# Full wwPDB NMR Structure Validation Report (i)

Apr 26, 2016 – 02:57 PM BST

| | | |
|---|---|---|
| PDB ID | : | 1IB9 |
| Title | : | SOLUTION STRUCTURE OF MCOTI-II, A MACROCYCLIC TRYPSIN INHIBITOR |
| Authors | : | Felizmenio-Quimio, M.E.; Daly, N.L.; Craik, D.J. |
| Deposited on | : | 2001-03-28 |

This is a Full wwPDB NMR Structure Validation 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 (i) symbol.

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

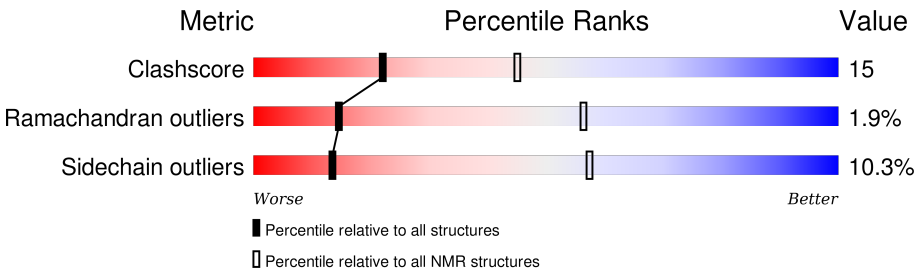
| | | |
|---|---|---|
| Cyrange | : | Kirchner and Güntert (2011) |
| NmrClust | : | Kelley et al. (1996) |
| MolProbity | : | 4.02b-467 |
| Mogul | : | unknown |
| Percentile statistics | : | 20151230.v01 (using entries in the PDB archive December 30th 2015) |
| RCI | : | v_1n_11_5_13_A (Berjanski et 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 (i)

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

The overall completeness of chemical shifts assignment is 47%.

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 | 34 | 41% 24% 35% |

# 2 Ensemble composition and analysis (i)

This entry contains 20 models. Model 4 is the overall representative, medoid model (most similar to other models). The authors have identified model 17 as representative, based on the following criterion: *lowest energy*.

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:13-A:34 (22) | 0.15 | 4 |

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. No single-model clusters were found.

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

# 3 Entry composition (i)

There is only 1 type of molecule in this entry. The entry contains 462 atoms, of which 226 are hydrogens and 0 are deuteriums.

- Molecule 1 is a protein called TRYPSIN INHIBITOR II.

| Mol | Chain | Residues | Atoms | | | | | | Trace |
|---|---|---|---|---|---|---|---|---|---|
| | | | Total | C | H | N | O | S | |
| 1 | A | 34 | 462 | 138 | 226 | 47 | 45 | 6 | 0 |

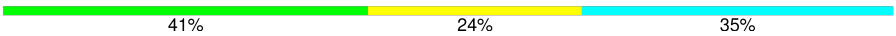
# 4 Residue-property plots (i)

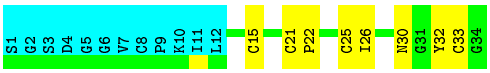
## 4.1 Average score per residue in the NMR ensemble

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: TRYPSIN INHIBITOR II

Chain A:



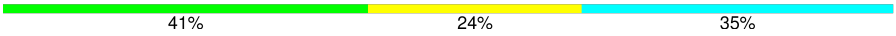

## 4.2 Scores per residue for each member of the ensemble

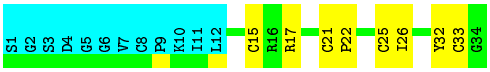
Colouring as in section 4.1 above.

### 4.2.1 Score per residue for model 1

- Molecule 1: TRYPSIN INHIBITOR II

Chain A:

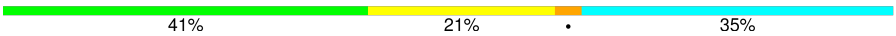


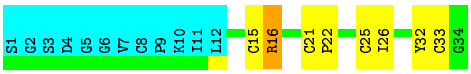




### 4.2.2 Score per residue for model 2

- Molecule 1: TRYPSIN INHIBITOR II

Chain A:

