A facile one pot route for the synthesis of imide tethered peptidomimetics

Veladi Panduranga, Girish Prabhu, Roopesh Kumar, Basavaprabhu and Vommina V. Sureshbabu

Room No.109, Peptide Research Laboratory, Department of Studies in Chemistry, Central College Campus, Dr. B. R. Ambedkar Veedhi, Bangalore University, Bangalore 560 001, India.

E-mail: sureshbabuvommina@rediffmail.com, hariccb@gmail.com, hariccb@hotmail.com

General information

All chemicals were used as obtained from Sigma Aldrich Company, USA. All the solvents were dried and purified using recommended procedures in the literature whenever necessary. High resolution mass spectra were recorded on a Micromass Q-TOF micromass spectrometer and ESMS on LCQ Deca XP MAX using electron spray ionization mode. $^1$H NMR and $^{13}$C NMR spectra were recorded on a Bruker AMX 300 MHz and 100 MHz spectrometer, respectively, at the Indian Institute of Science, Bangalore. RP-HPLC analysis of epimers was carried out by LCQ Deca XP MAX VWD at $\lambda = 272$nm; flow rate: 1.0 mL/min; column: Thermoscientific C18 syncronis, pore size-5 $\mu$m, diameter x length = 4.6 x 250 nm; method: gradient 0.1% TFA water-acetonitrile; acetonitrile 30-100% in 30 min. Melting points were determined in an open capillary and are uncorrected. TLC experiments were done using MERCK TLC aluminum sheets (silica gel 60 F254) and chromatograms were visualized by exposing in iodine chamber or UV-lamp. Column chromatography was performed on silica gel (100-200 mesh) using ethyl acetate and hexane mixtures as eluent.
Racemization study of Fmoc-L-Phg-ψ[CONHCO]-Ala-Boc

Racemization study of Fmoc-D-Phg-ψ[CONHCO]-Ala-Boc

Racemization study of Fmoc-L-Phg-ψ[CONHCO]-Ala-Boc and Fmoc-D-Phg-ψ[CONHCO]-Ala-Boc
Racemization study of Fmoc-L-Phe-ψ[CONHCO]-Phe-Ala-Boc

Racemization study of Fmoc-D-Phe-ψ[CONHCO]-Phe-Ala-Boc
Racemization study of Fmoc-L-Phe-ψ(CONHCO)-Phe-Ala-Boc and Fmoc-L-Phe-ψ(CONHCO)-Phe-Ala-Boc

\( ^1H\) NMR Spectrum of Compound Fmoc-Ala-ψ(CONHCO)-Phe-Boc 4a

\( ^{13}C\) NMR Spectrum of Compound Fmoc-Ala-ψ(CONHCO)-Phe-Boc 4a
HRMS of Compound Fmoc-Ala-ψ[CONHCO]-Phe-Boc 4a

\[ \text{1H NMR Spectrum of Compound Fmoc-Phe-ψ[CONHCO]-Val-Boc 4b} \]
$^{13}$C NMR Spectrum of Compound Fmoc-Phe-$\psi$[CONHCO]-Val-Boc 4b

HRMS of Compound Fmoc-Phe-$\psi$[CONHCO]-Val-Boc 4b
$^1$H NMR Spectrum of Compound Fmoc-Phe-$\psi$[CONHCO]-Gly-Cbz 4c

$^{13}$C NMR Spectrum of Compound Fmoc-Phe-$\psi$[CONHCO]-Gly-Cbz 4c
HRMS of Compound Fmoc-Phe-ψ[CONHCO]-Gly-Cbz 4c

\[ \text{HRMS of Compound Fmoc-Phe-ψ[CONHCO]-Gly-Cbz 4c} \]

\[ \text{1H NMR Spectrum of Compound Fmoc-Phg-ψ[CONHCO]-Ala-Boc 4d} \]
$^{13}$C NMR Spectrum of Compound Fmoc-Phg-$\psi$[CONHCO]-Ala-Boc 4d

HRMS of Compound Fmoc-Phg-$\psi$[CONHCO]-Ala-Boc 4d
\textbf{1H NMR Spectrum of Compound Fmoc-Val-ψ[CONHCO]-Leu-Cbz 4e}

\textbf{13C NMR Spectrum of Compound Fmoc-Val-ψ[CONHCO]-Leu-Cbz 4e}
HRMS of Compound Fmoc-Val-ψ[CONHCO]-Leu-Cbz 4e

\[ \text{HRMS} \]

\[ \text{HRMS of Compound Fmoc-Val-ψ[CONHCO]-Leu-Cbz 4e} \]

\[ \text{'H NMR Spectrum of Compound Boc-Gly-ψ[CONHCO]-Val-Cbz 4f} \]

\[ \text{'H NMR Spectrum of Compound Boc-Gly-ψ[CONHCO]-Val-Cbz 4f} \]
$^{13}$C NMR Spectrum of Compound Boc-Gly-$\psi$[CONHCO]-Val-Cbz 4f

ESMS of Compound Boc-Gly-$\psi$[CONHCO]-Val-Cbz 4f
$^1$H NMR Spectrum of Compound Fmoc-Leu-$\psi$[CONHCO]-Ph 4g

$^{13}$C NMR Spectrum of Compound Fmoc-Leu-$\psi$[CONHCO]-Ph 4g
HRMS of Compound Fmoc-Leu-$\psi$[CONHCO]-Ph 4g

$^1$H NMR Spectrum of Compound Cbz-Ala-Phe-$\psi$[CONHCO]-Ala-Fmoc 8a
$^{13}$C NMR Spectrum of Compound Cbz-Ala-Phe-$\psi$[CONHCO]-Ala-Fmoc 8a

HRMS of Compound Cbz-Ala-Phe-$\psi$[CONHCO]-Ala-Fmoc 8a
$^{1}H$ NMR Spectrum of Compound Boc-Ala-Phe-$\psi$[CONHCO]-Ala-Fmoc 8b

$^{13}C$ NMR Spectrum of Compound Boc-Ala-Phe-$\psi$[CONHCO]-Ala-Fmoc 8b
ESMS of Compound Boc-Ala-Phe-ψ[CONHCO]-Ala-Fmoc 8b

1H NMR Spectrum of Compound Boc-Phe-Val-ψ[CONHCO]-lle-Fmoc 8c
$^{13}$C NMR Spectrum of Compound Boc-Phe-Val-$\psi$(CONHCO)-lle-Fmoc 8c

HRMS of Compound Boc-Phe-Val-$\psi$(CONHCO)-lle-Fmoc 8c
$^1$H NMR Spectrum of Compound Cbz-Ala-Leu-$\psi$[CONHCO]-Phe-Fmoc 8d

$^{13}$C NMR Spectrum of Compound Cbz-Ala-Leu-$\psi$[CONHCO]-Phe-Fmoc 8d
HRMS of Compound Cbz-Ala-Leu-ψ[CONHCO]-Phe-Fmoc 8d

\[ \text{\textsuperscript{1}H NMR Spectrum of Compound Boc-Ala-Leu-ψ[CONHCO]-Gly-Cbz 8e} \]
$^{13}$C NMR Spectrum of Compound Boc-Ala-Leu-$\psi$(CONHCO)-Gly-Cbz 8e

ESMS of Compound Boc-Ala-Leu-$\psi$(CONHCO)-Gly-Cbz 8e
$^1$H NMR Spectrum of Compound Boc-Ala-Phe-ψ[CONHCO]-Phe-Ala-Cbz 8f

$^{13}$C NMR Spectrum of Compound Boc-Ala-Phe-ψ[CONHCO]-Phe-Ala-Cbz 8f
HRMS of Compound Boc-Ala-Phe-ψ[CONHCO]-Phe-Ala-Cbz 8f

$^1$H NMR Spectrum of Compound (S)- tert-butyl(1-oxo-1-(2-oxo-2H-chromene-3-carboxamido)-3-phenylpropan-2-yl)carbamate 10a
\(^{13}\)C NMR Spectrum of Compound (S)-tert-butyl(1-oxo-1-(2-oxo-2H-chromene-3-carboxamido)-3-phenylpropan-2-yl)carbamate 10a

HRMS Spectrum of Compound (S)-tert-butyl(1-oxo-1-(2-oxo-2H-chromene-3-carboxamido)-3-phenylpropan-2-yl)carbamate 10a
\^{1}H NMR Spectrum of Compound (S)-(9H-fluoren-9-yl)methyl(1-oxo-1-(2-oxo-2H-chromene-3-carboxamido)propan-2-yl)carbamate 10b

\^{13}C NMR Spectrum of Compound (S)-(9H-fluoren-9-yl)methyl(1-oxo-1-(2-oxo-2H-chromene-3-carboxamido)propan-2-yl)carbamate 10b
HRMS Spectrum of Compound (S)-(9H-fluoren-9-yl)methyl(1-oxo-1-(2-oxo-2H-chromene-3-carboxamido)propan-2-yl)carbamate 10b

\[ \text{Mol. For} = \text{C}_{28}\text{H}_{22}\text{N}_{2}\text{O}_{6}\text{Na} \]
Calcld. Mass = 505.1376 (M+Na)

\[ \text{334.1143} \]
\[ \text{505.9920} \]
\[ \text{685.4827} \]
\[ \text{791.4966} \]
\[ \text{842.7174} \]

\[ \text{1H NMR Spectrum of Compound (S)-benzyl(4-methyl-1-oxo-1-(2-oxo-2H-chromene-3-carboxamido)pentan-2-yl)carbamate 10c} \]
$^{13}$C NMR Spectrum of Compound (S)-benzyl(4-methyl-1-oxo-1-(2-oxo-2H-chromene-3-carboxamido)pentan-2-yl)carbamate 10c

HRMS Spectrum of Compound (S)-benzyl(4-methyl-1-oxo-1-(2-oxo-2H-chromene-3-carboxamido)pentan-2-yl)carbamate 10c