SmI₂-Promoted Intermolecular Radical Addition of Nitrones to α,β -Unsaturated Amides and Esters: Direct Synthesis of γ -Amino Acid and Peptide Mimics Via a Nitrogen Equivalent to the Ketyl Radical

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Supporting Information

General Methods. The ¹H NMR and ¹³C NMR were recorded at 200 MHz and 50 MHz, respectively. The chemical shifts are reported in ppm downfield to TMS (d = 0) for ¹H NMR and relative to the central CDCl₃ resonance (d = 77.16) for ¹³C. Solvents were dried according to standard procedures. Optical rotations were measured on a Perkin-Elmer 241 Polarimeter.

General Procedure for the Addition of Nitrones to Acrylamides:

The nitrone (0.5 mmol) and the acrylamide (0.5 mmol) were placed in a round bottomed flask fitted with a magnetic stirring bar whereafter the flask was flushed with Argon. Dry THF (10 ml) was added and the solution was cooled to –78°. SmI₂ (12 ml) was added drop wise and the solution was left stirring overnight. The reaction was quenched with sat. NH₄Cl (2 ml) at –78° and warmed to room temperature. 30 ml of water was added and the mixture was extracted three times with CH₂Cl₂ (3x100 ml). The organic phase was washed with sat. NaCl, dried with MgSO₄ and evaporated *in vacuo*. The compounds were purified by flash chromatography using Pentane:EtOAc as the eluent.

General Procedure for the Addition of Nitrones to Acrylates:

The nitrone (0.5 mmol) and the acrylate (0.5 mmol) were placed in a round bottomed flask fitted with a magnetic stirring bar where after the flask was flushed with Argon. Dry THF (10 ml) and *t*-BuOH (1.0 mmol) were added and the solution was cooled to –78°. SmI₂ (12 ml) was added drop wise and the solution was left stirring overnight. The reaction was quenched with sat. NH₄Cl (2 ml) at –78° and warmed to room temperature. 30 ml of water was added and the mixture was extracted three times with DCM (3*100 ml). The organic phase was washed with sat. NaCl, dried with MgSO₄ and evaporated *in vacuo*. The compounds were purified by flash chromatography using Pentane:EtOAc as the eluent.

(4-Benzyl-hydroxy-amino)-4-cyclohexylbutyrylamino)-acetic acid methyl ester.

¹H NMR (200 MHz, CDCl₃) δ (ppm) 7.16-7.42 (m, 5H), 7.01 (bt, 1H, *J* = 5.5 Hz), 5.80 (bs, 1H), 4.07 (d, 1H, *J* = 13.2 Hz), 3.95 (dd, 1H, *J* = 5.4, 18.6 Hz), 3.82 (dd, 1H, *J* = 4.4, 18.6 Hz), 3.77 (d, 1H, *J* = 13.2 Hz), 3.70 (s, 3H), 1.00-2.53 (m, 16H).

¹³C NMR (50 MHz, CDCl₃) δ (ppm) 176.36, 173.33, 140.99, 131.26 (2C), 130.29 (2C), 129.13, 73.65, 62.72, 54.51, 43.09, 39.48, 37.68, 34.67, 32.23, 29.09, 28.70 (2C), 24.76.

HRMS C₂₀H₃₀N₂O₄ [M+Na⁺]; calculated: 385.2103 found: 385.2102.

IR 3324, 3030, 2924, 1748, 1652, 1538 cm⁻¹.

2-(4-(Benzyl-hydroxy-amino)-4-cyclohexylbutyrylamino)-3-phenylpropionic acid methyl ester.

Mixture of stereoisomers; major a, minor b:

¹H NMR (200 MHz, CDCl₃) δ (ppm) 7.15-7.38 (m, 8H), 7.00-7.14 (m, 2H), 6.73 (d, 1Ha, *J* = 7.0 Hz), 6.35 (d, 1Hb, *J* = 7.8 Hz), 5.76 (s, 1Ha), 5.38 (s, 1Hb), 4.88 (dd, 1Hb, *J* = 6.0, 11.8), 4.84 (ddd, 1Ha, *J* = 6.1, 7.8, 17.8 Hz), 4.02 (d, 1Ha, *J* = 13.6), 3.95 (d, 1Hb, *J* = 13.8 Hz), 3.80 (d, 1Hb, *J* = 13.8 Hz), 3.75 (d, 1Ha, *J* = 13.6), 3.69 (s, 3Ha), 3.67 (s, 3Hb), 3.05 (d, 2Hb, *J* = 6.0), 3.03 (dd, 1Ha, *J* = 6.1, 14.0 Hz), 2.92 (dd, 1Ha, *J* = 6.1, 14.0 Hz), 0.92-2.48 (m, 16H)

¹³C NMR (50 MHz, CDCl₃) δ (ppm): Major isomer: 175.5, 174.6, 141.0, 138.01, 131.3 (4C), 130.3 (4C), 129.1 (2C), 72.0, 61.9, 55.1, 54.4, 40.0, 39.8, 36.9, 34.2, 32.2, 29.0, 28.7 (2C), 24.6. Minor isomer: 175.8, 175.0, 140.8, 138.1, 131.1 (4C), 130.6 (4C), 129.2 (2C), 73.0, 62.6, 55.0, 54.4, 40.0, 39.3, 37.4, 34.5, 32.2 (2C), 29.0, 24.60.

HRMS C₂₇H₃₆N₂O₄ [M+Na⁺]; calculated: 475.2573 found: 475.2569.

(4-(Benzyl-hydroxy-amino)-hexanoylamino)-acetic acid methyl ester

^1H NMR (200 MHz, CDCl_3) δ (ppm) 6.95-7.16 (m, 5H), 6.93 (t, 1H, $J = 5.5$ Hz), 5.75 (s, 1H), 3.77 (d, 1H, $J = 13.2$ Hz), 3.66 (t, 2H, $J = 5.0$ Hz), 3.46 (d, 1H, $J = 13.2$ Hz), 3.45 (s, 3H), 2.12-2.35 (m, 2H), 1.99 (ddd, 1H, $J = 4.7, 8.4, 14.7$ Hz), 1.50-1.82 (m, 3H), 1.11-1.34 (m, 1H), 0.86 (t, 3H, $J = 7.5$ Hz).
 ^{13}C NMR (50 MHz, CDCl_3) δ (ppm) 176.48, 173.24, 140.85, 131.16 (2C), 130.19 (2C), 129.02, 70.33, 62.026, 54.42, 43.03, 36.52, 27.72, 22.03, 13.75.

HRMS $\text{C}_{16}\text{H}_{24}\text{N}_2\text{O}_4$ $[\text{M}+\text{Na}^+]$; calculated: 331.1634 found: 331.1635.

IR 3342, 3062, 3031, 2958, 1752, 1648, 1542, 1496 cm^{-1} .

(4-(Benzyl-hydroxy-amino)-5-methyl-hexanoylamino)-acetic acid methyl ester.

^1H NMR (200 MHz, CDCl_3) δ (ppm) 7.20-7.35 (m, 5H), 7.10 (bt, 1H, $J = \text{Hz}$), 5.91 (bs, 1H), 4.04 (d, 1H, $J = 13.1$ Hz), 3.95 (dd, 1H, $J = 5.4, 18.6$ Hz), 3.81 (dd, 1H, $J = 4.6, 18.6$ Hz), 3.77 (d, 1H, $J = 13.1$ Hz), 3.68 (s, 3H), 2.20-2.51 (m, 4H), 1.94-2.13 (m, 1H), 1.63-1.78 (m, 1H), 1.00 (dd, 6H, $J = 6.8, 13.4$ Hz).

^{13}C NMR (50 MHz, CDCl_3) δ (ppm) 176.37, 173.21, 140.94, 131.20 (2C), 130.21 (2C), 129.05, 73.31, 62.61, 54.40, 43.01, 37.41, 29.04, 24.30, 23.93, 21.41.

HRMS $\text{C}_{17}\text{H}_{26}\text{N}_2\text{O}_4$ $[\text{M}+\text{Na}^+]$; calculated: 345.1790 found: 345.1798.

IR 3307, 3070, 3029, 1738, 1668, 1557 cm^{-1} .

2-(2-(4-(Benzyl-hydroxy-amino)-4-cyclohexyl-butyrylamino)-4-methyl-pentanoyl-amino)-3-phenyl-propionic acid methyl ester.

Mixture of stereoisomers; major a, minor b:

^1H NMR (200 MHz, CDCl_3) δ (ppm) 7.68 (d, 1Ha, $J = 8.8$ Hz), 7.07-7.31 (m, 10H), 6.79 (m, 1Ha+1Hb), 6.66 (d, 1Hb, $J = 8.4$ Hz), 4.73-4.82 (m, 1H), 4.38-4.54 (m, 1H), 4.11 (d, 1Ha, $J = 12.8$ Hz), 3.97 (d, 1Hb, $J = 13.6$ Hz), 3.85 (d, 1Hb, $J = 13.6$ Hz), 3.84 (d, 1Ha, $J = 12.8$ Hz), 3.68 (s, 3Ha), 3.66 (s, 3Hb), 2.96-3.15 (m, 2H), 0.99-2.42 (m, 19H), 0.89 (d, 6Ha, $J = 5.4$ Hz), 0.86 (d, 6Hb, $J = 6.6$ Hz).

HRMS $\text{C}_{33}\text{H}_{47}\text{N}_3\text{O}_5$ $[\text{M}+\text{Na}^+]$; calculated: 588.3413 found: 588.3434.

IR 3288, 3064, 2925, 2852, 1749, 1644, 1539 cm^{-1} .

(4-(Benzyl-hydroxy-amino)-4-cyclohexyl-2-methylbutyrylamino)-acetic acid methyl ester.

^1H NMR (200 MHz, CDCl_3) δ (ppm) 7.23-7.34 (m, 5H), 6.90 (bt, 1H, $J = 5.2$ Hz), 5.56 (bs, 1H), 4.03 (dd, 1H, $J = 5.6, 18.6$ Hz), 4.00 (d, 1H, $J = 13.0$ Hz), 3.81 (dd, 1H, $J = 4.6, 18.6$ Hz), 3.81 (d, 1H, $J = 13.0$ Hz), 3.69 (s, 3H), 2.63-2.76 (m, 2H), 1.65-2.08 (m, 8H), 1.16 (d, 3H, $J = 7.0$ Hz), 1.00-1.42 (m, 5H).

^{13}C NMR (50 MHz, CDCl_3) δ (ppm) 176.93, 171.34, 139.01, 129.18 (2C), 128.37 (2C), 127.15, 67.68, 59.25, 52.46, 40.96, 38.35, 37.56, 32.39, 32.00, 30.10, 26.99, 26.66 (2C), 17.67.

HRMS $\text{C}_{21}\text{H}_{32}\text{N}_2\text{O}_4$ $[\text{M}+\text{Na}^+]$; calculated: 399.2260 found: 399.2252.

IR 3311, 3063, 2971, 1756, 1661, 1538 cm^{-1} .

4-(Benzyl-hydroxy-amino)-4-cyclohexyl-N-(2-hydroxyindanyl)-butyramide.

Mixture of stereoisomers; major a, minor b:

^1H NMR (200 MHz, CDCl_3) δ (ppm) 7.59 (d, 1Ha, $J = 9.0$ Hz), 6.94-7.29 (m, 9H), 6.70 (d, 1Hb, $J = 8.6$ Hz), 5.29 (dd, 1H, $J = 5.2, 8.6$ Hz), 4.49 (dt, 1Hb, $J = 2.4, 5.2$ Hz), 4.35 (dt, 1Ha, $J = 1.6, 5.0$), 4.01 (d, 1Ha, $J = 12.8$ Hz), 3.99 (d, 1Hb, $J = 13.4$ Hz), 3.81 (d, 1Hb, $J = 13.4$ Hz), 3.71 (d, 1Ha, $J = 12.8$ Hz), 3.08 (dd, 1Hb, $J = 5.0, 16.5$ Hz), 3.02 (dd, 1Ha, $J = 5.0, 16.7$ Hz), 2.84 (dd, 1Hb, $J = 2.2, 16.5$ Hz), 2.68 (dd, 1Ha, $J = 1.4, 16.7$ Hz), 2.20-2.56 (m, 3H), 1.53-2.13 (m, 6H), 0.97-1.38 (m, 7H).

^{13}C NMR (50 MHz, CDCl_3) δ (ppm) 177.13, 143.02, 141.71, 140.43, 131.59 (2C), 130.20 (3C), 129.29, 129.12, 127.46, 126.60, 75.90, 74.24, 63.18, 59.38, 41.53, 39.08, 38.35, 34.76, 32.06, 29.11, 28.66 (2C), 25.08.

HRMS $\text{C}_{26}\text{H}_{34}\text{N}_2\text{O}_3$ $[\text{M}+\text{Na}^+]$; calculated: 445.2467 found: 445.2465.

IR 3478, 3290, 3075, 3025, 2919, 1649, 1530 cm^{-1} .

4-(Benzyl-hydroxy-amino)-4-cyclohexylbutyric acid butyl ester.

^1H NMR (200 MHz, CDCl_3) δ (ppm) 7.15-7.36 (m, 5H), 4.89 (s, 1H), 4.06 (dt, 1H, $J = 6.6, 10.6$ Hz), 3.99 (d, 1H, $J = 13.3$ Hz), 3.96 (dt, 1H, $J = 7.0, 10.6$ Hz), 3.79 (d, 1H, $J = 13.3$ Hz),

2.26-2.56 (m, 3H), 1.03-2.09 (m, 17H), 0.92 (t, 3H, J = 7.5 Hz).

^{13}C NMR (50 MHz, CDCl_3) δ (ppm) 175.11, 138.99, 129.07 (2C), 128.21 (2C), 127.03, 70.28, 64.46, 60.54, 37.77, 33.21, 32.20, 30.74, 30.28, 26.99, 26.69 (2C), 22.18, 19.26, 13.86.

HRMS $\text{C}_{21}\text{H}_{33}\text{NO}_3$ $[\text{M}+\text{Na}^+]$; calculated: 370.2358 found: 370.2359.

IR 3459, 3063, 3029, 2930, 1732, 1604, 1496 cm^{-1} .

4-(1-(Benzyl-hydroxy-amino)-cyclohexyl)-butyric acid butyl ester.

^1H NMR (200 MHz, CDCl_3) δ (ppm) 7.81-7.98 (m, 5H), 5.28 (s, 1H), 4.64 (t, 2H, J = 6.6 Hz), 4.46 (s, 2H), 3.06 (t, 2H, J = 7.3 Hz), 2.61 (t, 2H, J = 7.3 Hz), 1.92-2.41 (m, 14H), 1.55 (t, 3H, J = 7.7 Hz).

^{13}C NMR (50 MHz, CDCl_3) δ (ppm) 174.93, 139.89, 128.71 (2C), 127.96 (2C), 126.54, 77.54, 64.24, 61.76, 54.25, 33.80, 31.05, 30.51, 28.84, 27.24, 26.13, 24.29, 21.93, 19.02, 13.62.

HRMS $\text{C}_{20}\text{H}_{31}\text{NO}_3$ $[\text{M}+\text{Na}^+]$; calculated: 356.2202 found: 356.2208.

IR 3482, 3062, 3029, 2936, 1735, 1604, 1496 cm^{-1} .

4-(Benzyl-hydroxy-amino)-4-cyclohexyl-2-methyl-butyl butyric acid methyl ester.

Major stereoisomer :

^1H NMR (200 MHz, CDCl_3) δ (ppm) 6.96-7.13 (m, 5H), 4.52 (s, 1H), 3.73 (d, 1H, J = 13.2 Hz), 3.54 (d, 1H, J = 13.2 Hz), 3.34 (s, 3H), 2.47 (ddk, 1H, J = 4.4, 7.4, 14.6 Hz), 2.25 (dt, 1H, J = 4.0, 8.4 Hz), 1.37-1.81 (m, 8H), 1.93 (d, 3H, J = 7.1 Hz), 0.74-1.19 (m, 5H).

^{13}C NMR (50 MHz, CDCl_3) δ (ppm) 177.45, 138.68, 129.00 (2C), 128.01 (2C), 126.85, 67.22, 60.70, 51.39, 38.15, 37.24, 31.61, 30.89, 29.85, 26.73, 26.48 (2C), 16.81.

HRMS $\text{C}_{19}\text{H}_{29}\text{NO}_3$ $[\text{M}+\text{Na}^+]$; calculated: 342.2045 found: 342.2038.

IR 3222, 3025, 2924, 2850, 1732, 1496 cm^{-1} .

4-(Benzyl-hydroxy-amino)-4-cyclohexyl-3-methyl-butyl butyric acid ethyl ester.

^1H NMR (200 MHz, CDCl_3) δ (ppm) 7.09-7.19 (m, 5H), 4.12 (q, 2H, J = 7.0), 4.09 (d, 1H, J = 13.2 Hz), 3.88 (d, 1H, J = 13.2 Hz), 2.43-2.69 (m, 2H), 2.27 (dd, 1H, J = 6.6, 13.6 Hz), 1.44-2.11 (m, 6H), 1.25 (t, 3H, J = 7.0), 1.03 (d, 3H, J = 6.6 Hz), 0.79-1.36 (m, 6H).

^{13}C NMR (50 MHz, CDCl_3) δ (ppm) 173.7, 139.2, 129.1 (2C), 128.5, 128.2 (2C), 127.0, 73.1, 63.0, 60.1, 40.1, 37.7, 32.2, 31.3, 30.5, 26.7 (2C), 17.3, 14.3.

HRMS $\text{C}_{20}\text{H}_{31}\text{NO}_3$ $[\text{M}+\text{Na}^+]$; calculated: 356.2202 found: 356.2196.

IR 3396, 3055, 2929, 2852, 1731, 1578, 1452 cm^{-1} .

2-Benzyl-3-cyclohexyl-(1,2)oxazinan-6-one.

^1H NMR (200 MHz, CDCl_3) δ (ppm) 7.11-7.22 (m, 5H), 4.34 (d, 1H, J = 14.4 Hz), 3.89 (d, 1H, J = 14.4 Hz), 2.93 (ddd, 1H, J = 3.2, 5.4, 9.0 Hz), 2.67 (ddd, 1H, J = 7.8, 12.6, 14.8 Hz), 2.40 (ddd, 1H, J = 3.4, 6.0, 14.8 Hz), 2.05-2.20 (m, 1H), 0.77-1.97 (m, 12H).

^{13}C NMR (50 MHz, CDCl_3) δ (ppm) 175.29, 136.28, 128.71 (2C), 128.15 (2C), 127.23, 64.51, 58.42, 39.91, 29.72, 27.59, 26.40, 26.35, 25.68, 24.65, 21.61.

HRMS $\text{C}_{17}\text{H}_{23}\text{NO}_2$ $[\text{M}+\text{Na}^+]$; calculated: 296.1626 found: 296.1623.

IR 3062, 3031, 2928, 1760, 1496 cm^{-1} .

2-Benzyl-3-isopropyl-(1,2)oxazinan-6-one (6).

^1H NMR (200 MHz, CDCl_3) δ (ppm) 7.21-7.38 (m, 5H), 4.34 (d, 1H, J = 14.4 Hz), 3.87 (d, 1H, J = 14.4 Hz), 2.97 (ddd, 1H, J = 3.6, 5.8, 9.2 Hz), 2.68 (ddd, 1H, J = 7.8, 12.6, 14.8 Hz), 2.41 (ddd, 1H, J = 3.0, 6.0, 14.8 Hz), 1.98-2.28 (m, 2H), 1.68-1.89 (m, 1H), 0.98 (d, 3H, J = 4.0 Hz), 0.94 (d, 3H, J = 4.4 Hz).

^{13}C NMR (50 MHz, CDCl_3) δ (ppm) 177.49, 138.39, 131.06 (2C), 130.37 (2C), 129.49, 66.67, 60.49, 31.11, 29.90, 22.53, 21.66, 16.24.

HRMS $\text{C}_{14}\text{H}_{19}\text{NO}_2$ $[\text{M}+\text{Na}^+]$; calculated: 256.1313 found: 256.1316.

IR 3064, 3032, 2964, 2876, 1756, 1497 cm^{-1} .

4-(Benzyl-hydroxy-amino)-4-cyclohexylbutyric acid isosorbide ester (4c).

Major stereoisomer:

^1H NMR (200 MHz, CDCl_3) δ (ppm) 7.18-7.43 (m, 10H), 5.10 (d, 1H, $J = 3.2$ Hz), 4.93 (s, 1H), 4.76 (d, 1H, $J = 11.8$ Hz), 4.64 (t, 1H, $J = 4.4$ Hz), 4.55 (d, 1H, $J = 11.8$ Hz), 4.46 (d, 1H, $J = 4.4$ Hz), 3.94-4.12 (m, 4H), 3.74-3.90 (m, 2H), 3.62 (t, 1H, $J = 7.8$ Hz), 2.28-2.54 (m, 3H), 1.64-2.17 (m, 8H), 0.98-1.34 (m, 5H).

^{13}C NMR (50 MHz, CDCl_3) δ (ppm) 174.0, 138.8, 137.7, 129.2 (2C), 128.5 (2C), 128.3 (2C), 128.0 (3C), 127.1, 86.0, 80.6, 79.1, 78.8, 73.7, 72.6, 70.4, 69.9, 60.6, 37.6, 33.0, 32.2, 30.3, 27.0, 26.7 (2C), 22.1.

HRMS $\text{C}_{30}\text{H}_{39}\text{NO}_6$ [$\text{M}+\text{Na}^+$]; calculated: 532.2675 found: 532.2665.

4-(Benzyl-hydroxy-amino)-4-cyclohexyl-butyric acid menthol ester (4a).

Major stereoisomer:

^1H NMR (200 MHz, CDCl_3) δ (ppm) 7.18-7.39 (m, 5H), 4.85 (s, 1H), 4.71 (dt, 1H, $J = 4.4$, 10.6 Hz), 3.96 (d, 1H, $J = 13.4$), 3.87 (d, 1H, $J = 13.4$), 2.27-2.49 (m, 3H), 0.72-2.18 (m, 22H), 0.90 (d, 6H, $J = 9.8$ Hz), 0.78 (d, 3H, 7.0 Hz).

^{13}C NMR (50 MHz, CDCl_3) δ (ppm) 176.21, 140.77, 131.19 (2C), 130.03 (2C), 129.11, 76.21, 71.34, 62.48, 49.16, 43.07, 40.17, 36.36, 35.25, 33.76, 33.49, 32.29, 28.97, 28.72 (2C), 28.30, 25.49, 24.14, 23.97, 22.92, 18.43.

HRMS $\text{C}_{27}\text{H}_{43}\text{NO}_3$ [$\text{M}+\text{Na}^+$]; calculated: 452.3141 found: 452.3129.

Methyl (4S)-4-tert-Butoxycarbonylamino-5-methylhexanoate (7).

6 (40 mg, 0.171 mmol) was dissolved in MeOH (3 ml) and $\text{Pd}(\text{OH})_2/\text{C}$ and 1 drop of acetic acid was added. The reaction mixture was stirred under 25 bar of H_2 for 24 h, filtered and evaporated *in vacuo*. The crude amino acid was dissolved in MeOH (3 ml) and treated with NaHCO_3 (28.7 mg, 0.342 mmol) and $(\text{Boc})_2\text{O}$ (37.4 mg, 0.171 mmol). The mixture was stirred for 24 h, filtered and evaporated *in vacuo*. Half of the residue was dissolved in MeOH (0.7 ml) and toluene (1.7 ml) and 342 μL of a 2 M trimethylsilyl diazomethane solution in hexane was added drop wise. After 30 min the excess of trimethylsilyl diazomethane was decomposed by adding a few drops of acetic acid until the solution turned from yellow to colourless. Evaporation of the solvents afforded the crude product, which was purified by flash chromatography (Pentane:EtOAc, 5:1) to give the methyl (4S)-4-tert-butoxycarbonylamino-5-methylhexanoate (**7**) in overall quantitative yield.

$[\alpha]_D^{25} = -5.5^\circ$ ($c = 1.9$, CHCl_3)

^1H NMR (200 MHz, CDCl_3) δ (ppm) 0.88 (d, $J = 6.5$ Hz, 3H), 0.91 (d, $J = 6.6$ Hz, 3H), 1.43 (s, 9H), 1.59-1.91 (m, 3H), 2.37 (t, $J = 7.5$ Hz, 2H), 3.37-3.49 (m, 1H), 3.67 (s, 3H), 4.3 (d, $J = 9.9$ Hz, 1H).

^{13}C NMR (50 MHz, CDCl_3) δ (ppm) 174.4, 156.1, 79.1, 55.5, 51.8, 32.7, 31.4, 28.5 (3C), 27.8, 19.1, 17.9.

HRMS $\text{C}_{13}\text{H}_{25}\text{NO}_4$ [$\text{M}+\text{Na}^+$]; calculated: 282.1681 found: 282.1693.

IR 2966, 1738, 1682, 1518 cm^{-1} .