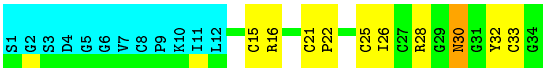


### 4.2.3 Score per residue for model 3

- Molecule 1: TRYPSIN INHIBITOR II

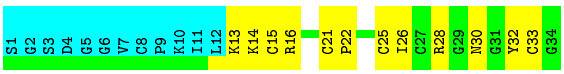
Chain A: 35% 26% • 35%




### 4.2.4 Score per residue for model 4 (medoid)

- Molecule 1: TRYPSIN INHIBITOR II

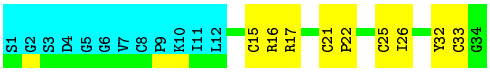
Chain A: 29% 35% 35%




### 4.2.5 Score per residue for model 5

- Molecule 1: TRYPSIN INHIBITOR II

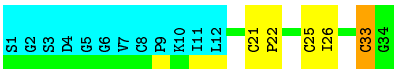
Chain A: 38% 26% 35%




### 4.2.6 Score per residue for model 6

- Molecule 1: TRYPSIN INHIBITOR II

Chain A: 50% 12% • 35%




### 4.2.7 Score per residue for model 7

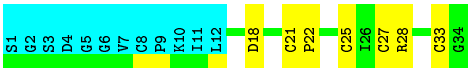
- Molecule 1: TRYPSIN INHIBITOR II

Chain A: 38% 26% 35%

### 4.2.8 Score per residue for model 8

- Molecule 1: TRYPSIN INHIBITOR II

Chain A: 44% 21% 35%




### 4.2.9 Score per residue for model 9

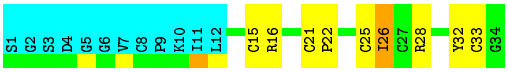
- Molecule 1: TRYPSIN INHIBITOR II

Chain A: 35% 24% 6% 35%

### 4.2.10 Score per residue for model 10

- Molecule 1: TRYPSIN INHIBITOR II

Chain A: 38% 24% • 35%




### 4.2.11 Score per residue for model 11

- Molecule 1: TRYPSIN INHIBITOR II

Chain A: 38% 26% 35%

### 4.2.12 Score per residue for model 12

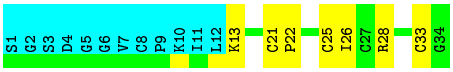
- Molecule 1: TRYPSIN INHIBITOR II

Chain A: 32% 32% 35%

### 4.2.13 Score per residue for model 13

- Molecule 1: TRYPSIN INHIBITOR II

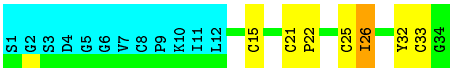
Chain A: 44% 21% 35%




### 4.2.14 Score per residue for model 14

- Molecule 1: TRYPSIN INHIBITOR II

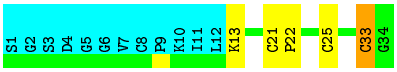
Chain A: 44% 18% • 35%




### 4.2.15 Score per residue for model 15

- Molecule 1: TRYPSIN INHIBITOR II

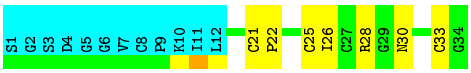
Chain A: 50% 12% • 35%




### 4.2.16 Score per residue for model 16

- Molecule 1: TRYPSIN INHIBITOR II

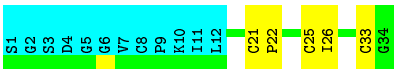
Chain A: 44% 21% 35%




### 4.2.17 Score per residue for model 17

- Molecule 1: TRYPSIN INHIBITOR II

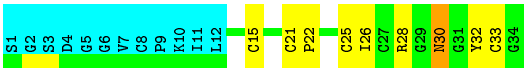
Chain A: 50% 15% 35%



### 4.2.18 Score per residue for model 18

- Molecule 1: TRYPSIN INHIBITOR II

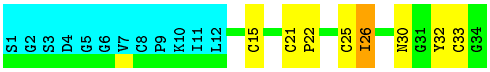
Chain A: 38% 24% • 35%




### 4.2.19 Score per residue for model 19

- Molecule 1: TRYPSIN INHIBITOR II

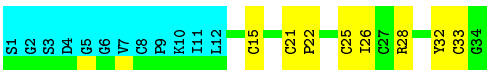
Chain A: 41% 21% • 35%




### 4.2.20 Score per residue for model 20

- Molecule 1: TRYPSIN INHIBITOR II

Chain A: 41% 24% 35%



# 5 Refinement protocol and experimental data overview (i)

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

Of the 50 calculated structures, 20 were deposited, based on the following criterion: *The submitted conformer models are those with the fewest number of restraint violations.*.

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

| Software name | Classification | Version |
|---|---|---|
| DYANA | structure solution | 1.5 |
| X-PLOR | structure solution | 3.851 |
| X-PLOR | refinement | 3.851 |

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 5028 |
|---|---|
| Number of chemical shift lists | 1 |
| Total number of shifts | 210 |
| Number of shifts mapped to atoms | 210 |
| Number of unparsed shifts | 0 |
| Number of shifts with mapping errors | 0 |
| Number of shifts with mapping warnings | 0 |
| Assignment completeness (well-defined parts) | 47% |

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)

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 | 1.15±0.01 | 0±0/160 (0.0±0.0%) | 1.28±0.02 | 1±1/210 (0.6±0.3%) |
| All | All | 1.15 | 0/3200 (0.0%) | 1.28 | 27/4200 (0.6%) |

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($^o$) | Ideal($^o$) | Models | |
|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | Worst | Total |
| 1 | A | 33 | CYS | CA-CB-SG | 6.44 | 125.59 | 114.00 | 11 | 13 |
| 1 | A | 28 | ARG | NE-CZ-NH2 | -5.89 | 117.35 | 120.30 | 3 | 9 |
| 1 | A | 16 | ARG | NE-CZ-NH2 | -5.44 | 117.58 | 120.30 | 9 | 3 |
| 1 | A | 17 | ARG | NE-CZ-NH2 | -5.31 | 117.65 | 120.30 | 5 | 2 |

There are no chirality outliers.

There are no planarity outliers.

## 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 | 159 | 148 | 148 | 5±1 |
| All | All | 3180 | 2960 | 2960 | 93 |

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 15.

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

| Atom-1 | Atom-2 | Clash(Å) | Distance(Å) | Models | |
|---|---|---|---|---|---|
| | | | | Worst | Total |
| 1:A:21:CYS:SG | 1:A:25:CYS:CB | 0.59 | 2.91 | 9 | 20 |
| 1:A:15:CYS:SG | 1:A:32:TYR:C | 0.58 | 2.82 | 9 | 14 |
| 1:A:21:CYS:SG | 1:A:22:PRO:HD2 | 0.57 | 2.40 | 2 | 20 |
| 1:A:21:CYS:SG | 1:A:25:CYS:SG | 0.52 | 3.08 | 4 | 10 |
| 1:A:22:PRO:O | 1:A:25:CYS:SG | 0.50 | 2.69 | 14 | 8 |
| 1:A:16:ARG:HD2 | 1:A:16:ARG:N | 0.46 | 2.25 | 2 | 1 |
| 1:A:26:ILE:N | 1:A:26:ILE:HD13 | 0.46 | 2.26 | 14 | 3 |
| 1:A:21:CYS:SG | 1:A:25:CYS:HB2 | 0.45 | 2.52 | 15 | 2 |
| 1:A:13:LYS:HB2 | 1:A:33:CYS:SG | 0.44 | 2.51 | 9 | 1 |
| 1:A:30:ASN:ND2 | 1:A:32:TYR:H | 0.43 | 2.12 | 9 | 3 |
| 1:A:21:CYS:SG | 1:A:22:PRO:CD | 0.42 | 3.08 | 14 | 6 |
| 1:A:13:LYS:HB3 | 1:A:33:CYS:SG | 0.41 | 2.56 | 15 | 1 |
| 1:A:14:LYS:HD2 | 1:A:14:LYS:N | 0.41 | 2.30 | 12 | 1 |
| 1:A:14:LYS:N | 1:A:14:LYS:HD3 | 0.41 | 2.30 | 7 | 1 |
| 1:A:15:CYS:SG | 1:A:32:TYR:O | 0.40 | 2.80 | 20 | 1 |
| 1:A:18:ASP:HA | 1:A:27:CYS:N | 0.40 | 2.32 | 8 | 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 | 21/34 (62%) | 19±1 (89±4%) | 2±1 (9±5%) | 0±0 (2±2%) | 14 | 56 |
| All | All | 420/680 (62%) | 373 (89%) | 39 (9%) | 8 (2%) | 14 | 56 |

