CA	1

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	208	ARG	Sidechain	7
1	A	148	ARG	Sidechain	5
1	A	164	ARG	Sidechain,Peptide	5
1	A	136	ARG	Sidechain	3
1	A	169	TYR	Peptide,Sidechain	3
1	A	156	ARG	Sidechain	3
1	A	145	TYR	Sidechain	2
1	A	142	GLY	Peptide	2
1	A	178	ASP	Peptide	1
1	A	198	PHE	Sidechain	1
1	A	162	TYR	Sidechain	1
1	A	225	TYR	Sidechain	1
1	A	155	TYR	Sidechain	1
1	A	128	TYR	Sidechain	1
1	A	149	TYR	Sidechain	1
1	A	163	TYR	Sidechain	1
1	A	226	TYR	Sidechain	1
1	A	157	TYR	Sidechain	1
1	A	177	ARG	Sidechain	1
1	A	150	TYR	Sidechain	1
1	A	151	ARG	Sidechain	1
1	A	175	PHE	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	859	797	799	2±2
All	All	17180	15940	15980	37

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

Atom-1	Atom-2	Clash(Å)	Distance(Å)	Models Worst	Models Total
1:A:149:TYR:CE2	1:A:205:ILE:HD13	0.71	2.19	16	1
1:A:175:PHE:CD2	1:A:176:VAL:HG13	0.57	2.35	14	1
1:A:161:VAL:HG21	1:A:214:CYS:HA	0.56	1.77	3	3
1:A:138:LEU:HD21	1:A:151:ARG:HE	0.52	1.65	4	1
1:A:128:TYR:CD2	1:A:182:ILE:HG21	0.51	2.40	12	1
1:A:182:ILE:HG22	1:A:186:GLN:HE22	0.50	1.65	11	1
1:A:149:TYR:CE2	1:A:205:ILE:CD1	0.49	2.93	16	1
1:A:173:ASN:HD22	1:A:173:ASN:C	0.49	2.10	8	2
1:A:139:ILE:HD13	1:A:209:VAL:HG22	0.48	1.86	9	1
1:A:128:TYR:CE2	1:A:182:ILE:HG13	0.48	2.44	12	1
1:A:149:TYR:CE2	1:A:150:TYR:HB2	0.48	2.44	16	1
1:A:172:GLN:CG	1:A:173:ASN:H	0.47	2.23	18	1
1:A:171:ASN:HD21	1:A:223:GLU:HG2	0.46	1.71	14	1
1:A:141:PHE:CZ	1:A:205:ILE:HG23	0.46	2.46	11	2
1:A:171:ASN:HA	1:A:175:PHE:CE2	0.46	2.46	14	3
1:A:175:PHE:CG	1:A:176:VAL:N	0.46	2.83	3	2
1:A:149:TYR:CG	1:A:150:TYR:N	0.46	2.84	16	1
1:A:149:TYR:CZ	1:A:150:TYR:HB2	0.46	2.46	16	1
1:A:149:TYR:CE2	1:A:150:TYR:CG	0.45	3.04	16	1
1:A:128:TYR:CZ	1:A:182:ILE:HG12	0.45	2.47	7	1
1:A:159:ASP:HA	1:A:213:MET:SD	0.42	2.53	8	1
1:A:150:TYR:CD2	1:A:157:TYR:CE2	0.42	3.07	4	1
1:A:203:MET:CE	1:A:206:MET:HG2	0.42	2.45	17	1
1:A:139:ILE:N	1:A:139:ILE:HD12	0.41	2.30	11	2
1:A:157:TYR:HB2	1:A:158:PRO:CD	0.41	2.46	14	1
1:A:203:MET:HE2	1:A:206:MET:SD	0.41	2.55	2	1
1:A:176:VAL:HG13	1:A:177:ARG:H	0.41	1.75	3	1

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Atom-1	Atom-2	Clash(Å)	Distance(Å)	Models Worst	Models Total
1:A:128:TYR:CE2	1:A:164:ARG:HD2	0.40	2.51	11	1
1:A:138:LEU:HD13	1:A:138:LEU:O	0.40	2.16	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	102/111 (92%)	86±3 (85±3%)	12±2 (12±2%)	3±2 (3±2%)	8	40
All	All	2040/2220 (92%)	1729 (85%)	246 (12%)	65 (3%)	8	40

All 21 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	172	GLN	8
1	A	195	GLY	7
1	A	170	SER	6
1	A	127	GLY	5
1	A	167	ASP	4
1	A	168	GLN	4
1	A	227	GLN	4
1	A	175	PHE	4
1	A	128	TYR	3
1	A	166	VAL	3
1	A	169	TYR	3
1	A	171	ASN	2
1	A	174	ASN	2
1	A	126	GLY	2
1	A	140	HIS	2
1	A	197	ASN	1
1	A	221	GLU	1
1	A	176	VAL	1
1	A	149	TYR	1
1	A	154	MET	1

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Mol	Chain	Res	Type	Models (Total)
1	A	155	TYR	1

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	95/100 (95%)	77±3 (81±3%)	18±3 (19±3%)	5	38
All	All	1900/2000 (95%)	1541 (81%)	359 (19%)	5	38

All 64 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	132	SER	17
1	A	225	TYR	17
1	A	208	ARG	16
1	A	136	ARG	16
1	A	134	MET	15
1	A	164	ARG	14
1	A	203	MET	14
1	A	197	ASN	13
1	A	227	GLN	11
1	A	166	VAL	11
1	A	226	TYR	8
1	A	217	GLN	8
1	A	138	LEU	8
1	A	174	ASN	8
1	A	153	ASN	7
1	A	177	ARG	7
1	A	147	ASP	7
1	A	155	TYR	7
1	A	140	HIS	7
1	A	218	TYR	6
1	A	182	ILE	6
1	A	204	LYS	5
1	A	130	LEU	5
1	A	206	MET	5

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Mol	Chain	Res	Type	Models (Total)
1	A	190	THR	5
1	A	223	GLU	5
1	A	176	VAL	5
1	A	168	GLN	5
1	A	187	HIS	5
1	A	157	TYR	4
1	A	171	ASN	4
1	A	150	TYR	4
1	A	188	THR	4
1	A	144	ASP	4
1	A	170	SER	4
1	A	128	TYR	4
1	A	172	GLN	4
1	A	173	ASN	4
1	A	198	PHE	4
1	A	151	ARG	4
1	A	148	ARG	4
1	A	219	GLN	3
1	A	186	GLN	3
1	A	194	LYS	3
1	A	221	GLU	3
1	A	200	GLU	3
1	A	199	THR	3
1	A	196	GLU	3
1	A	169	TYR	3
1	A	222	SER	3
1	A	175	PHE	3
1	A	167	ASP	2
1	A	178	ASP	2
1	A	193	THR	2
1	A	202	ASP	2
1	A	191	THR	2
1	A	220	LYS	1
1	A	160	GLN	1
1	A	163	TYR	1
1	A	154	MET	1
1	A	185	LYS	1
1	A	129	MET	1
1	A	214	CYS	1
1	A	135	SER	1

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 carbohydrates in this entry.

6.6 Ligand geometry (i)

There are no ligands in this entry.

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 80% for the well-defined parts and 80% for the entire structure.

7.1 Chemical shift list 1

File name: BMRB entry 6378

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	1259
Number of shifts mapped to atoms	1249
Number of unparsed shifts	0
Number of shifts with mapping errors	10
Number of shifts with mapping warnings	0
Number of shift outliers (ShiftChecker)	0

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

- Residue not found in structure. All 10 occurences are reported below.

Chain	Res	Type	Atom	Shift Data		
				Value	Uncertainty	Ambiguity
A	1	GLY	HA2	3.87	0.01	1
A	2	SER	CA	57.7	0.1	1
A	1	GLY	CA	42.8	0.1	1
A	2	SER	N	115.8	0.1	1
A	2	SER	CB	63.9	0.1	1
A	2	SER	HB3	3.8	0.01	1
A	2	SER	H	8.64	0.01	1
A	2	SER	HB2	3.8	0.01	1
A	2	SER	HA	4.8	0.01	1
A	1	GLY	HA3	3.87	0.01	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
$^{13}C_\alpha$	110	-0.14 ± 0.12	None needed (< 0.5 ppm)
$^{13}C_\beta$	102	0.55 ± 0.11	Should be applied
$^{13}C'$	0	—	—
^{15}N	103	0.04 ± 0.08	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 80%, i.e. 1044 atoms were assigned a chemical shift out of a possible 1311. 11 out of 11 assigned methyl groups (LEU and VAL) were assigned stereospecifically.

	Total	^{1}H	^{13}C	^{15}N
Backbone	388/504 (77%)	196/201 (98%)	99/204 (49%)	93/99 (94%)
Sidechain	556/670 (83%)	350/395 (89%)	188/235 (80%)	18/40 (45%)
Aromatic	100/137 (73%)	66/71 (93%)	34/64 (53%)	0/2 (0%)
Overall	1044/1311 (80%)	612/667 (92%)	321/503 (64%)	111/141 (79%)

The following table shows the completeness of the chemical shift assignments for the full structure. The overall completeness is 80%, i.e. 1117 atoms were assigned a chemical shift out of a possible 1400. 14 out of 14 assigned methyl groups (LEU and VAL) were assigned stereospecifically.

	Total	^{1}H	^{13}C	^{15}N
Backbone	424/549 (77%)	214/219 (98%)	108/222 (49%)	102/108 (94%)
Sidechain	593/714 (83%)	371/420 (88%)	203/251 (81%)	19/43 (44%)
Aromatic	100/137 (73%)	66/71 (93%)	34/64 (53%)	0/2 (0%)
Overall	1117/1400 (80%)	651/710 (92%)	345/537 (64%)	121/153 (79%)

7.1.4 Statistically unusual chemical shifts (i)

There are no statistically unusual chemical shifts.

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.

Random coil index (RCI) for chain A: