Electronic Supporting Information

Ti-mediated Direct and Stereoselective Mannich Reactions between Esters and Oxime Ethers

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General

All reactions were carried out in oven-dried glassware under an argon atmosphere. Flash column chromatography was performed with silica gel Merck 60 (230-400 mesh ASTM). TLC analysis was performed on 0.25 mm Silicagel Merck 60 F254 plates. Melting points were determined on a hot stage microscope apparatus (Yanagimoto) and were uncorrected. NMR spectra were recorded on a JEOL DELTA 300 spectrometer, operating at 300 MHz for 1H NMR and 75 MHz for 13C NMR. Chemical shifts (δ ppm) in CDCl3 were reported downfield from TMS (= 0) for 1H NMR. For 13C NMR, chemical shifts were reported in the scale relative to CDCl3 (77.00 ppm) as an internal reference. IR Spectra were recorded on a JASCO FT/IR-5300 spectrophotometer. Mass spectra were measured on a JEOL JMS-T100LC spectrometer.
Spectra data of new compounds 1-11 (Table 1)

Phenyl syn-2-butyl-3-(methoxyamino)decanoate (1)

\( \delta \) 0.88 (3H, t, \( J = 6.9 \) Hz), 0.93 (3H, t, \( J = 6.9 \) Hz), 1.19-1.68 (17H, m), 1.76-1.92 (1H, m), 2.76-2.85 (anti, 0.10H, m), 2.86-2.96 (syn, 0.90H, m), 3.12-3.26 (1H, m), 3.53 (anti, 0.30H, s), 3.54 (syn, 2.70H, s), 7.03-7.11 (2H, m), 7.18-7.26 (1H, m), 7.33-7.42 (2H, m); \(^{13}\)C NMR (75 MHz, CDCl\(_3\)): \( \delta \) 13.9, 14.0, 22.5, 22.6, 26.8, 27.7, 29.1, 29.6, 30.2, 31.5, 31.7, 47.4, 61.9, 62.7, 121.5, 125.6, 129.3, 150.8, 173.3; IR (neat) 2928, 2857, 1755, 1493, 1468, 1196 cm\(^{-1}\); HRMS (ESI) calcd for C\(_{21}\)H\(_{35}\)N\(_1\)O\(_3\) (M + Na\(^{+}\)) 372.2515, found 372.2513.

4-Nitrophenyl syn-2-butyl-3-(methoxyamino)decanoate (2)

\( \delta \) 0.88 (3H, t, \( J = 6.9 \) Hz), 0.94 (3H, t, \( J = 6.9 \) Hz), 1.18-1.67 (18H, m), 1.79-1.94 (1H, m), 2.85-2.96 (1H, m), 3.24-3.37 (1H, m), 3.53 (anti, 0.18H, s), 3.56 (syn, 2.82H, s), 7.23-7.32 (2H, m), 8.23-8.32 (2H, m); \(^{13}\)C NMR (75 MHz, CDCl\(_3\)): \( \delta \) 13.9, 14.0, 22.6, 22.6, 26.8, 26.9, 29.1, 29.5, 30.3, 31.7, 47.6, 61.9, 62.7, 122.4, 125.1, 145.2, 155.7, 172.6; IR (neat) 2930, 2859, 1765, 1526, 1346, 1208, 1105 cm\(^{-1}\); HRMS (ESI) calcd for C\(_{24}\)H\(_{38}\)N\(_2\)O\(_5\) (M + Na\(^{+}\)) 417.2365, found 417.2369.

4-Nitrophenyl syn-3-(methoxyamino)-3-phenylpropylhexanoate (3)

\( \delta \) 0.91 (3H, t, \( J = 7.2 \) Hz), 1.21-1.48 (4H, m), 1.50-1.64 (1H, m), 1.75-1.93 (3H, m), 2.59-2.78 (1H, m), 2.81-2.99 (2H, m), 3.19-3.33 (1H, m), 3.53 (anti, 0.15H, s), 3.54 (syn, 2.95H, s), 7.16-7.34 (7H, m), 8.22-8.29 (2H, m); \(^{13}\)C NMR (75 MHz, CDCl\(_3\)): \( \delta \) 13.8, 22.5, 27.2, 30.0, 30.8, 32.9, 47.6, 61.7, 62.0, 122.4, 125.1, 126.0, 128.3, 128.4, 141.3, 145.1, 155.5, 172.3; IR (neat) 2934, 1763, 1525, 1346, 1209, 1111 cm\(^{-1}\); HRMS (ESI) calcd for C\(_{25}\)H\(_{38}\)N\(_2\)O\(_5\) (M + Na\(^{+}\)) 423.1896, found 423.1891.

4-Nitrophenyl syn-2-butyl-3-(methoxyamino)trideca-12-enoate (4)

\( \delta \) 0.92 (3H, t, \( J = 6.9 \) Hz), 1.19-1.65 (19H, m), 1.77-1.93 (1H, m), 1.98-2.10 (2H, m), 2.74-2.84 (anti, 0.04H, m), 2.85-2.93 (syn, 0.96H, m), 3.15-3.23 (anti, 0.04H, m), 3.23-3.33 (syn, 0.96H, m), 3.50 (anti, 0.12H, s), 3.52 (syn, 2.88H, s), 7.23-7.31 (2H, m), 8.23-8.30 (2H, m); \(^{13}\)C NMR (75 MHz, CDCl\(_3\)): \( \delta \) 13.9, 22.6, 26.8, 28.8, 29.0, 29.3, 29.5, 30.2, 33.7, 47.6, 61.9, 62.6, 114.1, 122.4, 125.1, 139.0, 145.1, 155.7, 172.5; IR (neat) 2928, 2857, 1765, 1526, 1346, 1209, 1101 cm\(^{-1}\); HRMS (ESI) calcd for C\(_{22}\)H\(_{38}\)N\(_2\)O\(_5\) (M + Na\(^{+}\)) 457.2678, found 457.2670.

4-Nitrophenyl syn-2-(cyclohexyl(methoxyamino)methyl)hexanoate (5)

\( \delta \) 0.94 (3H, t, \( J = 7.2 \) Hz), 1.02-2.03 (17H, m), 2.74-2.84 (1H, m), 3.06 (anti, 0.26H, dd, \( J = 4.5 \) Hz, 7.9 Hz), 3.27 (syn, 0.74H, dd, \( J = 4.1 \) Hz, 7.9 Hz), 3.44 (anti, 0.78H, s), 3.45 (syn, 2.22H, s), 7.23-7.31 (2H, m), 8.22-8.31 (2H, m); \(^{13}\)C NMR (75 MHz, CDCl\(_3\)): \( \delta \) 14.0, 22.6, 22.8, 25.1, 26.1, 26.2, 26.3, 26.4, 26.5, 26.7, 27.6, 29.3, 29.8, 30.1, 30.4, 30.7, 31.0, 37.7, 38.4, 45.9, 47.1, 61.4, 66.8, 67.2, 122.4, 125.1, 145.1, 155.8, 156.0, 173.0; IR (neat) 2930, 2857, 1765, 1526, 1346, 1209 cm\(^{-1}\); HRMS (ESI) calcd for C\(_{20}\)H\(_{26}\)N\(_2\)O\(_5\) (M + Na\(^{+}\)) 401.2052, found 401.2046.

4-Nitrophenyl syn-3-(1-methoxyamino-1-phenyl)methylhexanoate (6)

\( \delta \) 0.92 (3H, t, \( J = 6.5 \) Hz), 1.29-1.51 (4H, m), 1.79-2.00 (2H, m), 3.06-3.19 (1H, m), 3.50...
(3H, s), 4.27 (1H, d, $J = 8.3$ Hz), 6.74-6.82 (2H, m), 7.29-7.43 (5H, m), 8.12-8.19 (2H, m); $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta$ 13.8, 22.4, 28.8, 29.8, 49.6, 62.0, 66.8, 122.3, 124.9, 128.0, 128.4, 139.2, 145.2, 155.1, 171.5; IR (neat) 2957, 2934, 2872, 1763, 1762, 1526, 1346, 1208, 1109 cm$^{-1}$; HRMS (ESI) calcd for C$_{20}$H$_{24}$N$_2$O$_5$ (M + Na$^+$) 395.1583, found 395.1587.

4-Nitrophenyl syn-2-[(methoxyamino)(phenyl)methyl]hexanoate (7) 

(syn / anti = >99 / 1). Colorless oil; $^1$H NMR (300 MHz, CDCl$_3$): $\delta$ 0.92 (3H, t, $J = 7.2$ Hz), 1.31-1.51 (4H, m), 1.80-1.94 (2H, m), 3.19 (1H, q, $J = 7.2$ Hz), 3.53 (3H, s), 4.42 (1H, d, $J = 7.9$ Hz), 5.94-6.03 (1H, brs), 6.30-6.41 (2H, m), 7.05-7.13 (2H, m), 7.39-7.44 (1H, m), 8.20-8.28 (2H, m); $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta$ 13.8, 22.4, 28.3, 29.6, 47.2, 60.1, 62.0, 108.4, 110.3, 122.3, 125.0, 142.0, 145.2, 155.1, 171.3; IR (neat) 2959, 2936, 1763, 1528, 1348, 1208 cm$^{-1}$; HRMS (ESI) calcd for C$_{18}$H$_{22}$N$_2$O$_6$ (M + Na$^+$) 385.1376, found 385.1384.

4-Nitrophenyl syn-2-methyl-3-(methoxyamino)decanoate (8) 

(syn / anti = 90 / 10). Colorless oil; $^1$H NMR (300 MHz, CDCl$_3$): $\delta$ 0.89 (3H, t, $J = 6.6$ Hz), 1.17-1.52 (12H, m), 2.82-3.04 (1H, m), 3.47 (syn, 2.70H, s), 3.51 (anti, 0.30H, s), 5.56-5.70 (1H, brs), 7.22-7.31 (2H, m), 8.21-8.31 (2H, m); $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta$ 9.8, 14.0, 22.6, 26.7, 29.1, 29.4, 29.5, 31.7, 61.7, 62.0, 122.4, 125.0, 145.1, 155.9, 173.0; IR (neat) 2930, 1765, 1593, 1526, 1348 cm$^{-1}$; HRMS (ESI) calcd for C$_{18}$H$_{28}$N$_2$O$_5$ (M + Na$^+$) 374.4927, found 374.4922.

4-Nitrophenyl 2syn-(but-3-enyl)-3-(methoxyamino)decanoate (9) 

(syn / anti = 95 / 5). Pale yellow oil; $^1$H NMR (300 MHz, CDCl$_3$): $\delta$ 0.89 (3H, t, $J = 7.2$ Hz), 1.19-1.69 (12H, m), 2.35-2.67 (2H, m), 2.89-2.97 (anti, 0.05H, m), 2.98-3.06 (syn, 0.95H, m), 3.19-3.26 (anti, 0.05H, m), 3.26-3.36 (syn, 0.95H, m), 3.51 (anti, 0.15H, s), 3.52 (syn, 2.85H, s), 5.07-5.22 (2H, m), 5.90 (1H, ddt, $J = 6.9$, 10.3, 16.9 Hz), 7.21-7.29 (2H, m), 8.23-8.30 (2H, m); $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta$ 14.0, 22.5, 26.7, 29.1, 29.5, 31.7, 47.1, 61.9, 62.1, 117.0, 122.4, 125.0, 135.5, 145.2, 155.6, 171.8; IR (neat) 2930, 2857, 1765, 1526, 1348, 1209, 1111 cm$^{-1}$; HRMS (ESI) calcd for C$_{20}$H$_{30}$N$_2$O$_5$ (M + Na$^+$) 401.2052, found 401.2044.

4-Nitrophenyl syn-3-(methoxyamino)-2-phenethyldecanoate (10) 

(syn / anti = 90 / 10). Colorless oil; $^1$H NMR (300 MHz, CDCl$_3$): $\delta$ 0.89 (3H, t, $J = 6.9$ Hz), 1.17-1.71 (12H, m), 2.90-2.98 (anti, 0.20H, m), 3.02-3.14 (syn, 1.80H, m), 3.23-3.41 (2H, m), 3.55 (anti, 0.30H, s), 6.87-7.02 (2H, m), 7.15-7.38 (5H, m), 8.13-8.28 (2H, m); $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta$ 0.89 (3H, t, $J = 6.9$ Hz), 1.17-1.71 (12H, m), 2.90-2.98 (anti, 0.20H, m), 3.02-3.14 (syn, 1.80H, m), 3.23-3.41 (2H, m), 3.55 (anti, 0.30H, s), 6.87-7.02 (2H, m), 7.15-7.38 (5H, m), 8.13-8.28 (2H, m); $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta$ 14.0, 22.5, 26.7, 29.1, 29.5, 31.7, 47.1, 61.9, 62.1, 117.0, 122.4, 125.0, 135.5, 145.2, 155.6, 171.8; IR (neat) 2930, 2857, 1765, 1526, 1348, 1209, 1111 cm$^{-1}$; HRMS (ESI) calcd for C$_{24}$H$_{32}$N$_2$O$_5$ (M + Na$^+$) 451.2209, found 451.2205.

4-Nitrophenyl syn-3-(methoxyamino)-2-chloropropyldecanoate (11) 

(syn / anti = 97 / 3). Colorless oil; $^1$H NMR (300 MHz, CDCl$_3$): $\delta$ 0.89 (3H, t, $J = 6.9$ Hz), 1.17-1.57 (12H, m), 1.72-2.09 (4H, m), 2.80-2.93 (1H, m), 3.58-3.70 (2H, m), 3.48 (anti, 0.09H, s), 5.07 (syn, 2.91H, s), 7.22-7.31 (2H, m), 8.24-8.30 (2H, m); $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta$ 14.0, 22.5, 26.7, 29.1, 29.4, 30.9, 31.6, 44.6, 46.8, 61.8, 62.4, 122.4, 145.1, 155.5, 172.0; IR (neat) 2930, 2857, 1765, 1526, 1346 cm$^{-1}$; HRMS (ESI) calcd for C$_{26}$H$_{31}$ClN$_2$O$_5$ (M + Na$^+$) 436.4850, found 436.4856.
Spectra data of new compounds 12-19 (Table 2)

Methyl syn-2-methoxy-3-methoxyaminodecanoate (12)

(syn / anti = 2 / 98). Colorless oil; 1H NMR (300 MHz, CDCl3): δ 0.87 (3H, t, J = 6.9 Hz), 1.14-1.50 (12H, m), 3.17-3.28 (1H, m), 3.46 (3H, s), 3.53 (3H, s), 3.77 (3H, s), 4.17 (anti, 0.98H, d, J = 3.4 Hz), 4.31 (anti, 0.02H, d, J = 7.6 Hz); 13C NMR (75 MHz, CDCl3): δ 13.9, 22.5, 26.5, 28.9, 29.3, 31.6, 51.5, 59.0, 61.3, 61.8, 79.0, 172.3; IR (neat) 2930, 2857, 1753, 1466, 1437, 1269, 1200, 1142 cm⁻¹; HRMS (ESI) calcd for C19H31N1O6S1 (M + Na⁺) 424.1770, found 424.1766.

Methyl syn-2-benzyloxy-3-methoxyaminodecanoate (13)

(syn / anti = 1 / >99). Colorless oil; 1H NMR (300 MHz, CDCl3): δ 0.87 (3H, t, J = 7.9 Hz), 1.11-1.45 (12H, m), 3.13-3.27 (1H, m), 3.44 (3H, s), 3.74 (3H, s), 4.23 (1H, d, J = 3.4 Hz), 4.68 (1H, d, J = 12.4 Hz), 4.71 (1H, d, J = 12.4 Hz), 7.26-7.38 (5H, m); 13C NMR (75 MHz, CDCl3): δ 14.1, 22.6, 26.6, 29.0, 29.4, 31.7, 51.7, 59.2, 62.3, 75.9, 79.2, 127.7, 128.2, 128.5, 137.9, 172.5; IR (neat) 2930, 2857, 1753, 1466, 1437, 1269, 1200, 1142 cm⁻¹; HRMS (ESI) calcd for C19H31N1O4 (M + Na⁺) 350.2307, found 350.2306.

Methyl syn-2-benzyloxy-3-methoxyaminodecanoate (14)

(syn / anti = 4 / 96). Colorless oil; 1H NMR (300 MHz, CDCl3): δ 0.87 (3H, t, J = 7.2 Hz), 1.14-1.48 (12H, m), 3.20-3.32 (1H, m), 3.47 (3H, s), 3.76 (3H, s), 4.39 (1H, d, J = 3.8 Hz), 4.43 (anti, 0.96H, d, J = 11.4 Hz), 4.79 (anti, 0.96H, d, J = 11.4 Hz), 5.14 (syn, 0.04H, d, J = 12.4 Hz), 5.27 (syn, 0.04H, d, J = 12.4 Hz), 7.23-7.40 (5H, m); 13C NMR (75 MHz, CDCl3): δ 14.0, 22.6, 26.6, 26.8, 29.0, 29.4, 31.7, 51.7, 61.3, 62.0, 73.1, 77.1, 127.8, 127.9, 128.3, 137.5, 127.6; IR (neat) 2924, 2859, 1752, 1456, 1138 cm⁻¹; HRMS (ESI) calcd for C19H31N1O4 (M + Na⁺) 350.2307, found 350.2306.

Methyl 3-methoxymino-2-tosyloxydecanoate (15)

(syn / anti = 9 / 91). Colorless oil; 1H NMR (300 MHz, CDCl3): δ 0.88 (3H, t, J = 6.9 Hz), 1.15-1.47 (12H, m), 2.45 (3H, s), 3.36 (anti, 2.67H, s), 3.38-3.45 (1H, m), 3.42 (syn, 0.33H, s), 3.64 (anti, 2.72H, s), 3.69 (syn, 0.28H, s), 4.95 (anti, 0.91H, d, J = 3.1 Hz), 5.34 (syn, 0.09H, d, J = 3.4 Hz), 7.31-7.37 (2H, m), 7.80-7.88 (2H, m); 13C NMR (75 MHz, CDCl3): δ 14.0, 21.6, 22.6, 26.2, 27.9, 29.0, 29.3, 31.7, 52.3, 61.4, 61.5, 76.3, 128.1, 129.6, 133.2, 145.1, 168.7; IR (neat) 2928, 2859, 1767, 1375, 1179 cm⁻¹; HRMS (ESI) calcd for C19H31N1O4S1 (M + Na⁺) 424.1770, found 424.1766.

Methyl 2-allyloxy-3-methoxyaminophenylpentanoate (16)

(syn / anti = 1 / >99). Colorless oil; 1H NMR (300 MHz, CDCl3): δ 1.54-1.75 (2H, m), 2.54-2.69 (1H, m), 2.75-2.89 (1H, m), 3.21-3.30 (1H, m), 3.53 (3H, s), 3.66 (3H, s), 3.89-4.00 (1H, m), 4.18-4.27 (1H, m), 4.32 (1H, d, J = 3.4 Hz), 5.15-5.33 (2H, m), 5.92 (1H, ddt, J = 6.2, 10.3, 17.2 Hz), 7.13-7.31 (5H, m); 13C NMR (75 MHz, CDCl3): δ 28.4, 32.5, 51.7, 60.9, 61.5, 72.2, 76.6, 117.7, 125.8, 128.2, 128.4, 134.0, 141.8, 172.4; IR (neat) 3268, 2949, 1752, 1454, 1435, 1269, 1208, 1138 cm⁻¹; HRMS (ESI) calcd for C19H33N1O4 (M + Na⁺) 350.2307, found 350.2306.

Methyl 2-allyloxy-3-methoxyaminotridec-12-enolate (17)

(syn / anti = 1 / >99). Colorless oil; 1H NMR (300 MHz, CDCl3): δ 1.16-1.50 (14H, m), 1.97-2.09 (2H, m), 3.18-3.31 (1H, m), 3.52 (3H, s), 3.76 (3H, s), 3.91-4.01 (1H, m), 4.19-4.28 (1H, m), 4.33 (1H, d, J = 3.4 Hz), 4.88-5.04 (2H, m), 5.16-5.33 (2H, m), 5.81 (1H, ddt, J = 6.5, 10.3, 17.2 Hz), 5.93 (1H, ddt, J = 6.2, 10.3, 16.9 Hz); 13C NMR (75 MHz, CDCl3): δ 26.6, 26.7, 28.8, 29.0, 29.2, 29.3, 29.4, 33.7, 51.7, 61.3, 61.9, 72.2, 76.6, 114.0, 117.7, 134.0, 139.1, 172.6; IR (neat) 2928, 2857, 1753, 1460, 1437, 1265, 1206, 1140 cm⁻¹; HRMS (ESI) calcd for C19H33N1O4 (M + Na⁺) 350.2307, found 350.2306.
Methyl 2-allyloxy-3-methoxyaminodecanoate (18)

(syn/anti = 1/99). Colorless oil; $^1$H NMR (300 MHz, CDCl$_3$): $\delta$ 0.87 (3H, t, $J = 6.9$ Hz), 1.16-1.49 (12H, m), 3.19-3.31 (1H, m), 3.52 (3H, s), 3.75 (3H, s), 3.91-4.01 (1H, m), 4.19-4.28 (1H, m), 4.33 (1H, d, $J = 3.1$ Hz), 5.14-5.34 (2H, m), 5.93 (1H, ddt, $J = 5.9, 10.3, 17.2$ Hz); $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta$ 14.0, 22.6, 26.6, 26.7, 29.0, 29.4, 31.7, 51.7, 61.3, 61.9, 72.2, 76.6, 117.7, 134.0, 172.6; IR (neat) 2953, 2855, 1753, 1462, 1453, 1267, 1204, 1140 cm$^{-1}$; HRMS (ESI) calcd for C$_{15}$H$_{29}$N$_1$O$_4$ (M + Na$^+$) 310.1994, found 310.1998.

Methyl 3-cyclohexyl-2-allyloxy-3-methoxyamino-propanoate (19)

(syn/anti = 1/99). Colorless oil; $^1$H NMR (300 MHz, CDCl$_3$): $\delta$ 0.85-1.34 (5H, m), 1.42-1.56 (1H, m), 1.57-1.80 (4H, m), 1.87-2.00 (1H, m), 3.10 (1H, dd, $J = 4.8, 5.49$ Hz), 3.47 (3H, s), 3.75 (3H, s), 3.88-3.98 (1H, m), 4.20-4.29 (1H, m), 4.30 (1H, d, $J = 4.1$ Hz), 5.14-5.33 (2H, m), 5.92 (1H, ddt, $J = 5.9, 10.3, 16.9$ Hz); $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta$ 26.3, 26.5, 28.9, 30.6, 37.3, 51.6, 61.0, 66.5, 72.2, 76.6, 117.5, 134.1, 173.0; IR (neat) 2928, 2855, 1752, 1451, 1263, 1209, 1150, 1105, 1022 cm$^{-1}$; HRMS (ESI) calcd for C$_{14}$H$_{25}$N$_1$O$_4$ (M + Na$^+$) 294.1681, found 294.1685.
Spectra data of new compounds 20-37 (Table 3)

**Methyl 3-(methoxyamino)-2,2-dimethyldecanoate (20)**

Colorless oil; $^1$H NMR (300 MHz, CDCl$_3$) $\delta$ 0.88 (3H, t, $J = 6.5$ Hz), 1.06-1.42 (11H, m), 1.15 (6H, s), 1.44-1.55 (1H, m), 3.11 (1H, dd, $J = 2.4$ Hz, $J = 10.0$ Hz), 3.41 (3H, s), 3.65 (3H, s), 5.56 (1H, br s); $^{13}$C NMR (75 MHz, CDCl$_3$) $\delta$ 14.1, 20.2, 22.6, 23.2, 27.6, 28.0, 29.2, 29.7, 31.8, 45.5, 51.5, 61.3, 66.2, 178.1; IR (neat) 2928, 2857, 1736, 1466, 1435, 1263, 1192, 1142; HRMS (ESI) calcd for C$_{14}$H$_{29}$N$_1$O$_3$ (M + Na$^+$) 282.2045, found 282.2040.

**Methyl 3-(methoxyamino)-2,2-dimethyltridec-12-enoate (21)**

Colorless oil; $^1$H NMR (300 MHz, CDCl$_3$) $\delta$ 1.08-1.56 (14H, m), 1.15 (6H, s), 1.97-2.09 (2H, m), 3.11 (1H, dd, $J = 2.1$ Hz, $J = 9.6$ Hz), 3.41 (3H, s), 3.65 (3H, s), 4.86-5.04 (2H, m), 5.56 (1H, br s), 5.81 (1H, ddt, $J = 6.5$ Hz, $J = 10.5$ Hz, $J = 17.1$ Hz); $^{13}$C NMR (75 MHz, CDCl$_3$) $\delta$ 20.2, 23.2, 27.6, 28.0, 28.9, 29.1, 29.4, 29.7, 45.5, 51.6, 61.3, 66.2, 114.1, 139.2, 178.2; IR (neat) 2930, 2855, 1736, 1466, 1435, 1262, 1192, 1138; HRMS (ESI) calcd for C$_{17}$H$_{33}$N$_1$O$_3$ (M + Na$^+$) 322.2358, found 322.2366.

**Methyl 3-(methoxyamino)-2,2-dimethyl-5-phenylpentanoate (22)**

Pale yellow oil; $^1$H NMR (300 MHz, CDCl$_3$) $\delta$ 1.148 (3H, s), 1.150 (3H, s), 1.47-1.62 (1H, m), 1.65-1.78 (1H, m), 2.65 (1H, ddd, $J = 6.5$ Hz, $J = 9.6$ Hz, $J_{gem} = 13.8$ Hz), 2.89 (1H, ddd, $J = 5.2$ Hz, $J = 10.0$ Hz, $J_{gem} = 13.8$ Hz), 3.16 (1H, dd, $J = 2.1$ Hz, $J = 10.0$ Hz), 3.44 (3H, s), 3.63 (3H, s), 5.66 (1H, br s), 7.15-7.34 (5H, m); $^{13}$C NMR (75 MHz, CDCl$_3$) $\delta$ 20.5, 23.2, 30.4, 33.9, 45.7, 51.6, 61.4, 65.6, 125.9, 128.4, 140.0, 177.9; IR (neat) 2978, 2948, 1732, 1456, 1435, 1269, 1192, 1134; HRMS (ESI) calcd for C$_{15}$H$_{23}$N$_1$O$_3$ (M + Na$^+$) 288.1576, found 288.1576.

**Methyl 3-(methoxyamino)-2,2,5-trimethylhexanoate (23)**

Yellow oil; $^1$H NMR (300 MHz, CDCl$_3$) $\delta$ 0.925 (3H, d, $J = 6.5$ Hz), 0.927 (3H, d, $J = 6.5$ Hz), 1.02-1.19 (2H, m), 1.14 (3H, s), 1.15 (3H, s), 1.65-1.82 (1H, m), 3.23 (1H, ddd, $J = 3.4$ Hz, $J = 5.2$ Hz, $J = 10.0$ Hz), 3.41 (3H, s), 3.66 (3H, s), 5.53 (1H, br s); $^{13}$C NMR (75 MHz, CDCl$_3$) $\delta$ 20.0, 21.6, 23.0, 23.9, 25.6, 37.3, 45.4, 51.5, 61.2, 63.9, 178.2; IR (neat) 2955, 2872, 1734, 1468, 1435, 1262, 1192, 1140; HRMS (ESI) calcd for C$_{11}$H$_{23}$N$_1$O$_3$ (M + Na$^+$) 240.1576, found 240.1574.

**Methyl 3-(methoxyamino)-2,2-dimethyl-5-phenylhexanoate (24)**

Pale yellow oil; $^1$H NMR (300 MHz, CDCl$_3$) $\delta$ 1.04 (2.13H, s), 1.09 (2.13H, s), 1.15 (0.87H, s), 1.17 (0.87H, s), 1.26 (2.13H, d, $J = 6.9$ Hz, $J_{gem} = 13.8$ Hz), 1.29 (0.87H, d, $J = 6.9$ Hz) 1.42-1.59 (0.71H and 0.58H , m), 1.65 (0.71H, ddd, $J = 2.1$ Hz, $J = 10.7$ Hz, $J_{gem} = 14.5$ Hz), 2.81 (0.71H, dd, $J = 2.1$ Hz, $J = 10.7$ Hz), 2.90-3.06 (1H, m) 3.29 (0.29H, dd, $J = 3.1$ Hz, $J = 9.3$ Hz), 3.34 (0.87H, s), 3.40 (2.13H, s), 3.54 (2.13H, s), 3.66 (0.87H, s), 7.15-7.35 (5H, m); $^{13}$C NMR (75 MHz, CDCl$_3$) $\delta$ 20.3, 20.7, 21.1, 22.9, 23.1, 23.7, 36.7, 37.2, 37.4, 37.5, 45.5, 45.6, 51.5, 51.6, 61.2, 61.3, 63.6, 64.2, 126.1, 126.8, 127.2, 128.4, 128.5, 146.3, 147.7, 177.8, 178.0; IR (neat) 2951, 1732, 1454, 1435, 1260, 1192, 1138; HRMS (ESI) calcd for C$_{16}$H$_{25}$N$_1$O$_3$ (M + Na$^+$) 302.1732, found 302.1729.

**Methyl 7-benzyloxy-3-(methoxyamino)-2,2-dimethylheptanoate (25)**

Pale yellow oil; $^1$H NMR (300 MHz, CDCl$_3$) $\delta$ 0.925 (3H, d, $J = 6.5$ Hz), 0.927 (3H, d, $J = 6.5$ Hz), 1.02-1.19 (2H, m), 1.14 (3H, s), 1.15 (3H, s), 1.65-1.82 (1H, m), 3.23 (1H, ddd, $J = 3.4$ Hz, $J = 8.9$ Hz), 3.41 (3H, s), 3.66 (3H, s), 5.53 (1H, br s); $^{13}$C NMR (75 MHz, CDCl$_3$) $\delta$ 20.2, 21.6, 23.0, 23.9, 25.6, 37.3, 45.4, 51.5, 61.2, 63.9, 178.2; IR (neat) 2955, 2872, 1734, 1468, 1435, 1262, 1192, 1140; HRMS (ESI) calcd for C$_{18}$H$_{29}$N$_1$O$_4$ (M + Na$^+$) 346.1994, found 346.1992.
Methyl 3-(methoxyamino)-2,2-dimethyl-3-phenylpropanoate (26)
Pale yellow oil; $^1H$ NMR (300 MHz, CDCl$_3$) $\delta$ 1.03 (3H, s), 1.16 (3H, s), 3.43 (3H, s), 3.70 (3H, s), 4.38 (1H, s), 5.99 (1H, br s), 7.25-7.35 (5H, m); $^{13}$C NMR (75 MHz, CDCl$_3$) $\delta$ 19.0, 24.7, 45.4, 51.8, 61.8, 70.3, 127.7, 127.9, 128.7, 137.9, 177.4; IR (neat) 2980, 2949, 1734, 1468, 1252, 1192, 1136, 1057; HRMS (ESI) calcd for C$_{13}$H$_{19}$N$_1$O$_3$ (M + Na$^+$) 260.1263, found 260.1260.

Methyl 3-(methoxyamino)-2,2-dimethyl-3-(2-naphthyl)propanoate (27)
Yellow oil; $^1H$ NMR (300 MHz, CDCl$_3$) $\delta$ 1.08 (3H, s), 1.21 (3H, s), 3.45 (3H, s), 3.72 (3H, s), 4.55 (1H, s), 6.12 (1H, br s), 7.42-7.51 (3H, m) 7.73-7.86 (4H, m); $^{13}$C NMR (75 MHz, CDCl$_3$) $\delta$ 19.3, 24.8, 45.7, 51.9, 61.9, 70.4, 125.9, 126.0, 126.7, 127.4, 127.6, 128.0, 132.9, 133.0, 135.5, 177.4; IR (neat) 2980, 2948, 1734, 1468, 1258, 1192, 1134, 1053; HRMS (ESI) calcd for C$_{17}$H$_{21}$N$_1$O$_3$ (M + Na$^+$) 310.1419, found 310.1422.

Methyl 3-(3-chlorophenyl)-3-(methoxyamino)-2,2-dimethylpropanoate (28)
Pale yellow oil; $^1H$ NMR (300 MHz, CDCl$_3$) $\delta$ 1.05 (3H, s), 1.15 (3H, s), 3.43 (3H, s), 3.71 (3H, s), 4.33 (1H, s), 6.02 (1H, brs), 7.16-7.34 (4H, m); $^{13}$C NMR (75 MHz, CDCl$_3$) $\delta$ 19.4, 24.5, 45.4, 52.0, 61.9, 69.9, 127.0, 127.9, 128.8, 129.1, 133.9, 140.3, 177.0; IR (neat) 2982, 2949, 1734, 1470, 1433, 1269, 1250, 129.1, 133.9, 140.3, 177.0; HRMS (ESI) calcd for C$_{13}$H$_{18}$Cl$_1$N$_1$O$_3$ (M + Na$^+$) 294.0873, found 294.0871.

Methyl 3-(4-trifluorophenyl)-3-(methoxyamino)-2,2-dimethylpropanoate (29)
Pale yellow oil; $^1H$ NMR (300 MHz, CDCl$_3$) $\delta$ 1.05 (3H, s), 1.15 (3H, s), 3.42 (3H, s), 3.71 (3H, s), 4.41 (1H, s), 6.09 (1H, br s), 7.45 (2H, d, $J$ = 8.3 Hz), 7.59 (2H, d, $J$ = 8.3 Hz); $^{13}$C NMR (75 MHz, CDCl$_3$) $\delta$ 19.4, 24.5, 45.4, 52.0, 61.9, 69.9, 124.1 [d, $^{1J}$ (13C, 19F) = 271 Hz], 124.8 [q, $^{3J}$ (13C, 19F) = 3 Hz], 129.1, 129.9 [q, $^{3J}$ (13C, 19F) = 32 Hz]; IR (neat) 2984, 2951, 1732, 1468, 1327, 1258, 1165, 1127, 1069, 1019; HRMS (ESI) calcd for C$_{14}$H$_{18}$F$_3$N$_1$O$_3$ (M + Na$^+$) 328.1136, found 328.1132.

Methyl 3-(methoxyamino)-2,2-dimethyl-3-(2-thiophenyl)propanoate (30)
Yellow oil; $^1H$ NMR (300 MHz, CDCl$_3$) $\delta$ 1.11 (3H, s), 1.26 (3H, s), 3.46 (3H, s), 3.71 (3H, s), 4.68 (1H, d, $J$ = 6.5 Hz), 5.85 (1H, br d, $J$ = 6.5 Hz), 6.95-7.01 (2H, m) 7.21-7.27 (2H, m); $^{13}$C NMR (75 MHz, CDCl$_3$) $\delta$ 19.4, 24.6, 45.5, 51.9, 61.8, 66.5, 124.7, 126.3, 126.9, 140.7, 177.2; IR (neat) 2982, 2948, 1730, 1466, 1435, 1262, 1217, 1192, 1136; HRMS (ESI) calcd for C$_{11}$H$_{17}$N$_1$O$_3$S (M + Na$^+$) 266.0827, found 266.0828.

Methyl 2-(1-(methoxyamino)octyl)carboxylate (31)
Pale yellow oil; $^1H$ NMR (300 MHz, CDCl$_3$) $\delta$ 0.88 (3H, t, $J$ = 6.9 Hz), 1.04-1.71 (20H, m), 2.03-2.15 (2H, m), 2.79 (1H, dd, $J$ = 2.1 Hz, $J$ = 10.0 Hz), 3.43 (3H, s), 3.67 (3H, s), 5.68 (1H, br s); $^{13}$C NMR (75 MHz, CDCl$_3$) $\delta$ 22.6, 23.2, 23.4, 24.1, 24.6, 45.5, 51.9, 61.8, 66.5, 124.7, 126.3, 126.9, 140.7, 177.2; IR (neat) 2982, 2948, 1730, 1466, 1435, 1262, 1217, 1192, 1136; HRMS (ESI) calcd for C$_{17}$H$_{33}$N$_1$O$_3$ (M + Na$^+$) 322.2358, found 322.2361.

Methyl 2-ethyl-3-(methoxyamino)-2-methyldecanoate (32)
Major product; Pale yellow oil; $^1H$ NMR (300 MHz, CDCl$_3$) $\delta$ 0.83 (3H, t, $J$ = 6.9 Hz), 1.11 (3H, s), 1.18-1.38 (10H, m), 1.45-1.59 (2H, m), 1.57 (1H, dq, $J$ = 7.6 Hz, $J_{gem}$ = 13.8 Hz), 1.84 (1H, dq, $J$ = 7.6 Hz, $J_{gem}$ = 13.8 Hz), 3.02 (1H, dd, $J$ = 3.1 Hz, $J$ = 8.6 Hz), 3.45 (3H, s), 3.66 (3H, s), 5.55 (1H, br s); $^{13}$C NMR (75 MHz, CDCl$_3$) $\delta$ 22.6, 23.2, 23.4, 24.1, 24.6, 45.5, 51.9, 61.8, 66.5, 124.7, 126.3, 126.9, 140.7, 177.2; IR (neat) 2982, 2948, 1730, 1466, 1435, 1262, 1217, 1192, 1136; HRMS (ESI) calcd for C$_{15}$H$_{31}$N$_1$O$_3$ (M + Na$^+$) 296.2202, found 296.2200.
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Dihydro-3-(1-(methoxyamino)octyl)-3-methylfurane-2(3\text{H})-one (34)

(Major stereoisomers). Pale yellow oil; \(^1\)H NMR (300 MHz, CDCl\(_3\)) \(\delta\) 0.86 (3H, t, \(J = 6.9\) Hz), 0.87 (10H, m), 1.23 (3H, s), 1.42-1.60 (2H, m), 1.83 (1H, ddd, \(J = 7.6\) Hz, \(J_{gem} = 8.9\) Hz, \(J_{gem} = 13.1\) Hz), 2.58 (1H, ddd, \(J = 7.6\) Hz, \(J = 8.9\) Hz, \(J_{gem} = 13.1\) Hz), 3.13 (1H, dd, \(J = 1.7\) Hz, \(J = 9.3\) Hz), 3.44 (3H, s), 4.19-4.31 (1H, m), 4.32 (1H, dt, \(J = 5.2\) Hz, \(J = 8.9\) Hz) 5.59 (1H, br s); 13C NMR (75 MHz, CDCl\(_3\)) \(\delta\) 14.1, 22.2, 22.6, 26.9, 27.6, 29.1, 29.7, 30.3, 31.8, 32.0, 51.7, 60.0, 61.1, 63.9, 126.7, 127.6, 128.4, 139.2, 175.4; IR (neat) 2930, 2857, 1732, 1454, 1225, 1134, 1080, 1038, 1007; HRMS (ESI) calcd for C\(_{14}\)H\(_{27}\)N\(_1\)O\(_3\) (M + Na\(^{+}\)) 280.1889, found 280.1891.

Methyl 2-ethyl-3-(methoxyamino)-2-phenyldecanoate (35)

Major stereoisomers; Colorless oil; 1H NMR (300 MHz, CDCl\(_3\)) \(\delta\) 0.82 (3H, t, \(J = 7.2\) Hz), 0.87 (3H, t, \(J = 6.9\) Hz), 1.17-1.59 (12H, m), 2.15 (1H, dq, \(J = 7.2\) Hz, \(J_{gem} = 14.3\) Hz), 2.31 (1H, dq, \(J = 7.2\) Hz, \(J_{gem} = 14.3\) Hz), 3.45 (3H, s), 3.51 (1H, dd, \(J = 1.7\) Hz, \(J = 10.2\) Hz), 3.73 (3H, s), 5.43 (1H, br s), 7.20-7.35 (5H, m); 13C NMR (75 MHz, CDCl\(_3\)) \(\delta\) 9.4, 14.1, 22.6, 27.8, 28.8, 29.2, 29.7, 31.8, 32.0, 51.7, 60.0, 61.1, 63.9, 126.7, 127.6, 128.4, 139.2, 175.4; IR (neat) 2928, 2857, 1765, 1460, 1196, 1088, 1033; HRMS (ESI) calcd for C\(_{20}\)H\(_{33}\)N\(_1\)O\(_3\) (M + Na\(^{+}\)) 358.2358, found 358.2362.

Methyl 7-benzyloxy-2-ethyl-3-(methoxyamino)-2-phenylheptanoate (36)

Major stereoisomers; Pale yellow oil; 1H NMR (300 MHz, CDCl\(_3\)) \(\delta\) 0.82 (3H, t, \(J = 7.2\) Hz), 1.41-1.71 (6H, m), 2.15 (1H, dq, \(J = 7.2\) Hz, \(J_{gem} = 14.5\) Hz), 2.31 (1H, dq, \(J = 7.2\) Hz, \(J_{gem} = 14.5\) Hz), 3.37-3.47 (2H, m), 3.37 (3H, s), 3.48-3.55 (1H, m), 3.82 (3H, s), 4.48 (2H, s), 4.83 (1H, br s), 7.19-7.37 (10H, m); \(^1\)C NMR (75 MHz, CDCl\(_3\)) \(\delta\) 9.4, 24.3, 28.7, 29.7, 31.8, 51.7, 60.0, 61.1, 63.7, 70.3, 72.8, 126.7, 127.4, 127.6, 128.3, 128.4, 138.7, 139.2, 175.3; IR (neat) 2942, 2865, 1728, 1454, 1225, 1105; HRMS (ESI) calcd for C\(_{24}\)H\(_{33}\)N\(_1\)O\(_4\) (M + Na\(^{+}\)) 422.2307, found 422.2303.

Methyl 2-((methoxyamino)(phenyl)methyl)-2-phenylbutanoate (37)

Major stereoisomers; Colorless crystals; mp 55-57 °C. 1H NMR (300 MHz, CDCl\(_3\)) \(\delta\) 0.81 (3H x 6/7, t, \(J = 7.2\) Hz), 0.86 (3H x 1/7, t, \(J = 7.2\) Hz), 1.85 (1H, dq, \(J = 7.2\) Hz, \(J_{gem} = 14.5\) Hz), 2.06 (1H, dq, \(J = 7.2\) Hz, \(J_{gem} = 14.5\) Hz), 3.37 (3H x 6/7, s), 3.43 (3H x 1/7, s), 3.73 (3H x 6/7, s), 3.79 (3H x 1/7, s), 4.69 (1H x 6/7, d, \(J = 6.2\) Hz), 4.83 (1H x 6/7, d, \(J = 9.3\) Hz), 6.45 (1H x 6/7, br d, \(J = 6.2\) Hz), 6.59 (1H x 1/7, br d, \(J = 9.3\) Hz), 7.04-7.11 (4H, m), 7.17-7.32 (6H, m); \(^1\)C NMR (75 MHz, CDCl\(_3\)) \(\delta\) 9.7, 28.3, 51.8, 59.4, 61.9, 69.9, 126.9, 127.4, 127.5, 127.7, 128.0, 138.9, 139.6, 174.5; IR (neat) 2930, 2859, 1780, 1466, 1437, 1219, 1192, 1165; HRMS (ESI) calcd for C\(_{15}\)H\(_{31}\)N\(_1\)O\(_3\) (M + Na\(^{+}\)) 336.1576, found 336.1579.
Derivitization of \textit{syn}-3-(methoxyamino)-2-substituted esters 2 and 18 to the corresponding \textit{syn}-3-amino-2-substituted esters and the determination of \textit{syn}- or \textit{anti}-seleoc hemistry

**Methyl \textit{syn}-3-amino-2-butyldecanoate**

![Methyl syn-3-amino-2-butyldecanoate](image)

**4-Nitrophenyl \textit{syn}-2-buty l-3-(methoxyamino)decanoate 2** (784 mg, 2.0 mmol) and NaOMe (108 mg, 2.0 mmol) in MeOH (2.0 mL) was stirred at room temperature for 5 h. Water was added to the mixture, which was extracted twice with Et₂O. The combined organic phase was washed with water, brine, dried (Na₂SO₄) and concentrated. The obtained crude oil was purified by SiO₂-column chromatography to give the desired methyl ester. A suspension of the ester (288 mg, 1.0 mmol) and Zn powder (654 mg, 10.0 mmol) in AcOH (2.0 mL) and H₂O (2.0 mL) was heated at 100 °C for 3 h under an Ar atmosphere. Water was added to the mixture, which was extracted five times with CHCl₃. The combined organic phase was washed with water, brine, dried (Na₂SO₄) and concentrated. The obtained crude oil was purified by SiO₂-column chromatography to give the desired product (220 mg, 85%).

Pale yellow oil; ¹H NMR (300 MHz, CDCl₃) δ 0.88 (3H, t, \(J = 6.9\) Hz), 0.89 (3H, t, \(J = 6.9\) Hz), 1.15-1.74 (18H, m), 2.33 (1H, ddd, \(J = 4.1\) Hz, \(J = 5.5\) Hz, \(J = 10.7\) Hz), 2.86-2.96 (1H, m), 3.69 (3H, s); ¹³C NMR (75 MHz, CDCl₃) δ 13.9, 14.0, 22.6, 22.7, 26.4, 27.2, 29.2, 29.5, 30.0, 31.7, 35.4, 51.3, 52.2, 53.1, 175.8; IR (neat) 2928, 2857, 1734, 1460, 1437, 1217, 1192, 1165; HRMS (ESI) calcd for C₁₅H₃₁N₁O₂ (M + Na⁺) 258.2433, found 258.2430.

**Syn-3-butyl-4-hexylazetidinone**

A mixture of methyl \textit{syn}-3-amino-2-butyldecanoate (200 mg, 0.78 mmol) and KOH (218 mg, 39 mmol) - H₂O (5:1; 1.0 mL) was stirred for 10 h at room temperature. 1M HCl aqueous solution was added to the mixture to adjust pH 7. The mixture was extracted three times with CHCl₃, which was washed with water, brine, dried (Na₂SO₄) and concentrated to give crude β-aminoacid (170 mg). Et₃N (236 mg, 2.33 mmol) was added to a stirred solution of the β-aminoacid and 2-chloro-1-methylpyridinium iodide (238 mg, 0.93 mmol) in CH₃CN (2.0 mL) at 45-55 °C under an Ar atmosphere. After stirring at the same temperature for 19 h, water was added to the mixture, which was extracted five times with CHCl₃. The combined organic phase was washed with water, brine, and dried (Na₂SO₄) and concentrated to give the desired product β-lactam (90 mg, 54%).

A mixture of methyl \textit{syn}-3-amino-2-butyldecanoate (200 mg, 0.78 mmol) and KOH (218 mg, 39 mmol) - H₂O (5:1; 1.0 mL) was stirred for 10 h at room temperature. 1M HCl aqueous solution was added to the mixture to adjust pH 7. The mixture was extracted three times with CHCl₃, which was washed with water, brine, dried (Na₂SO₄) and concentrated to give crude β-aminoacid (170 mg). Et₃N (236 mg, 2.33 mmol) was added to a stirred solution of the β-aminoacid and 2-chloro-1-methylpyridinium iodide (238 mg, 0.93 mmol) in CH₃CN (2.0 mL) at 45-55 °C under an Ar atmosphere. After stirring at the same temperature for 19 h, water was added to the mixture, which was extracted five times with CHCl₃. The combined organic phase was washed with water, brine, and dried (Na₂SO₄) and concentrated to give the desired product β-lactam (90 mg, 54%).

\(\text{(syn / anti} = 90 / 10\). colorless oil; ¹H NMR (300 MHz, CDCl₃): δ 0.89 (3H, t, \(J = 6.9\) Hz), 0.91 (3H, t, \(J = 6.9\) Hz), 2.68-2.76 (anti, 0.10H, m), 3.09-3.20 (\textit{syn}, 0.90H, m), 3.27 (\textit{syn}, 0.10H, ddd, \(J = 6.9\), 6.9, 2.1 Hz), 3.63 (anti, 0.90H, ddd, \(J = 9.3, 5.2, 4.5\) Hz), 5.87-6.00 (1H, brs); ¹³C NMR (75 MHz, CDCl₃): δ 13.9, 14.0, 22.5, 22.7, 24.4, 26.3, 26.7, 28.2, 29.1, 29.4, 30.2, 30.7, 31.7, 35.1, 52.3, 52.9, 55.3, 56.8, 171.7, 172.4; IR (neat) 3248, 2928, 2857, 1752, 1464, 1379 cm⁻¹. HRMS (ESI) calcd for C₁₄H₂₇N₁O₁ (M + Na⁺) 258.2433, found 258.2430.
Anti-3-butyl-2-allyloxy-4-hexylazetidinone

A similar procedure using methyl 2-allyloxy-3-methoxyaminodecanoate 18 (137 mg, 0.50 mmol) gave the desired product (58 mg, 55%). (syn / anti = 1 / >99).

Yellow oil; $^1$H NMR (300 MHz, CDCl$_3$): $\delta$ 0.89 (3H, t, $J = 6.9$ Hz), 1.19-1.49 (10H, m), 1.55-1.70 (2H, m), 3.57 ($anti$, 1H, ddd, $J = 6.9$, 6.9, 1.7 Hz), 4.08-4.18 (1H, m), 4.21-4.31 (2H, m), 5.19-5.38 (2H, m), 5.84-6.01 (1H, m), 6.02-6.17 (1H, brs); $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta$ 13.9, 22.4, 26.1, 28.9, 29.1, 31.5, 33.2, 57.3, 86.1, 117.8, 133.7, 166.8, 167.9; IR (neat) 3274, 2926, 2857, 1763, 1182, 1144 cm$^{-1}$. HRMS (ESI) calcd for C$_{13}$H$_{23}$N$_1$O$_2$ (M + Na$^+$) 248.1626, found 248.1631.

Methyl 3-amino-2,2-dimethyldecanoate

A similar procedure using methyl 3-(methoxyamino)-2,2-dimethyldecanoate 28 (144 mg, 0.50 mmol) and Zn powder (327 mg, 5.0 mmol) gave the desired product (100 mg, 87%).

Pale yellow oil; $^1$H NMR (300 MHz, CDCl$_3$) $\delta$ 0.88 (3H, t, $J = 6.9$ Hz), 1.13 (3H, s), 1.14 (3H, s), 1.21-1.60 (12H, m), 2.83 (1H, dd, $J = 2.1$ Hz, $J = 10.7$ Hz), 3.68 (3H, s); $^{13}$C NMR (75 MHz, CDCl$_3$) $\delta$ 14.1, 20.9, 21.3, 22.6, 27.3, 29.3, 29.8, 31.8, 32.5, 47.6, 51.6, 57.7, 178.1; IR (neat) 2928, 2857, 1732, 1466, 1389, 1265, 1192, 1134; HRMS (ESI) calcd for C$_{13}$H$_{27}$N$_1$O$_2$ (M + Na$^+$) 230.2120, found 230.2119.