All 1 unique Ramachandran outliers are listed below.

| Mol | Chain | Res | Type | Models (Total) |
|---|---|---|---|---|
| 1 | A | 33 | CYS | 8 |

### 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 | 17/26 (65%) | 15±1 (90±6%) | 2±1 (10±6%) | 13 57 |
| All | All | 340/520 (65%) | 305 (90%) | 35 (10%) | 13 57 |

All 6 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 | 26 | ILE | 18 |
| 1 | A | 30 | ASN | 8 |
| 1 | A | 16 | ARG | 4 |
| 1 | A | 13 | LYS | 3 |
| 1 | A | 17 | ARG | 1 |
| 1 | A | 14 | LYS | 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 47% for the well-defined parts and 48% for the entire structure.

## 7.1 Chemical shift list 1

File name: BMRB entry 5028

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 | 210 |
| Number of shifts mapped to atoms | 210 |
| Number of unparsed shifts | 0 |
| Number of shifts with mapping errors | 0 |
| Number of shifts with mapping warnings | 0 |
| Number of shift outliers (ShiftChecker) | 0 |

### 7.1.2 Chemical shift referencing (i)

No chemical shift referencing corrections were calculated (not enough data).

### 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 47%, i.e. 118 atoms were assigned a chemical shift out of a possible 252. 0 out of 0 assigned methyl groups (LEU and VAL) were assigned stereospecifically.

| | **Total** | $^{1}$**H** | $^{13}$**C** | $^{15}$**N** |
|---|---|---|---|---|
| Backbone | 43/108 (40%) | 43/43 (100%) | 0/44 (0%) | 0/21 (0%) |
| Sidechain | 71/136 (52%) | 71/83 (86%) | 0/41 (0%) | 0/12 (0%) |
| Aromatic | 4/8 (50%) | 4/4 (100%) | 0/4 (0%) | 0/0 (—%) |
| Overall | 118/252 (47%) | 118/130 (91%) | 0/89 (0%) | 0/33 (0%) |

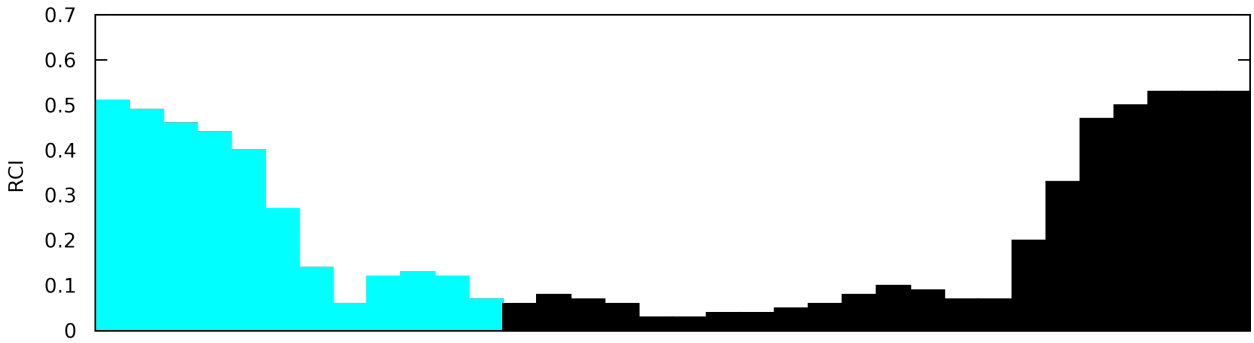
The following table shows the completeness of the chemical shift assignments for the full structure. The overall completeness is 48%, i.e. 176 atoms were assigned a chemical shift out of a possible 369. 0 out of 2 assigned methyl groups (LEU and VAL) were assigned stereospecifically.

| | Total | $^{1}$H | $^{13}$C | $^{15}$N |
|---|---|---|---|---|
| Backbone | 66/166 (40%) | 66/66 (100%) | 0/68 (0%) | 0/32 (0%) |
| Sidechain | 106/195 (54%) | 106/118 (90%) | 0/64 (0%) | 0/13 (0%) |
| Aromatic | 4/8 (50%) | 4/4 (100%) | 0/4 (0%) | 0/0 (—%) |
| Overall | 176/369 (48%) | 176/188 (94%) | 0/136 (0%) | 0/45 (0%) |

### 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: