Supporting information for

Assembly of Peptides Derived from β-Sheet Regions of β-Amyloid

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HPLC trace
mass spectrum
1D \(^1\)H NMR spectrum at 0.15 mM in D\(_2\)O at 600 MHz and 293 K
2D TOCSY spectrum at 0.15 mM in D\(_2\)O at 600 MHz and 293 K
2D ROESY spectrum at 0.15 mM in D\(_2\)O at 600 MHz and 293 K
2D DOSY spectrum at 0.15 mM in D\(_2\)O at 500 MHz and 298 K
1D \(^1\)H NMR spectrum at 8.0 mM in D\(_2\)O at 600 MHz and 298 K
2D TOCSY spectrum at 8.0 mM in D\(_2\)O at 600 MHz and 298 K
2D NOESY spectrum at 8.0 mM in D\(_2\)O at 600 MHz and 298 K
2D EXSY spectrum at 8.0 mM in D\(_2\)O at 600 MHz and 318 K
2D DOSY spectrum at 8.0 mM in D\(_2\)O at 500 MHz and 298 K

Peptide 1b
HPLC trace
mass spectrum
1D \(^1\)H NMR spectrum at 1.0 mM in D\(_2\)O at 600 MHz and 293 K
2D TOCSY spectrum at 1.0 mM in D\(_2\)O at 600 MHz and 293 K
2D ROESY spectrum at 1.0 mM in D\(_2\)O at 600 MHz and 293 K
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1D \(^1\)H NMR spectrum at 16.0 mM in D\(_2\)O at 600 MHz and 293 K
2D TOCSY spectrum at 16.0 mM in D\(_2\)O at 600 MHz and 293 K
2D NOESY spectrum at 16.0 mM in D\(_2\)O at 600 MHz and 293 K
2D DOSY spectrum at 16.0 mM in D\(_2\)O at 500 MHz and 298 K

Fmoc-[\(^{15}\)N]Phe-OH
1D \(^1\)H NMR spectrum in CDCl\(_3\) at 500 MHz and 298 K
1D \(^{13}\)C NMR spectrum in CDCl\(_3\) at 500 MHz and 298 K

Fmoc-[\(^{15}\)N]Gly-OH
1D \(^1\)H NMR spectrum in CDCl\(_3\) at 500 MHz and 298 K
1D \(^{13}\)C NMR spectrum in CDCl\(_3\) at 500 MHz and 298 K

Peptide [\(^{15}\)N]1a
2D \(^1\)H,\(^{15}\)N HSQC at 8.0 mM in 9:1 H\(_2\)O/D\(_2\)O at 600 MHz and 293 K

Peptide [\(^{15}\)N]1b
2D \(^1\)H,\(^{15}\)N HSQC at 8.0 mM in 9:1 H\(_2\)O/D\(_2\)O at 600 MHz and 293 K
I. SUPPLEMENTAL FIGURES

Figure S1. $^1$H NMR spectra of peptide 1a at various concentrations in D$_2$O at 600 MHz and 298 K. The 0.3 mM sample contains DSA as an internal standard, which is marked by an asterisk (*).
Figure S2. Expansions of the NOESY spectrum of peptide 1a at 8.0 mM in D$_2$O at 600 MHz and 298 K. Key NOEs associated with β-sheet folding and dimerization are highlighted in blue. The δ Orn pro-$R$ δ-protons are designated Ornδ$R$; the δ Orn pro-$R$ δ-protons are designated Ornδ$R$. 

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Figure S3. Expansions of the NOESY spectrum of peptide 1a at 8.0 mM in D$_2$O at 600 MHz and 298 K. Key interlayer NOEs associated with tetramerization are highlighted in blue.
Figure S4. $^1$H NMR spectra of peptide 1b at various concentrations in D$_2$O at 600 MHz and 298 K. The 1.0 mM sample contains DSA as an internal standard, which is marked by an asterisk (*).
Figure S5. Expansions of the NOESY spectrum of peptide 1b at 16.0 mM in D2O at 600 MHz and 293 K. Key NOEs associated with β-sheet folding and dimerization are highlighted in red. The G33 pro-R α-proton is designated G33α; the δOrn pro-R δ-protons are designated OrnδR; the δOrn pro-R δ-protons are designated OrnδR.
Figure S6. Expansions of the NOESY spectrum of peptide 1b at 8.0 mM in D₂O at 600 MHz and 293 K. Key interlayer NOEs associated with tetramerization are highlighted in red.
Figure S7. $^{15}$N-Edited NOESY spectrum of peptide $[^{15}\text{N}]\text{1a}$ at 8.0 mM in 9:1 H$_2$O/D$_2$O at 600 MHz and 293 K.

Figure S8. $^{15}$N-Edited NOESY spectrum of peptide $[^{15}\text{N}]\text{1b}$ at 8.0 mM in 9:1 H$_2$O/D$_2$O at 600 MHz and 293 K. Crosspeaks associated with chemical exchange between the monomers and tetramers are labeled EX.
II. MATERIALS AND METHODS

General

$N,N$-Dimethylformamide (DMF), 2,4,6-collidine, and piperidine were purchased from Alfa Aesar and used without further purification. HPLC grade acetonitrile (CH$_3$CN) was purchased from VWR International and used without further purification. Methylene chloride (CH$_2$Cl$_2$) was purchased from Fisher Scientific, stored under argon, and passed through a column of alumina before use.$^1$ Boc-Orn(Fmoc)-OH, HCTU, HBTU and HOBT were purchased from GL Biochem Ltd (Shanghai). 2-Chlorotrityl chloride resin and Fmoc protected amino acids were purchased from Chem-Impex International. $N,N$-Diisopropylethylamine (DIPEA), $N$-methylmorpholine (NMM), trifluoroacetic acid (TFA), and triisopropylsilane (TIPS) were purchased from Oakwood Chemical. Isotopically labeled glycine ($^{15}$N, 98%), phenylalanine ($^{15}$N, 98%), and deuterium oxide (D, 99.96%) were purchased from Cambridge Isotope Laboratories, Inc. Fmoc-Hao-OH was synthesized according to previously reported procedures.$^2$
Synthesis of Peptides 1

Resin Loading. 2-Chlorotrityl chloride resin (300 mg, 1.1 meq/g, 100–200 mesh) was suspended in ca. 8 mL of CH₂Cl₂ in a 10-mL Bio-Rad Poly-Prep column and allowed to swell (15 min). The CH₂Cl₂ was drained and a solution of Boc-Orn(Fmoc)-OH (0.22 mmol, 100.0 mg) in CH₂Cl₂ (7.6 mL) and 2,4,6-collidine (0.4 mL), was added. The suspension was agitated gently overnight (10–12 h) and the solution was drained. The capping solution 17:2:1 CH₂Cl₂/MeOH/DIPEA (8 mL) was added. The mixture was agitated gently (1 h), and then the solution was drained.
Solid-Phase Peptide Synthesis. The loaded resin was transferred to a solid-phase peptide synthesis vessel with DMF (3 × 2 mL). Successive rounds of solid-phase peptide synthesis were performed on a PS3™ Peptide Synthesizer (Protein Technologies) using the following conditions: The Fmoc deprotection steps (2 × 5 min) were performed with a 20% piperidine in DMF solution. The coupling steps (1 × 20 min) were performed for the amino acids (4 equiv) with HCTU (4 equiv) and a 20% 2,4,6-collidine in DMF solution. The unnatural amino acid Fmoc-Hao-OH (2 equiv) was coupled twice with 2 equiv of HCTU per coupling (60 min) to achieve complete coupling. DMF was used to rinse the resin after each deprotection (6 × 3 mL) and after each amino acid coupling (6 × 3 mL).

Cleavage from Resin. After the synthesis of each peptide was complete, the resin was transferred into the Poly-Prep column with CH₂Cl₂ (ca. 2 mL) and the solution was drained. The solid-phase peptide synthesis vessel was rinsed with ca. two additional portions of CH₂Cl₂ to ensure the complete transfer of the resin and the removal of DMF. A 1:4 HFIP/CH₂Cl₂ solution (8 mL) was added to the resin and the mixture was agitated gently. After 1 h, the solution was drained into a 250-mL round-bottom flask and the treatment with HFIP/CH₂Cl₂ solution was repeated. The combined solutions were evaporated under vacuum to give the protected linear peptides 1.

Cyclization. The protected linear peptides 1 were cyclized with HBTU (5 equiv), HOBt (5 equiv), and NMM (8 equiv) in a solution of DMF (125 mL). The solution was stirred under N₂ overnight (12–24 h), and then the DMF was evaporated under vacuum. The peptides were placed under vacuum (ca. 0.1 mmHg) overnight to ensure complete removal of any residual DMF.
Deprotection. The protected cyclic peptides 1 were deprotected under acidic conditions with a solution of 18:1:1 TFA/triisopropylsilane/H₂O (10 mL). The solution was stirred for 2 h, then evaporated under vacuum. For peptides containing a methionine (1b and [¹⁵N]1b), 50 mg of dithiothreitol (DTT) was added to the solution to prevent sulfur oxidation.

RP-HPLC Purification. The peptides were suspended in a solution of 20% aqueous CH₃CN (ca. 8 mL) and the suspensions were filtered through a 0.2 μm filter. The purity of each peptide was analyzed by analytical RP-HPLC on a Phenomenex Aeris 2.6μ XB-C18 column (150 mm x 4.6 mm) with a 5–100% gradient over 20 min of CH₃CN in H₂O with 0.1% TFA at 1.0 mL/min. The purification of each peptides was performed by preparative RP-HPLC on an Agilent Zorbax 7 μM SB-C18 Prep HT column (21.2 mm x 250 mm) with a 15–30% gradient over 10 min and 30–60% gradient over 45 min of CH₃CN in H₂O with 0.1% TFA at 15.0 mL/min. The pure fractions were combined and concentrated under vacuum. The peptides were re-suspended in a solution of H₂O with 0.1% TFA (ca. 10–15 mL), then lyophilized to give peptides 1 as a white powder in 8–22% yield (30–80 mg) based on the resin loading of the first amino acid Boc-Orn(Fmoc)-OH).
Fmoc-Protection of $^{15}$N-Labeled Amino Acids

Fmoc-$^{15}$N-Phe-OH: A 100-mL one-neck round-bottom flask equipped with a magnetic stirring bar was charged with $^{15}$N-labeled phenylalanine (1.0 g, 6 mmol) and a solution of 1:1 CH$_3$CN/H$_2$O (50 mL). Et$_3$N (0.6 g, 6 mmol) and Fmoc-OSu (1.9 g, 5.7 mmol) were added, then the reaction mixture was stirred until the solution turned clear (ca. 15 min). Additional Et$_3$N was added until the pH was roughly 8.5, then the mixture was stirred for 1 h. The mixture was poured into a solution of 1.0 M HCl (250 mL) in a 400-mL beaker while stirring vigorously. The Fmoc-$^{15}$N-Phe-OH precipitated from the solution and the solid was isolated by filtering the mixture through a sintered glass filter funnel with a medium frit. The funnel was covered with a piece of filter paper and the solid was dried by aspirating air through the funnel. The solid was suspended in ca. 200 mL of EtOAc to form a turbid solution. The solution was stirred vigorously for 10 min, dried over MgSO$_4$, filtered, and then concentrated under vacuum to give a white solid. The isolated solid was ground into a fine powder to give ca. 1.94 g (92%). $^1$H NMR (500 MHz, CDCl$_3$): $\delta$ 7.77 (d, $J = 7.5$ Hz, 2H), 7.55 (t, $J = 6.2$ Hz, 2H), 7.40 (t, $J = 7.4$ Hz, 2H), 6.80 (m, 5H), 7.15 (d, $J = 6.6$ Hz, 2H), 5.19 (dd, $J = 91.9$, 8.2 Hz, 1H), 4.70 (m, 1H), 4.46 (dd, $J = 10.4$, 7.3 Hz, 1H), 4.37 (t, $J = 8.7$ Hz, 1H), 4.21 (t, $J = 6.7$ Hz, 1H), 3.18 (m, 2H); $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ 175.2, 156.0 (d, $^{1}J_{CN} = 25$ Hz), 144.0, 141.6, 135.6, 129.6, 129.0, 128.0, 127.6, 127.3, 125.3, 120.3, 67.3, 54.7 (d, $^{1}J_{CN} = 13.8$ Hz), 47.4, 37.9, 30.0.
Fmoc-[\textsuperscript{15}N]Gly-OH: A 100-mL one-neck round-bottom flask equipped with a magnetic stirring bar was charged with \textsuperscript{15}N-labeled glycine (1.0 g, 13 mmol) and a solution of 1:1 CH\textsubscript{3}CN/H\textsubscript{2}O (50 mL). Et\textsubscript{3}N (1.3 g, 13 mmol) and Fmoc-OSu (4.2 g, 12.5 mmol) were added, then the reaction mixture was stirred until the solution turned clear (ca. 15 min). Additional Et\textsubscript{3}N was added until the pH was roughly 8.5, then the mixture was stirred for 1 h. The mixture was poured into a solution of 1.0 M HCl (250 mL) in a 400-mL beaker while stirring vigorously. The Fmoc-[\textsuperscript{15}N]Gly-OH precipitated from the solution and the solid was isolated by filtering the mixture through a sintered glass filter funnel with a medium frit. The funnel was covered with a piece of filter paper and the solid was dried by aspirating air through the funnel. The solid was suspended in ca. 200 mL of EtOAc to form a turbid solution. The solution was stirred vigorously for 10 min, dried over MgSO\textsubscript{4}, filtered, and then concentrated under vacuum to give a white solid. The isolated solid was ground into a fine powder to give ca. 3.48 g (92%) isolated yield. \textsuperscript{1}H NMR (500 MHz, CDCl\textsubscript{3}): \textsuperscript{\delta} 7.77 (d, \textit{J} = 7.5 Hz, 2H), 7.60 (d, \textit{J} = 7.4 Hz, 2H), 7.40 (t, \textit{J} = 7.5 Hz, 2H), 7.32 (t, \textit{J} = 7.5, 1 Hz, 2H), 5.28 (dt \textit{J} = 92.6, 5.6 Hz), 4.43 (d, \textit{J} = 7.0 Hz, 2H), 4.24 (t, \textit{J} = 7.0 Hz, 1H), 4.04 (d, 5.5 Hz, 2H); \textsuperscript{13}C NMR (125 MHz, CDCl\textsubscript{3}): \textsuperscript{\delta} 173.6, 156.5, 144.0, 141.6, 128.0, 127.3, 125.3, 120.3, 67.6, 47.3, 42.6 (d, \textit{J}_{\text{CN}} = 13.8 Hz).
NMR Spectroscopy of Peptides 1

Sample Preparation. NMR spectroscopy of peptides 1a and 1b was performed in D$_2$O. The solutions were prepared by dissolving a weighed portion of the peptide in the appropriate volume of solvent. The molecular weights of the peptides were calculated as the TFA salts with all amino groups assumed to be protonated (1a, M.W. 2223.85 and 1b, M.W. 2099.91). The solutions were allowed to stand for 24 h to allow complete hydrogen to deuterium exchange of the amide NH protons.

$^1$H NMR, TOCSY, ROESY, and NOESY Data Collection. NMR spectra were recorded on a Bruker 600 MHz spectrometer with a TBI probe. Presaturation water suppression was applied as needed. TOCSY spectra were recorded with 2048 points in the $f_2$ dimension and 512 increments in the $f_1$ dimension with a 150-ms spin-lock mixing time. ROESY spectra were recorded with 2048 points in the $f_2$ dimension and 512 increments in the $f_1$ dimension with a 200-ms spin-lock mixing time. NOESY spectra were recorded with 2048 points in the $f_2$ dimension and 512 increments in the $f_1$ dimension with a 150-ms mixing time.

$^1$H NMR, TOCSY, ROESY, and NOESY Data Processing. NMR spectra were processed with Bruker XwinNMR software. Automatic baseline correction was applied in both dimensions after phasing the spectra. TOCSY and ROESY spectra were Fourier transformed to a final matrix size of 2048 x 1024 real points using a Qsine weighting function and forward linear prediction. NOESY spectra were Fourier transformed to a final matrix size of 2048 x 2048 real points using a Qsine weighting function and forward linear prediction.
**Diffusion-Ordered Spectroscopy (DOSY) Experiments.** DOSY experiments were performed on a Bruker 500 MHz spectrometer equipped with a TCI cryoprobe, with a diffusion delay ($\Delta$) of 75-ms and a diffusion gradient length ($\delta$) of 2.5-ms. Sixteen sets of FIDs were recorded with the gradient strength incremented from 5%–95% using a linear ramp. The combined FIDs were Fourier transformed in Bruker's TopSpin™ software to give a pseudo-2D spectrum. After phasing and performing baseline correction, each pseudo-2D spectrum was processed with logarithmic scaling on the Y-axis. The Y-axis was calibrated to the diffusion coefficient of the residual HOD peak in D$_2$O ($1.9 \times 10^{-9}$ m$^2$/s at 298 K). The diffusion coefficients of the peptides were read and converted from logarithmic values to linear values.

**NMR Spectroscopy of Peptides $^{15}$N1**

*Sample Preparation.* NMR spectroscopy of peptides $^{15}$N1a and $^{15}$N1b was performed in 9:1 H$_2$O/D$_2$O. The solutions were prepared by dissolving a weighed portion of the peptide in the appropriate volume of solvent. The molecular weights of the peptides were calculated as the TFA salts with all amino groups assumed to be protonated ($^{15}$N1a, M.W. 2224.85 and $^{15}$N1b, M.W. 2100.91). 4,4-Dimethyl-4-silapentane-1-ammonium trifluoroacetate (DSA) was added as an internal standard for referencing chemical shifts.
1H NMR, 1H,15N HSQC, and 1H,15N NOESY-HSQC (15N-edited NOESY) Data Collection. NMR spectra were recorded on a Bruker 600 MHz spectrometer with either a TBI probe or a BBFO cryoprobe. Gradient water suppression was applied as needed. 1H,15N HSQC spectra were recorded with 1024 points in the $f_2$ dimension and 512 increments in the $f_1$ dimension. 1H,15N NOESY-HSQC spectra were recorded with a 150-ms mixing time, and with 2048 points in the $f_3$ dimension ($1H$), 1 increment in the $f_2$ dimension ($15N$), and 1024 increments in the $f_1$ dimension ($1H$).

1H NMR, 1H,15N HSQC, and 1H,15N NOESY-HSQC (15N-edited NOESY) Data Processing. NMR spectra were Fourier transformed in Bruker XwinNMR software with forward linear prediction and a Qsinc weighting function. Automatic baseline correction was applied in both dimensions after phasing the spectra. The 1H,15N HSQC spectra were processed to a final matrix size of 2048 x 1024 real points and with GB = 0.1 in the $f_2$ dimension. The 1H,15N NOESY-HSQC spectra were processed to a final 2D matrix size of 4096 x 2048 real points ($f_3, f_1$) and with GB = 0.05 in both dimensions.
**Molecular Modeling of Peptides 1a and 1b.**

Molecular models of the tetramers of peptides 1a and 1b were generated from the X-ray crystallographic structure of a similar macrocyclic β-sheet peptide (PDB 3T4G). This peptide contains AIIGLMV (Aβ30–36) in the heptapeptide strand and KFFBrK in positions R8-R11 in the template strand. The PDB coordinates were imported into PyMOL. Symmetry mates were generated to create two hydrogen-bonded dimers sandwiched on the surface displaying the side chains of A30, I32, L34, and V36. The alignment of each dimer was shifted by two residues to match the alignment of the dimers of peptides 1a and 1b. The residues of the dimers were mutated to match peptide 1a or peptide 1b, and the side chain torsion angles of χ₁ and χ₂ were adjusted for Ile (180° and 60°) and Phe (180°). The dimers were then rotated manually to reflect the observed interlayer NOEs between Ile₁₁ and the methoxy group of Hao.

The coordinates were exported from PyMOL. [Note that .pdb was used, but .mol2 file format is actually preferable and is recommended instead of .pdb.] The file was imported into MacroModel with the Maestro user interface. Atom types and bond orders were edited as needed to correct errors in bond type and charge. Distance constraints were applied to reflect the folding and dimerization of the macrocycles. Four interlayer distance constraints between the δ-methyl group of Ile₁₁ and the methoxy group of Hao were applied to reflect the observed interlayer contacts. Minimization was performed with the MMFFs force field and GB/SA water solvation. All constraints were removed and minimization was repeated to generate a minimum-energy conformation (local minimum). The coordinates were exported in .pdb file format and imported into PyMOL.
III. REFERENCES

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IV. CHARACTERIZATION DATA
RP-HPLC of peptide 1a

MS (ESI) of peptide 1a

| Peak | RetTime | Type | Width | Area   | Height | Area   | %    |
|------|---------|------|-------|--------|--------|--------|------|
| #    | [min]   |      | [min] | [mAU]  | [s]    | [mAU]  | [%]  |
| 1    | 9.997   | BV   | 0.1928| 2.50833e4 | 1820.18323 | 100.0000 |

Totals:
2.50833e4 1820.18323

calculated m/z for C_{85}H_{130}N_{20}O_{21}:

- [M+H]^+: 1767.98
- [2M+3H]^3+: 1178.98
- [M+2H]^2+: 884.49
- [M+3H]^3+: 590.00

column: Aeris XB-C18 2.6µ
dimensions: 150 mm x 4.6 mm
mobile phase: A: H_2O, 0.1% TFA
B: CH_3CN, 0.1% TFA
gradient: A/B (95:5) to (0:100) in 20 min
flow rate: 1.0 mL/min
detection: VWD, wavelength = 214 nm
temperature: 298 K
NT_iv_1a-1 24 (0.440) Sb (1,10.00); Sm (Mn, 4x3.00); Cm (23:31) TOF MS ES+

1760 1765 1770 1775 1780 1785 1790 1795 1800 1805 1810

% 0 100

[5+H]+ 
[2M+4H]2+

1768.73
1768.24
1767.75
1769.24
1769.74
1770.22
1797.46

1770.73
1770.22
1797.46

1806.71

[2M+Na+K]2+

1160 1165 1170 1175 1180 1185 1190 1195 1200 1205 1210

% 0 100

[2M+3H]3+

1179.16
1179.49
1179.82

1178.83
1180.16
1180.50

1179.49
1178.83

468

1180.50

1180.71

[2M+Na+K]2+

S22
$^1$H NMR of peptide 1a, 0.15 mM in D$_2$O at 600 MHz and 293 K

![NMR spectrum image]

Current Data Parameters

USER             ntruex
NAME     NT_iv_130_1a_0.15mM_p
EXPNO                11
PROCNO                1

F2 - Acquisition Parameters
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PULPROG            zg30
TD                97922
SOLVENT             D2O
NS                  512
DS                    2
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A Q            5.0919938 sec
RG                 12.7
DW               52.000 usec
DE                13.70 usec
TE                293.0 K
D1           0.10000000 sec

--- CHANNEL f1 ---
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NUC1                 1H
P1                12.00 usec

F2 - Processing parameters
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WDW                  EM
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GB                    0
PC                 1.00

1D NMR plot parameters
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F1                 5401.17 Hz
F2P               0.000 ppm
F2                 0.00 Hz
PPMCM           0.39474 ppm/cm
HZCM          236.89343 Hz/cm

--- CHANNEL f2 ---
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NUC2                 2H
P2                12.00 usec

F2 - Processing parameters
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SF          500.1299881 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.00

10 MHz plot parameters
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CY                2.16 cm
FP                5.000 ppm
F1             540.17 Hz
F2                0.000 ppm
F3                0.00 Hz
SNOM          0.99414 ppm/cm
SNM          268.8043 Hz/cm
$^1$H NMR of peptide 1a, 0.15 mM in D$_2$O at 600 MHz and 293 K

Current Data Parameters
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PROC  1

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TD  97922
SOLVENT  D$_2$O
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DS  2
SWH     9615.385 Hz
A Q  5.0919938 sec
RG  12.7
DW  52.000 usec
DE  13.70 usec
TE  293.0 K
D1  0.10000000 sec

--- GRAPHICAL FT KW---
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SR2  36
P1  2.00 sec

FT - Processing parameters
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W0  0.000
LB  1.00 Hz
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PC  1.00

1D NMR plot parameters:
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CY  2.06 cm
FP  5120000
P1  5521200 Hz
DF  14000000
F2  4440.96 Hz
SBNM  0.07955 ppm/cm
SNW  47.3988 Hz/cm
\(^1\)H NMR of peptide 1a, 0.15 mM in D\(_2\)O at 600 MHz and 293 K
$^1$H NMR of peptide 1a, 0.15 mM in D$_2$O at 600 MHz and 293 K
$^1$H NMR of peptide 1a, 0.15 mM in D$_2$O at 600 MHz and 293 K

Current Data Parameters
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NAME: _iv_150_1a_0.15mM_p
EXPERIMENT: 1

F2 - Acquisition Parameters
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PULPROG: zg30
TD: 97922
SOLVENT: D$_2$O
NS: 512
DS: 2
SWH: 9615.385 Hz
FIDRES: 0.098194 Hz
A Q: 5.0919938 sec
RG: 12.7
DW: 52.000 usec
DE: 13.70 usec
TE: 293.0 K
D1: 0.1000000 sec

F2 - Processing parameters
SI: 65536
SF: 600.1299881 MHz
WDW: EM
SSB: 0
LB: 1.00 Hz
PC: 1.00

1D NMR plot parameters
CX: 22.80 cm
CY: 15.00 cm
F1P: 3.900 ppm
F2P: 2.100 ppm
PPMCM: 0.07895 ppm/cm
HZCM: 47.37869 Hz/cm
$^1$H NMR of peptide 1a, 0.15 mM in D$_2$O at 600 MHz and 293 K

Current Data Parameters

USER: coroes
NAME: _nt_130_1a_0.15mM_p
EXPERIMENT: -
PROCEDURE: -

F1 - Acquisition Parameters
Date: 20160423
Time: 21.26
INSTRUM: av600
PROBHD: 5 mm CPBBO BB-
PULPROG: zg30
TD: 97922
SOLVENT: D$_2$O
NS: 512
DS: 2
SWH: 9615.385 Hz
A Q: 5.0919938 sec
RG: 12.7
DW: 52.000 usec
DE: 13.70 usec
TE: 293.0 K
D1: 0.10000000 sec

F1 - Processing parameters
SI: 65536
SF: 600.1299881 MHz
WDW: EM
SSB: 0
LB: 1.00 Hz
GB: 0
PC: 1.00

$^1$D NMR plot parameters
CX: 22.80 cm
CY: 15.00 cm
F1P: 2.100 ppm
F1: 1260.27 Hz
F2P: 0.300 ppm
F2: 180.04 Hz
PPMCM: 0.07895 ppm/cm
HZCM: 47.37869 Hz/cm
$^1$H NMR 2D TOCSY of peptide 1a with presaturation suppression of the HOD peak
0.15 mM in D$_2$O at 600 MHz and 293 K with 150-ms spin-lock mixing time
$^1$H NMR 2D TOCSY of peptide 1a with presaturation suppression of the HOD peak
0.15 mM in D$_2$O at 600 MHz and 293 K with 150-ms spin-lock mixing time
$^{1}$H NMR 2D ROESY of peptide 1a with presaturation suppression of the HOD peak 0.15 mM in $\text{D}_2\text{O}$ at 600 MHz and 293 K with 200-ms spin-lock mixing time
$^1$H NMR 2D ROESY of peptide 1a with presaturation suppression of the HOD peak 0.15 mM in D$_2$O at 600 MHz and 293 K with 200-ms spin-lock mixing time.
$^{1}H$ NMR DOSY of peptide 1a, 0.15 mM in D$_2$O at 500 MHz and 298 K

Calculations for peptide 1a at 0.15 mM

$D_{HOD} = 19.0 \times 10^{-10}$ m$^2$/s $^a$

$log (D_{HOD}) = -8.721$

$D_{monomer} \colon log(D) = -9.69; D = 10^{-9.69} = 20.4 \pm 1.7 \times 10^{-11}$ m$^2$/s

$D_{tetramer} \colon log(D) = -9.90; D = 10^{-9.90} = 12.6 \pm 1.6 \times 10^{-11}$ m$^2$/s

$^a$Longsworth, L. G. J. Phys. Chem. 1960, 64, 1914–1917.
$^1$H NMR of peptide 1a, 8 mM in D$_2$O at 600 MHz and 298 K
$^1$H NMR of peptide 1a, 8 mM in D$_2$O at 600 MHz and 298 K
$^1$H NMR of peptide 1a, 8 mM in D$_2$O at 600 MHz and 298 K

Current Data Parameters

- **NAME**: NT_iii_112_1a_8mM_D2O
- **EXPNO**: 21
- **PROCNO**: 1

**F2 - Acquisition Parameters**

- **Date**: 20150825
- **Time**: 21.12
- **INSTRUM**: av600
- **PROBHD**: 5 mm TBI 1H/13
- **PULPROG**: zg30
- **TD**: 97922
- **SOLVENT**: D$_2$O
- **NS**: 8
- **DS**: 2
- **SWH**: 9615.385 Hz
- **FIDRES**: 0.098194 Hz
- **A Q**: 5.0919938 sec
- **RG**: 181
- **DW**: 52.000 usec
- **DE**: 14.54 usec
- **TE**: 298.0 K
- **D1**: 0.10000000 sec
- **TD0**: 1

**F1 - Processing parameters**

- **SI**: 65536
- **SF**: 600.1300240 MHz
- **WDW**: EM
- **SSB**: 0
- **LB**: 0.30 Hz
- **GB**: 0
- **PC**: 1.00

**1D NMR plot parameters**

- **CX**: 2.86 cm
- **CY**: 2.00 cm
- **FP**: 1.00 ppm
- **FI**: 450.98 Hz
- **HZ**: 4000 ppm
- **R1**: 348.15 Hz
- **HCM**: 0.0045 ppm/cm
- **RMW**: 44.9655 Hz/cm
1H NMR of peptide 1a, 8 mM in D2O at 600 MHz and 298 K
$^1$H NMR of peptide 1a, 8 mM in D$_2$O at 600 MHz and 298 K
1H NMR of peptide 1a, 8 mM in D2O at 600 MHz and 298 K
$^1$H NMR 2D TOCSY of peptide 1a with presaturation suppression of the HOD peak
8 mM in D$_2$O at 600 MHz and 298 K with 150-ms spin-lock mixing time
$^1$H NMR 2D TOCSY of peptide 1a with presaturation suppression of the HOD peak
8 mM in D$_2$O at 600 MHz and 298 K with 150-ms spin-lock mixing time
$^1$H NMR 2D TOCSY of peptide 1a with presaturation suppression of the HOD peak
8 mM in D$_2$O at 600 MHz and 298 K with 150-ms spin-lock mixing time
$^1$H NMR 2D NOESY of peptide 1a with presaturation suppression of the HOD peak
8 mM in D$_2$O at 600 MHz and 298 K with 150-ms mixing time
$^1$H NMR 2D NOESY of peptide 1a with presaturation suppression of the HOD peak
8 mM in D$_2$O at 600 MHz and 298 K with 150-ms mixing time
$^1$H NMR 2D NOESY of peptide 1a with presaturation suppression of the HOD peak 8 mM in D$_2$O at 600 MHz and 298 K with 150-ms mixing time
$^1$H NMR 2D NOESY of peptide 1a with presaturation suppression of the HOD peak
8 mM in D$_2$O at 600 MHz and 298 K with 150-ms mixing time
$^1$H NMR 2D EXSY of peptide 1a with presaturation suppression of the HOD peak

8 mM in D$_2$O at 600 MHz and 318 K with 200-ms spin-lock mixing time

Exchange crosspeaks are shown in red;
ROE crosspeaks are shown in black.
$^1$H NMR 2D EXSY of peptide 1a with presaturation suppression of the HOD peak
8 mM in D$_2$O at 600 MHz and 318 K with 200-ms spin-lock mixing time
Exchange crosspeaks are shown in red; ROE crosspeaks are shown in black.
$^1$H NMR 2D EXSY of peptide 1a with presaturation suppression of the HOD peak
8 mM in D$_2$O at 600 MHz and 318 K with 200-ms spin-lock mixing time
Exchange crosspeaks are shown in red;
ROE crosspeaks are shown in black.
1H NMR DOSY of peptide 1a, 8 mM in D2O at 500 MHz and 298 K
tetramer predominates

Calculations for peptide 1a at 8.0 mM

\[ D_{\text{HOD}} = 19.0 \times 10^{-10} \text{ m}^2/\text{s} \]

\[ \log (D_{\text{HOD}}) = -8.721 \]

\[ D_{\text{tetramer}}: \log(D) = -9.928; \quad D = 10^{-9.928} = 11.8 \pm 1.0 \times 10^{-11} \text{ m}^2/\text{s} \]

\(^a\)Longsworth, L. G. J. Phys. Chem. 1960, 64, 1914–1917.
RP-HPLC of peptide 1b

MS (ESI) of peptide 1b

Peak RetTime Type Width Area Height Area
# [min] [min] mAU *s [mAU ] %
----- | ------- | ---- |-------|----------|----------|--------|
1 10.621 BV 0.2642 2.29549e4 1098.58997 100.0000
Totals : 2.29549e4 1098.58997

calculated m/z for C_{77}H_{134}N_{20}O_{17}S_{1}:  

| [M+H]^+ | 1644.00 |
| [2M+3H]^3+ | 1096.33 |
| [M+2H]^2+ | 822.51 |
| [M+3H]^3+ | 548.67 |

Aeris XB-C18 2.6µ
150 mm x 4.6 mm
A: H$_2$O, 0.1% TFA
B: CH$_3$CN, 0.1% TFA
A/B (95:5) to (0:100) in 20 min
1.0 mL/min
VWD, wavelength = 214 nm
298 K

column: Aeris XB-C18 2.6µ
dimensions: 150 mm x 4.6 mm
mobile phase: A: H$_2$O, 0.1% TFA
B: CH$_3$CN, 0.1% TFA
gradient: A/B (95:5) to (0:100) in 20 min
flow rate: 1.0 mL/min
detection: VWD, wavelength = 214 nm
temperature: 298 K
$^1$H NMR of peptide 1b, 1 mM in D$_2$O at 600 MHz and 293 K

Current Data Parameters

| Parameter       | Value     |
|-----------------|-----------|
| Date_           | 20160121  |
| Time             | 20.50     |
| INSTRUM          | av600     |
| PROBHD           | 5 mm TBI 1H/13 |
| PULPROG         | zg30      |
| TD               | 97922     |
| SOLVENT         | D$_2$O    |
| NS               | 32        |
| DS               | 2         |
| SWH             | 9615.385 Hz |
| A Q             | 5.0919938 sec |
| RG               | 322       |
| DW               | 52.000 usec |
| DE               | 14.54 usec |
| TE               | 292.9 K   |
| D1               | 0.10000000 sec |

F2 - Acquisition Parameters

| Parameter       | Value     |
|-----------------|-----------|
| SI               | 65536     |
| SF               | 600.1299898 MHz |
| WDW             | EM        |
| LB               | 0.30 Hz   |
| GB               | 0         |
| PC               | 1.00      |

F2 - Processing parameters

| Parameter       | Value     |
|-----------------|-----------|
| DI               | 0         |
| DP               | 0.10 Hz   |
| PE               | 0         |

1D NMR plot parameters

| Parameter       | Value     |
|-----------------|-----------|
| CX              | 22.80 cm  |
| CY              | 600.11 ppm |
| F1P             | -0.100 ppm |
| F2P             | -0.100 ppm |
| F1              | -0.100 Hz |
| F2              | -0.100 Hz |
| PPMCM           | 0.37719 ppm/cm |
| HZCM            | 226.3648 Hz/cm |
$^1$H NMR of peptide 1b, 1 mM in D$_2$O at 600 MHz and 293 K
$^1$H NMR of peptide 1b, 1 mM in D2O at 600 MHz and 293 K

Current Data Parameters

USER: nitex

NAME: MT_iv_32

PROCNO: 1

F2 - Acquisition Parameters

Date: 20160121

Time: 20.50

INSTRUM: av600

Solvent: D2O

NS: 32

DS: 2

SWH: 9615.385 Hz

FIDRES: 0.098194 Hz

A Q: 5.0919938 sec

RG: 322

DW: 52.0 usec

DE: 14.54 usec

TE: 292.9 K

D1: 0.10000000 sec

TD0: 1

Channel f1:

FSO1: 600.1342009 MHz

F1: 8.00 usec

Channel F2:

HF: 605.26 MHz

F2: 0.1299898 MHz

DM: 51.000 usec

DG: 14.54 usec

D1: 292.9 K

D1: 0.10000000 sec

D2: 1

1D NMR plot parameters

CX: 22.80 cm

CY: 600.00 cm

F1P: 5.500 ppm

F2P: 3.800 ppm

PMCM: 0.07456 ppm/cm

HZCM: 44.74654 Hz/cm
1H NMR of peptide 1b, 1 mM in D2O at 600 MHz and 293 K

---

Current Data Parameters

USER: ntruex
NAME: MT_iv_32
EXPER: 11
PROCNO: 1

F2 - Acquisition Parameters

Date: 20160121
Time: 20:50
INSTRUM: av600
PROBHD: 5 mm TBI 1H/13
TD: 97922
SOLVENT: D2O
NS: 32
DW: 52.000 usec
TE: 292.9 K
D1: 0.10000000 sec

F2 - Processing parameters

SI: 65536
SF: 600.1299898 MHz
LB: 0.30 Hz
GB: 0
PC: 1.00

1D NMR plot parameters

CX: 22.80 cm
CY: 10.00 cm
F1P: 2.300 ppm
F2P: 2.300 ppm
F1: 180.30 Hz
F2: 180.30 Hz
PPCM: 0.0679 ppm/cm
HCMD: 39.4622 Hz/cm
**1H NMR of peptide 1b, 1 mM in D₂O at 600 MHz and 293 K**

| ppm | Integral | 2.038 | 0 | 2.024 | 7 | 2.004 | 3 | 1.997 | 5 | 1.989 | 6 | 1.926 | 1 | 1.919 | 8 | 1.913 | 7 | 1.909 | 4 | 1.899 | 0 | 1.888 | 0 | 1.881 | 7 | 1.876 | 3 | 1.870 | 4 | 1.864 | 2 | 1.856 | 8 | 1.852 | 4 | 1.846 | 6 | 1.842 | 4 | 1.836 | 3 | 1.825 | 0 | 1.809 | 7 | 1.803 | 6 | 1.794 | 6 | 1.779 | 7 | 1.774 | 2 | 1.767 | 1 | 1.753 | 9 | 1.741 | 1 | 1.728 | 1 | 1.711 | 3 | 1.698 | 5 | 1.685 | 1 | 1.672 | 2 | 1.642 | 9 | 1.639 | 4 | 1.558 | 3 | 1.554 | 3 | 1.543 | 1 | 1.524 | 4 | 1.519 | 8 | 1.512 | 2 | 1.502 | 7 | 1.496 | 4 | 1.481 | 4 | 1.475 | 8 | 1.472 | 2 | 1.459 | 0 | 1.454 | 1 | 1.447 | 1 | 1.441 | 3 | 1.428 | 5 | 1.423 | 9 | 1.418 | 6 | 1.411 | 9 | 1.370 | 2 | 1.358 | 2 | 1.224 | 4 | 1.217 | 5 | 1.212 | 1 | 1.200 | 4 | 1.187 | 9 | 1.133 | 4 | 1.120 | 7 | 0.961 | 7 | 0.950 | 4 | 0.901 | 6 | 0.896 | 9 | 0.889 | 4 | 0.885 | 6 | 0.874 | 0 | 0.859 | 8 | 0.853 | 7 | 0.846 | 6 | 0.842 | 3 | 0.813 | 3 | 0.801 | 7 | 0.797 | 9 | 0.792 | 3 | 0.788 | 2 | 0.779 | 8 | 0.774 | 2 | 0.763 | 4 | 0.736 | 7 | 0.725 | 1 | 0.717 | 6 | 0.552 | 8 | 0.545 | 6 | 0.538 | 5 | 0.531 | 9 | 0.524 | 0 |

**Current Data Parameters**

**USER**

**NAME**

**MET_iv_92**

**PROCNO**

**F2 - Acquisition Parameters**

**Date**

**Time**

**INSTRUM**

**PROBHD**

**PULPROG**

**TD**

**SOLVENT**

**NS**

**DS**

**SWH**

**A Q**

**RG**

**DW**

**DE**

**TE**

**D1**

**======== CHANNEL f1 ========**

**SFO1**

**NUC1**

**P1**

**F2 - Processing parameters**

**SI**

**SF**

**WDW**

**LB**

**GB**

**PC**

**1D NMR plot parameters**

**CX**

**CY**

**F1P**

**F1**

**F2P**

**PPCM**

**HZCM**

**CS**

**CL**

**FL**

**F2**

**F1**

**F2N**

**HDCM**

**CS**

**CL**

**FL**

**F2**

**F1**

**F2N**

**HDCM**
$^1$H NMR 2D TOCSY of macrocycle 1b with presaturation suppression of the HOD peak
1 mM in D$_2$O at 600 MHz and 293 K with 150-ms spin-lock mixing time
$^1$H NMR 2D TOCSY of macrocycle 1b with presaturation suppression of the HOD peak
1 mM in D$_2$O at 600 MHz and 293 K with 150-ms spin-lock mixing time
$^1$H NMR 2D ROESY of macrocycle 1b with presaturation suppression of the HOD peak
1 mM in D$_2$O at 600 MHz and 293 K with 200-ms spin-lock mixing time
$^1$H NMR 2D ROESY of macrocycle 1b with presaturation suppression of the HOD peak
1 mM in D$_2$O at 600 MHz and 293 K with 200-ms spin-lock mixing time
\(^1\)H NMR DOSY of peptide 1b, 1 mM in D\(_2\)O at 500 MHz and 298 K monomer predominates

Calculations for peptide 1b at 1.0 mM

\[ D_{HOD} = 19.0 \times 10^{-10} \text{ m}^2/\text{s} \]

\[ \log (D_{HOD}) = -8.721 \]

\[ D_{\text{monomer}}: \log(D) = -9.712; D = 10^{-9.712} = 19.4 \pm 1.7 \times 10^{-11} \text{ m}^2/\text{s} \]

\(^a\)Longsworth, L. G. J. Phys. Chem. 1960, 64, 1914–1917.
**1H NMR of peptide 1b, 16 mM in D2O at 600 MHz and 293 K**

| ppm  | Integral 0.152 | 2.000 | 0.127 | 0.093 | 1.145 | 4.025 | 1.900 | 1.024 | 5.856 | 1.215 | 3.254 | 0.579 | 3.121 | 1.220 | 1.099 | 2.536 | 2.147 | 2.286 | 1.358 | 5.988 | 27.540 | 18.115 | 32.339 | 3.031 | 2.542 |
|------|---------------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|------|-------|

**Current Data Parameters**

| USER | mtmax | NAME | NT_iv_122 |
|------|-------|------|-----------|
| USER |       | NAME | NT_iv_122 |

**F2 - Acquisition Parameters**

| Date_ | 20160331 |
|-------|-----------|
| Time  | 20.34     |
| INSTRUM | av600   |
| PROBHD | 5 mm CPBBO BB- |
| PULPROG | zg30 |
| TD    | 97922     |
| SOLVENT | D2O |
| NS    | 32        |
| DS    | 2         |
| SWH   | 9615.385 Hz |
| FIDRES | 0.098194 Hz |
| A Q   | 5.0919938 sec |
| RG    | 9         |
| DW    | 52.000 usec |
| DE    | 13.70 usec |
| TE    | 293.0 K   |
| D1    | 0.10000000 sec |

**F2 - Processing parameters**

| SI    | 65536 |
| SF    | 600.1299883 MHz |
| WDW   | EM |
| SSB   | 0 |
| LB    | 1.00 Hz |
| GB    | 0 |
| PC    | 1.00 |

**1D NMR plot parameters**

| CX    | 22.40 cm |
| CY    | 15.00 cm |
| F1P   | 9.000 ppm |
| F1    | 5801.17 Hz |
| F2P   | -10.000 ppm |
| F2    | -40.01 Hz |
| HZCM  | 239,5239 Hz/cm |
| PMCM  | 0.3331 pm/cm |
| HNMR  | 239,5239 Hz/cm |

**1H NMR of peptide 1b, 16 mM in D2O at 600 MHz and 293 K**

![1H NMR spectrum of peptide 1b](image)
$^1$H NMR of peptide 1b, 16 mM in D$_2$O at 600 MHz and 293 K
$^{1}$H NMR of peptide 1b, 16 mM in D$_2$O at 600 MHz and 293 K
$^1$H NMR of peptide 1b, 16 mM in D$_2$O at 600 MHz and 293 K

**Current Data Parameters**
- USER: mtmax
- NAME: mt_iv_132
- SEQNO: 12
- PROCNO: 1

**F2 - Acquisition Parameters**
- Data: 20140116
- Time: 20.34
- INSTRUM: av600
- RUNNO: 5 mm SW60 0H-
- FIDRES: 0.9762
- SEQUENT: 320
- NS: 32
- DS: 2
- SW: 9615.385 Hz
- FIDRES: 0.098194 Hz
- A: 5.0919938 sec
- RG: 9
- DW: 52.000 usec
- DE: 293.0 K
- D1: 0.10000000 sec

**1D NMR plot parameters**
- CX: 22.80 cm
- CY: 15.00 cm
- F1X: 4.000 ppm
- P1: 200.0 usec
- F2X: 1.100 ppm
- P2: 120.0 usec
- FHCM: 0.0011 ppm/cm
- NHCM: 25.2713 Hz/cm
$^1$H NMR of peptide 1b, 16 mM in D$_2$O at 600 MHz and 293 K
$^1$H NMR 2D TOCSY of macrocycle 1b with presaturation suppression of the HOD peak
16 mM in D$_2$O at 600 MHz and 293 K with 150-ms spin-lock mixing time
$^1$H NMR 2D TOCSY of macrocycle 1b with presaturation suppression of the HOD peak
16 mM in D$_2$O at 600 MHz and 293 K with 150-ms spin-lock mixing time
$^1$H NMR 2D TOCSY of macrocycle 1b with presaturation suppression of the HOD peak
16 mM in D$_2$O at 600 MHz and 293 K with 150-ms spin-lock mixing time
$^1$H NMR 2D NOESY of macrocycle 1b with presaturation suppression of the HOD peak
16 mM in D$_2$O at 600 MHz and 293 K with 150-ms mixing time
\(^1\)H NMR 2D NOESY of macrocycle 1b with presaturation suppression of the HOD peak
16 mM in D\(_2\)O at 600 MHz and 293 K with 150-ms mixing time
$^1$H NMR 2D NOESY of macrocycle 1b with presaturation suppression of the HOD peak
16 mM in D$_2$O at 600 MHz and 293 K with 150-ms mixing time
$^{1}H$ NMR DOSY of peptide $1b$, 16 mM in D$_2$O at 500 MHz and 298 K
tetramer predominates

calculations for peptide $1b$ at 16.0 mM

$D_{HOD} = 19.0 \times 10^{-10}$ m$^2$/s $^a$

$\log (D_{HOD}) = -8.721$

$D_{tetramer}: \log(D) = -9.924; D = 10^{-9.924} = 11.9 \pm 1.1 \times 10^{-11}$ m$^2$/s

$^a$Longsworth, L. G. J. Phys. Chem. 1960, 64, 1914–1917.
$^{1}H$ NMR of Fmoc-$^{[15}N$]Phe-OH in CDCl$_3$ at 500 MHz and 298 K

Current Data Parameters

**USER**

**NAME**

**EXPNO**

**PROCNO**

**F2 - Acquisition Parameters**

**Date**

**Time**

**INSTRUM**

**PROBHD**

**PULPROG**

**TD**

**SOLVENT**

**NS**

**DS**

**SWH**

**FIDRES**

**AQ**

**RG**

**DW**

**DE**

**TE**

**D1**

**MCREST**

**MCWRK**

**======== CHANNEL f1 ========**

**NUC1**

**P1**

**PL1**

**SFO1**

**F2 - Processing parameters**

**SI**

**SF**

**WDW**

**SSB**

**LB**

**GB**

**PC**

**1D NMR plot parameters**

**CX**

**CY**

**FIFP**

**F1**

**F2**

**FINCH**

**HCH**

**H2O**

**XX**

**1H NMR of Fmoc-$^{[15}N$]Phe-OH in CDCl$_3$ at 500 MHz and 298 K**

**CHCl$_3$**

**H2O**
**1H NMR of Fmoc-[\textsuperscript{15}N]Phe-OH in CDCl\textsubscript{3} at 500 MHz and 298 K**

![NMR Spectrum](image_url)

**Current Data Parameters**
- **NAME**: Fmoc-[\textsuperscript{15}N]Phe-OH
- **INSTRUM**: cryo500
- **PROBHD**: 5 mm CPTCI 1H-
- **PULPROG**: SpinEchoPG30gp.prd
- **TD**: 65536
- **SOLVENT**: CDCl\textsubscript{3}
- **NS**: 1024
- **DS**: 16
- **SWH**: 30303.031 Hz
- **FIDRES**: 0.462388 Hz
- **AQ**: 1.0814105 sec
- **RG**: 7298.2
- **DW**: 16.500 usec
- **DE**: 6.00 usec
- **TE**: 298.0 K
- **D1**: 0.25000000 sec
- **D11**: 0.03000000 sec
- **D16**: 0.00020000 sec
- **D17**: 0.00019600 sec
- **MCWRK**: 0.01500000 sec
- **P2**: 33.10 usec

**F2 - Acquisition Parameters**
- **PL0**: 120.00 dB
- **PL1**: -1.00 dB
- **SFO1**: 125.7942548 MHz
- **SP1**: 2.70 dB
- **SP2**: 2.70 dB
- **SPNAM1**: Crp60,0.5,20.
- **SPNAM2**: Crp60comp.4
- **SPOFF1**: 0.00 Hz
- **SPOFF2**: 0.00 Hz

**F2 - Processing parameters**
- **SI**: 65536
- **SF**: 125.7803954 MHz
- **WDW**: EM
- **SSB**: 0
- **LB**: 3.00 Hz
- **GB**: 0
- **PC**: 1.00

**1D NMR plot parameters**
- **CX**: 22.80 cm
- **CY**: 25.00 cm
- **F1P**: 200.000 ppm
- **F1**: 25156.08 Hz
- **F2P**: 20.000 ppm
- **F2**: 2515.61 Hz
- **PPMCM**: 7.89474 ppm/cm
- **HZCM**: 993.00317 Hz/cm
$^{1}H$ NMR of Fmoc-[\textsuperscript{15}N]$\text{Gly-OH}$ in CDCl\textsubscript{3} at 500 MHz and 298 K

\begin{figure}
\centering
\includegraphics[width=\textwidth]{nmr_spectrum.png}
\caption{\textsuperscript{1}H NMR spectrum of Fmoc-[\textsuperscript{15}N]$\text{Gly-OH}$ in CDCl\textsubscript{3} at 500 MHz and 298 K.}
\end{figure}

Current Data Parameters
- USER: intax
- NAME: NT_iv_133_15N_gly
- EXPNO: 151
- PROCNO: 1

F2 - Acquisition Parameters
- Data: 20160525
- TIM: 23:03
- INSTRUM: cryo500
- PROBHD: 5 mm CPTCI 1H-
- PULPROG: zg30
- TD: 81728
- SOLVENT: CDCl\textsubscript{3}
- NS: 32
- DS: 2
- SWH: 8012.820 Hz
- FIDRES: 0.098043 Hz
- A Q: 5.0998774 sec
- RG: 5
- DW: 62.400 usec
- DE: 6.00 usec
- TE: 298.0 K
- D1: 0.10000000 sec
- MCREST: 0.00000000 sec
- MCWRK: 0.01500000 sec

F2 - Processing parameters
- SI: 65536
- SF: 500.2200313 MHz
- WDW: EM
- SSB: 0
- LB: 0.30 Hz
- PC: 4.00

1D NMR plot parameters:
- CX: 22.80 cm
- CY: 15.00 cm
- F1P: 9.000 ppm
- F1: 4001.98 Hz
- FFP: 0.000 ppm
- F2: 0.00 Hz
- PHCM: 0.39474 ppm/cm
- HCM: 197.45528 Hz/cm
13C NMR of Fmoc-[15N]Gly-OH in CDCl₃ at 500 MHz and 298 K

--- Chart with peaks and chemical structures ---

--- Table of NMR Data Parameters ---

| Parameter | Value |
|-----------|-------|
| USER      | ntruex |
| NAME      | NT_iv_133_15N_gly |
| EXPNO     | 152 |
| PROCNO    | 1 |

F2 - Acquisition Parameters

- Date: 20160525
- Time: 23:10
- INSTRUM: cryo500
- PROBHD: 5 mm CPTCI 1H-
- PULPROG: SpinEchogp30gp.prd
- TD: 65536
- SOLVENT: CDCl3
- NS: 1024
- DS: 16
- FIDRES: 0.462388 Hz
- A Q: 1.0814105 sec
- RG: 3251
- DW: 16.500 usec
- DE: 6.000 usec
- TE: 298.0 K
- d11: 0.03000000 sec
- d16: 0.00020000 sec
- d17: 0.00019600 sec
- D1: 0.25000000 sec
- D2: 0.01500000 sec
- P2: 33.10 usec

--- CHANNEL f1 ---

- NUC1: 13C
- P1: 16.55 usec
- P11: 500.00 usec
- P12: 2000.00 usec
- P13: 120.00 DB
- SFO1: 125.7942548 MHz
- SP1: 120.00 DB
- SP2: 2.70 dB
- SPNAM1: Crp60,0.5,20.1
- SPNAM2: Crp60comp.4
- SPOFF1: 0.00 Hz
- SPOFF2: 0.00 Hz

--- CHANNEL f2 ---

- CPDPRG2: waltz16
- NUC2: 1H
- PCPD2: 100.00 usec
- P2: 1.40 DB
- P12: 24.50 DB
- SFO2: 500.2225011 MHz

--- GRADIENT CHANNEL ---

- GPNAM1: SINE.100
- GPNAM2: SINE.100
- GPX1: 0.00 %
- GPX2: 0.00 %
- GPY1: 0.00 %
- GPY2: 50.00 %
- GPZ1: 30.00 %
- GPZ2: 0.00 %
- p15: 500.00 usec
- p16: 1000.00 usec

F2 - Processing parameters

- SI: 65536
- SF: 125.7803950 MHz
- WDW: EM
- SSB: 0
- LB: 2.00 Hz
- GB: 0
- PC: 1.00

--- 1D NMR plot parameters ---

- CX: 22.80 cm
- C1: 25.00 cm
- F1: 25156.08 Hz
- T1: 125.7942548 MHz
- PPMCM: 7.89474 ppm/cm
- HZCM: 993.00317 Hz/cm
$^1\text{H}, ^{15}\text{N}$ HSQC of peptide $[^{15}\text{N}]1\text{a}$ in 9:1 H$_2$O/D$_2$O at 600 MHz and 293 K
8.0 mM total concentration
$^1$H,$^{15}$N HSQC of peptide $[^{15}$N]1b in 9:1 H$_2$O/D$_2$O at 600 MHz and 293 K
8.0 mM total concentration