Table 3. Backbone torsion angles ϕ and ψ in TTR(105–115) fibrils

Residue	ϕ Measurement, °		ψ Measurement, °			ϕ, °*	ψ, °*
	TALOS	$H{-}N_i{-}C^{\alpha}_i{-}H$	TALOS	$H{-}N_{i+1}{-}C^{\alpha}_i{-}H$	$N{-}C^{\alpha}{-}C'{-}N$		
Y105	—	—	—	−180–90 150–180	−168–−160 160–168	—	164 ± 4†
T106	—	−139 – −101	—	90–150	−140–140	−120 ± 19	115 ± 25
I107	−145– −105	−143–−97	131–155	90–150	−137–137	−124 ± 19	134 ± 3
A108	−139–−95	-142 – -98	120–146	92–148	−140–140	−119 ± 21	130 ± 10
A109	−145– −117	−141–−99	111–147	90–150	−139–139	−129 ± 12	125 ± 14
L110	−130–−94	−139– −101	104–136	90–150	−144–144	−116 ± 15	120 ± 16
L111	−140–−96	−140– −100	122–152	90–150	−153–153	−120 ± 20	136 ± 14
S112	−131–−87	−139– −101	109–163	—	−146–146	−116 ± 15	128 ± 19
P113	−112–−50	—	107–145	90–150	−157–157	-81 ± 31	126 ± 19
Y114	−139–−85	−139– −101	123–153	—	—	−120 ± 19	138 ± 15
S115	—	—	—	—	—	—	—

Backbone torsion angles were predicted based on NMR chemical shifts by using the program TALOS (1) and were measured directly by using 3D dipolar-chemical shift correlation experiments, $^{1}H^{N}_i{-}^{15}N_i{-}^{13}C^{\alpha}_i{-}^{1}H^{\alpha}_i$ (2, 3), $^{1}H^{N}_{i+1}{-}^{15}N_{i+1}{-}^{13}C^{\alpha}_i{-}^{1}H^{\alpha}_i$ (3), and $^{15}N_i{-}^{13}C^{\alpha}_i{-}^{13}C'_i{-}^{15}N_{i+1}$ (4, 5). Uncertainties for $^{1}H^{N}_i{-}^{15}N_i{-}^{13}C^{\alpha}_i{-}^{1}H^{\alpha}_i$, $^{1}H^{N}_{i+1}{-}^{15}N_{i+1}{-}^{13}C^{\alpha}_i{-}^{1}H^{\alpha}_i$, and $^{15}N_i{-}^{13}C^{\alpha}_i{-}^{13}C'_i{-}^{15}N_{i+1}$ experiments contain systematic contributions from the potential 2–3° variations in three-atom bond angles (6) and contributions due to random

noise (obtained using an F-test approach with a 90% confidence limit). For the $^{1}H^{N}_{i}$–$^{15}N_{i}$–$^{13}C^{\alpha}_{i}$–$^{1}H^{\alpha}_{i}$ and $^{1}H^{N}_{i+1}$–$^{15}N_{i+1}$–$^{13}C^{\alpha}_{i}$–$^{1}H^{\alpha}_{i}$ experiments the combination of systematic and random uncertainties leads to the coalescence of characteristic pairs of degenerate solutions (about $\phi = -120^{o}$ for $^{1}H^{N}_{i}$–$^{15}N_{i}$–$^{13}C^{\alpha}_{i}$–$^{1}H^{\alpha}_{i}$ and $\psi = 120^{o}$ for $^{1}H^{N}_{i+1}$–$^{15}N_{i+1}$–$^{13}C^{\alpha}_{i}$–$^{1}H^{\alpha}_{i}$) into single minima.

*ϕ and ψ values used in the calculation of the ensemble of low-energy peptide structures were obtained by combining the results of the TALOS and dipolar tensor correlation experiments to obtain a single self-consistent solution.

†For Y105 the only solution consistent with the Y105 $^{13}C^{\beta}$–T106 ^{15}N distance of 3.3 ± 0.33 Å (Table 2) is $\psi = 164 \pm 4°$. The $\psi = -164 \pm 4°$ solution corresponds to a distance of ≈2.8 Å and was therefore neglected.

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