# A Highly Enantioselective Route to Either Enantiomer of Both α- and β-Amino Acid Derivatives

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## Supporting Information

**General**. Chemicals and solvents were either purchased *puriss p.A.* from commercial suppliers or purified by standard techniques. 5-Hexenal was prepared from 5-hexenol according to Farquhar et al.[1] For thin-layer chromatography (TLC), silica gel plates Merck 60 F254 were used and compounds were visualized by irradiation with UV light and/ or by treatment with a solution of phosphomolybdic acid (25g), $Ce(SO_4)_2 \bullet H_2O$) (10g), conc. $H_2SO_4$ (60 mL), and $H_2O$ (940 mL) followed by heating or by treatment with a solution of *p*-anisaldehyde (23 mL), conc. $H_2SO_4$ (35 mL), acetic acid (10 mL), and ethanol (900 mL) followed by heating. Flash chromatography was performed using silica gel Merck 60 (particle size 0.040-0.063 mm), $^1$H NMR and $^{13}$C NMR spectra were recorded on Bruker AMX 300, AMX 250, AMX 500 and AMX 400 instruments. The chemical shifts are given in δ relative to TMS (δ = 0 ppm). The spectra were in $CDCl_3$ at room temperature. High-resolution mass spectra were recorded on an Ion Spec Fourier Transform Mass Spectrometer using dihydrobenzoic acid (DHB) as the matrix. HPLC was carried out using a Hitachi organizer consisting of a D-2500 Chromato-Integrator, a L-4000 UV-Detector, and a L-6200A Intelligent Pump. Optical rotations were recorded on a Perkin Elmer 241 Polarimeter (λ = 589 nm, 1dm cell).

**General procedure for the catalytic asymmetric Mannich-reaction between *N*-PMP-protected α-imino ethyl glyoxylate and aldehyde donors:**

In a typical experiment, *N*-PMP-protected α-imino ethyl glyoxylate (0.5 mmol) was dissolved in anhydrous dioxane and the corresponding aldehyde donor (0.75 mmol) was added, followed by *L*-proline (5 mol%). The total volume of the reaction mixture was 5 mL. After stirring for 2-24h at room temperature, the mixture was worked up by addition of half-saturated ammonium chloride solution and extraction with ethyl acetate. The combined organic layers were dried ($MgSO_4$), filtered, concentrated and the residue purified by column chromatography (silica, hexanes/ethyl acetate mixtures) to afford the corresponding Mannich addition product. The ee's of all products were determined by chiral HPLC analysis.

*Ethyl (2S, 3S)-3-formyl-2-(p-methoxyphenylamino)-4-methyl-pentanoate* (**1**): $^1$H NMR (250 MHz): δ = 1.03 (d, 3H, *J* = 6.6 Hz, CHC*H*$_3$), 1.17 (d, 1H, *J* = 6.6 Hz, CHC*H*$_3$), 1. 22 (t, 3H, OCH$_2$C*H*$_3$), 2.34 (m, 1H), 2.56 (m, 1H), 3.74 (s, 3H, OC*H*$_3$), 3.87 (bs, 1H, ArN*H*CH), 4.15 (m, 2H, OC*H*$_2$CH$_3$), 4.17 (bs, 1H), 6.66 (d, 2H, *J* = 7.0 Hz, Ar*H*), 6.78 (d, 2H, *J* = 7.0 Hz, Ar*H*), 9.78 (bs, 1H, CHC*H*O); $^{13}$C NMR (125 MHz): δ = 203.9, 172.9, 153.4, 140.5. 116.0, 115.1, 61.7, 59.8, 57.3, 55.9, 26.6, 21.2, 20.0, 14.4; HPLC (Daicel

(1) Farquhar, D.; Cherif, A.; Bakina, E.; Nelson, J. A. *J. Med. Chem.* **1998**, *41*, 965.
(2) Córdova, A.; Notz, W.; Barbas III, C. F. *J. Org Chem* **2001**, In press.
(3) Greff, Z.; Horvath, Z.; Nyitrai, J.; Kajtar-Peredy, M.; Brlik, J. *J. Chem. Research* **1990**, *M*, 1201.
(4) Palomo, C.; Aizpurua, J. M.; Gracenea, J. J.; Garcia-Granada, S.; Pertierra, P. *Eur. J. Org. Chem.* **1998**, 2201.

Chiralpak AS, hexane/i-PrOH = 99:1, flow rate 1.0 mL/min, λ = 254 nm); $t_R$ (major) = 8.82 min; $t_R$ (minor) = 13.24 min; HRMS: Calcd for $C_{16}H_{23}NO_4$ ($M+Na^+$): 316.1519, found: 316.1521.

*Ethyl (2S, 3S)-3-formyl-2-(p-methoxyphenylamino)-butanoate* (**2**):
$^1$H NMR (250 MHz) ~ 1.1:1 mixture of diastereomers, * denotes minor diastereomer, δ = 1.10 - 1.40 (m, 12*H*), 2.87 (m, 2*H*), 3.73 (bs, 3H*, 3H, OC$H_3$), 3.91 (bs, 1H*, 1H, ArN*H*CH), 4.16 (m, 2H*, 2H, OC$H_2$CH$_3$), 4.38 (bs, 1H*), 4.49 (bs, 1H), 6.67 (m, 2H*, 2H, ArH), 6.77 (m, 2H*, 2H, ArH), 9.72 (bs, 1H*, 1H, CHC*H*O); $^{13}$C NMR (100 MHz): δ = 201.8, 201.7, 172.3, 171.7, 153.4, 153.1, 140.4, 140.1, 116.3, 115.6, 114.8, 114.7, 61.6, 61.5, 58.6, 58.4, 55.6, 55.5, 48.4, 48.1, 14.1, 14.1, 9.8, 9.0; HPLC (Daicel Chiralpak AS, hexane/i-PrOH = 99:1, flow rate 1.0 mL/min, λ = 254 nm); $t_R$ (major) = 18.83 min; $t_R$ (major)* = 19.99 min; $t_R$ (minor) = 23.04 min; $t_R$ (minor)* = 26.35 min. HRMS; Calcd for $C_{14}H_{19}NO_4$ ($M+Na^+$): 265.1309, found: 265.1316.

*Ethyl (2S, 3S)-3-formyl-2-(p-methoxyphenylamino)-pentanoate* (**3**): $^1$H NMR (250 MHz) ~ 1.8:1 mixture of diastereomers, *denotes minor diastereomer, δ = 1.01 (m, 3H, 2H*), 1.23 (m, 3H, 2H*), 1.65-1.68 (m, 1H, 2H*), 1.89 (m, 1H), 2.67 (m, 1H, 0.5H*), 3.67 (s, 3H, 2H*, OC$H_3$), 3,73 (bs, 1H, 0.5H*, ArN*H*CH), 3.95 (bs, 1H, ArN*H*CH), 4.17 (m, 2H, H*, OCH$_2$CH$_3$), 4.25 (bs, 0.5H*), 4.33 (bs, 1H), 6.65 (d, 2H, 1H*, Ar*H*), 6.77 (d, 2H, 1H*, Ar*H*), 9.67 (bs, 0.5H, C*H*O); $^{13}$C NMR (100 MHz): (major diastereomer) δ = 202.8, 172.7, 153.5, 140.7, 116.3, 115.1, 61.8, 58.3, 55.9, 55.6, 18.8, 14.4, 12.3; HPLC (Daicel Chiralpak AS, hexane/i-PrOH = 99:1, flow rate 1.0 mL/min, λ = 254 nm); $t_R$ (major) = 11.19 min; $t_R$ (major)* = 12.84 min; $t_R$ (minor) = 13.74 min; $t_R$ (minor)* = 18.75 min. HRMS: Calcd for $C_{15}H_{21}NO_4$ ($M+Na^+$): 279.1465, found: 279.1467.

*Ethyl (2S, 3S)-3-formyl-2-(p-methoxyphenylamino)-heptanoate* (**4**): $^1$H NMR (250 MHz) ~ 3:1 mixture of diastereomers, * denotes minor diastereomer, δ = 0.8-1.0 (m, 3H, 1.5H*), 1.23 (m, 6H), 1.24-1.50 (m, 8H), 1.60 (m, 1H, 0.4H*), 1.71 (m 0.4H*), 1.85 (m, 1H), 2.71 (m, 1H, 0.5H*), 3.73 (s, 3H, 1H*, OC$H_3$), 3.98 (bs, 1H, 0.3H*, ArN*H*CH), 4.18 (m, 2H, 0.6H*, OC$H_2$CH$_3$), 4.26 (d, 0.3H*, *J* = 6.3Hz), 4.35 (d, 1H, *J* = 4.8 Hz), 6.65 (d, 2H, 0.6H*, Ar*H*), 6.76 (d, 2H, 0.6H*, Ar*H*), 9.66 (d, 0.3H*, C*H*O), 9.71 (d, 1*H*, CHO); $^{13}$C NMR (major diastereomer) (100 MHz): δ = 202.9, 172.7, 153.5, 140.7, 116.3, 115.0, 61.8, 58.6, 55.9, 53.9, 29.9, 25.1, 22.9, 14.4, 14.0; HPLC (Daicel Chiralpak AS, hexane/i-PrOH = 99:1, flow rate 1.0 mL/min, λ = 254 nm); $t_R$ (major) = 8.90 min; $t_R$ (minor) = 11.00 min; HRMS: Calcd for $C_{17}H_{25}NO_4$ ($M+Na^+$): 307.1778, found: 307.1781.

*Ethyl (2S, 3S)-3-formyl-2-(p-methoxyphenylamino)-octanoate* (**5**): $^1$H NMR (400 MHz) δ = 0.89 (t, 3H, J = 4.8Hz), 1.24 (m, 3H, OC$H_2$CH$_3$), 1.31 (bs, 6H), 1.59 (m, 1H), 1.87 (m, 1H), 2.71 (m, 1H), 3.74 (s, 3H, OC$H_3$), 4.14 (m, 2H, OC$H_2$CH$_3$), 4.35 (bs, 1H), 6.65 (d, 2H, *J* = 7.3 Hz, Ar*H*), 6.77 (d, 2H, *J* = 7.3 Hz, Ar*H*), 9.71 (d, 1H, *J* = 1.8 Hz, C*H*O); $^{13}$C NMR (100 MHz): δ = 203.0, 172.7, 153.6, 140.8, 116.4, 115.1, 61.8, 58.7, 55.9, 54.0, 31.9, 27.4, 25.4, 22.6, 14.4, 14.2; HPLC (Daicel Chiralpak AS, hexane/i-PrOH = 99:1, flow rate 1.0 mL/min, λ = 254 nm); $t_R$ (major) = 7.67 min; $t_R$ (minor) = 9.32 min; $[\alpha]_D$ = -22.1 (c = 0.7 , $CH_2Cl_2$); HRMS: Calcd for $C_{18}H_{27}NO_4$ ($M+H^+$): 322.2013, found: 322.2021.

*Ethyl (2S, 3S)-3-formyl-2-(p-methoxyphenylamino)-undec-5-enoate* (**6**): $^1$H NMR (400 MHz) δ = 0.88 (t, 3H, CH$_2$C$H_3$), 1.2-1.4 (m, 11H), 2.00 (m, 2H), 2.39 (m, 1H), 2.42 (m, 1H), 3.73 (s, 3H, OC$H_3$), 4.17 (m, 2H, OC$H_2$CH$_3$), 4.35 (bs, 1H), 5.42 (m, 1H, CH$_2$C*H*=CH), 5.55 (m. 1H, CH$_2$C*H*=CH), 6.66 (d, 2H, *J* = 7.0 Hz, Ar*H*), 6.78 (d, 2H, *J* = 7.0 Hz, Ar*H*), 9.71 (bs, 1H, CHC*H*O); $^{13}$C NMR (100 MHz): δ = 202.3, 172.3, 153.2, 140.4, 134.6, 125.2, 115.9, 114.7, 61.4, 57.6, 55.5, 53.4, 32.4, 31.3, 28.9, 28.7, 22.4, 14.04, 14.02, 13.9; HPLC (Daicel Chiralpak AS, hexane/i-PrOH = 99:1, flow rate 1.0 mL/min, λ = 254 nm); $t_R$ (major) = 6.69 min; $t_R$ (minor) = 7.90 min; $[\alpha]_D$ = -23.7 (c = 6.6 , $CH_2Cl_2$); HRMS: Calcd for $C_{21}H_{31}NO_4$ ($M+Na^+$): 384.2145, found: 384.2145.

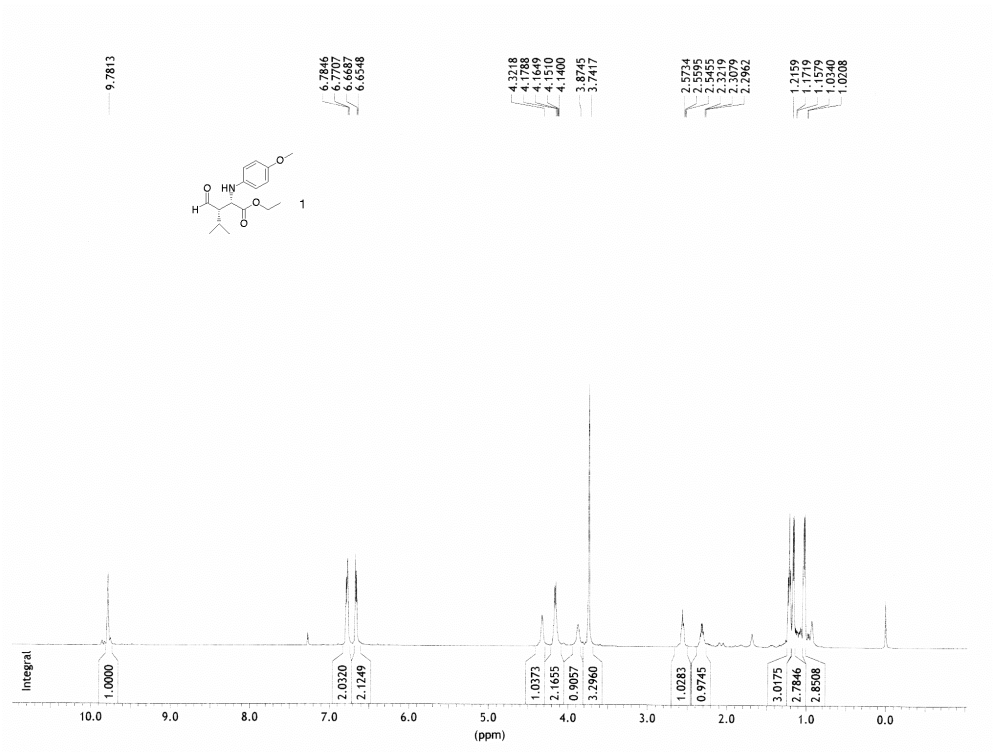
*Ethyl (2S, 3S)-3-formyl-2-(p-methoxyphenylamino)-hept-6-enoate* (**7**):
$^1$H NMR (400 MHz) δ = 1.24 (t, 3H, OCH$_2$C*H*$_3$), 1.2-1.4 (m, 11H), 1.7 (m, 1H), 1.97-2.28 (m, 4H), 2.77 (m, 1H), 3.73 (s, 3H, OC*H*$_3$), 4.17 (m, 2H, OC*H*$_2$CH$_3$), 4.36 (d, *J* = 4.4 Hz, 1H), 5.04 (m, 2H, C*H*=CH$_2$), 5.75 (m. 1H, CH$_2$C*H*=CH$_2$), 6.66 (d, 2H, *J* = 8.8 Hz, Ar*H*), 6.78 (d, 2H, *J* = 8.8 Hz, Ar*H*), 9.71 (bs, 1H, C*H*C*H*O); $^{13}$C NMR (100 MHz): $^{13}$C NMR (100 MHz): δ = 202.4, 172.5, 153.6, 140.6, 137.3, 116.4, 115.0, 61.8, 58.6, 55.9, 53.1, 31.6, 24.5, 14.3;
HPLC (Daicel Chiralpak AS, hexane/i-PrOH = 99:1, flow rate 1.0 mL/min, λ = 254 nm); $t_R$ (major) = 9.94 min; $t_R$ (minor) = 12.86 min; $[\alpha]_D$ = -14.4 (c = 4.3, $CH_2Cl_2$); HRMS: Calcd for $C_{17}H_{23}NO_4$ (M+Na$^+$): 328.1519, found: 328.1525.

*Ethyl (2S, 3S)-3-(methoxycarbonyl)-2-(p-methoxyphenylamino)-4-methyl-pentanoate* (**8**): Crude product **1** (~1 mmol) (*vide supra*) was oxidized with $NaClO_2$ (6 mmol) according to ref. 2 **prior** to purification by chromatography. The corresponding acid was obtained as a slightly red solid after silica gel column chromatography (EtOAc/Hexanes=3:1) and removal of the solvent in vacuo. $^1$H NMR (400 MHz) δ = 0.95 (d, 3H, *J* = 6.6 Hz, CHC*H*$_3$), 1.11 (d, 1H, *J* = 6.6 Hz, CHC*H*$_3$), 1. 21 (t, 3H, OCH$_2$C*H*$_3$), 2.30 (m, 1H), 2.71 (m, 1H), 2.99 (s, 3H, OC*H*$_3$), 4.15 (m, 2H, OC*H*$_2$CH$_3$), 4.28 (bs, 1H), 6.66 (d, 2H, J = 7.0 Hz, Ar*H*), 6.76 (d, 2H, J = 7.0 Hz, Ar*H*); $^{13}$C NMR (100 MHz): δ = 177. 1, 172.7, 153.4, 140.3, 116.2, 115, 61.6, 58.0, 55.9, 54.9, 27.2, 21.3, 19.9, 14.3. The acid was dissolved in $Et_2O$ and esterified with $CH_2N_2$ (3 mmol) according to standard procedures to afford methyl ester **8** as a clear oil: 0.29 g (89%); $^1$H NMR (400 MHz) δ = 0.99 (d, 3H, *J* = 6.6 Hz, CHC*H*$_3$), 1.11 (d, 1H, *J* = 6.6 Hz, CHC*H*$_3$), 1. 22 (t, 3H, OCH$_2$C*H*$_3$), 2.34 (m, 1H), 2.69 (m, 1H), 3.69 (s, 3H, OC*H*$_3$), 3.74 (s, 3H, OC*H*$_3$), 3.92 (bs, 1H, ArN*H*CH), 4.15 (m, 2H, OC*H*$_2$CH$_3$), 4.28 (bs, 1H), 6.66 (d, 2H, J = 7.0 Hz, Ar*H*), 6.76 (d, 2H, J = 7.0 Hz, Ar*H*); $^{13}$C NMR (100 MHz): δ = 173.15, 172.8, 153.2, 140.7, 115.8, 115.1, 61.5, 57.8, 56.0, 55.4, 51.9, 27.4, 21.3, 19.8, 14.4. HPLC (Daicel Chiralpak AS, hexane/i-PrOH = 99:1, flow rate 1.0 mL/min, λ = 254 nm); $t_R$ (major) = 7.00 min; $t_R$ (minor) = 8.98 min; $[\alpha]_D$ = -14.4 (c = 7.4, $CH_2Cl_2$); HRMS: Calcd for $C_{16}H_{25}NO_5$ (M+Na$^+$): 346.1676, found: 346.1633.

*Lactam (3S,4S)*-(**9**):
A solution of LHMDS (0.2 mmol) in THF was slowly added to a cooled (-20 °C) solution of amino acid ester **8** (0.1 mmol) in dry THF (4 mL). After stirring for 30 min at this temperature, the mixture was quenched with 1M HCl solution, extracted with EtOAc, and the combined organic layers were dried ($MgSO_4$), filtered, and the solvent was removed in vacuo to yield lactam **9** after flash column chromatography (hexanes:EtOAc-4:1) as clear crystals: 28 mg (96%); m. p. 83-84 °C (lit. m. p. 84 °C)[3]; $^1$H NMR (400 MHz) δ = 0.93 (d, 3H, *J* = 6.6 Hz, CHC*H*$_3$), 1.21 (d, 1H, *J* = 6.6 Hz, CHC*H*$_3$), 1. 28 (t, 3H, *J* = 7.0 Hz, OCH$_2$C*H*$_3$), 2.12 (m, 1H, C*H*(CH$_3$)$_2$), 3.28 (dd, 1H, $J_{\alpha,3}$ = 10.0 Hz, $J_{2,3}$ = 5.9 Hz, 3-*H*), 3.78 (s, 3H, OC*H*$_3$), 4.25 (m, 2H, OC*H*$_2$CH$_3$), 4.55 (d, 1H, $J_{3,2}$ = 5.9 Hz, 2-H), 6.85 (d, *J* = 8.2 Hz, Ar*H*), 7.22 (d, *J* = 8.2 Hz, Ar*H*); $^{13}$C NMR (100 MHz): δ = 169.3, 165.2, 156.2, 131.0, 117.7, 114.4, 61.7, 60.5, 55.5, 55.4, 26.2, 21.6, 20.5, 14.1; HPLC (Daicel Chiralpak AS, hexane/i-PrOH = 99:1, flow rate 1.0 mL/min, λ = 254 nm); $t_R$ (major) = 4.75 min; $t_R$ (minor) = 7.12 min; $[\alpha]_D$ = -77 (c = 0.7 , $CH_2Cl_2$); HRMS: Calcd for $C_{16}H_{21}NO_4$ (M+H$^+$): 292.1543, found: 292.1535.

*Lactam (3S,4S)*-(**10**):
To a solution of **9** (0.1 mmol) in dioxane:$H_2O$ (2:1, 3mL) was added LiOH (0.8 mmol). The mixture was stirred overnight, quenched with a 1M HCl solution and extracted with EtOAc. The combined organic fractions were dried ($MgSO_4$), filtered and the solvent removed under reduced pressure. The crude acid was dissolved in $Et_2O$ and esterified with $CH_2N_2$ (0.5 mmol) according to standard procedures to afford methyl ester **10** as a white solid: 25 mg (92%); m. p. 143-144 °C (lit. m. p. 143-145 °C)[4]; $^1$H NMR (400 MHz) δ = 0.91 (d, 3H, *J* = 6.6 Hz, CHC*H*$_3$), 1.21 (d, 1H, *J* = 6.6 Hz, CHC*H*$_3$), 2.11 (m, 1H, C*H*(CH$_3$)$_2$), 3.28 (dd, 1H, $J_{\alpha,3}$ = 10.0 Hz, $J_{2,3}$ = 5.9 Hz, 3-*H*), 3.78 (s, 3H, OC*H*$_3$), 3.80 (s, 3H, OCH$_3$), 4.57 (d, 1H, $J_{3,2}$ = 5.9 Hz, 2-H), 6.85 (d, *J* = 8.2 Hz, Ar*H*), 7.22 (d, *J* = 8.2 Hz, Ar*H*); $^{13}$C NMR (100 MHz): δ = 169.9,

165.0, 156.3, 130.8, 117.7, 114.4, 60.6, 55.5, 55.3, 52.4, 26.2, 21.5, 20.5; HPLC (Daicel Chiralpak AS, hexane/i-PrOH = 99:1, flow rate 1.0 mL/min, $\lambda$ = 254 nm); $t_R$ (major) = 4.65 min; $t_R$ (minor) = 6.70 min; $[\alpha]_D$ = - 95 (c = 0.5 , $CH_2Cl_2$) (lit. $[\alpha]_D$ = - 108.7 (c = 1.0 , $CH_2Cl_2$); HRMS: Calcd for $C_{15}H_{19}NO_4$ ($M^+$): 277.1309, found: 277.1314.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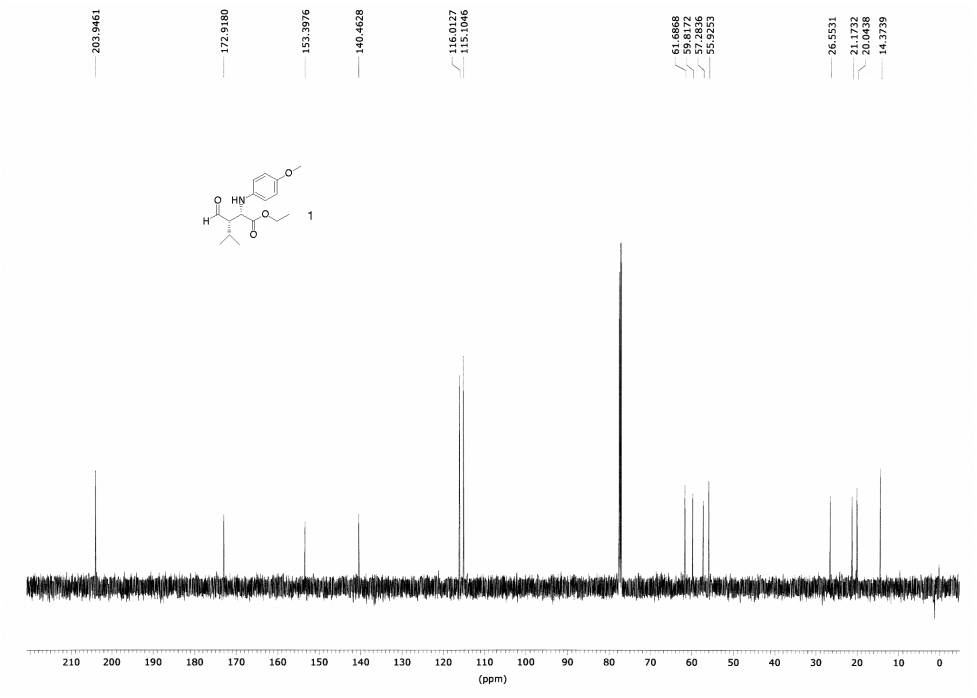
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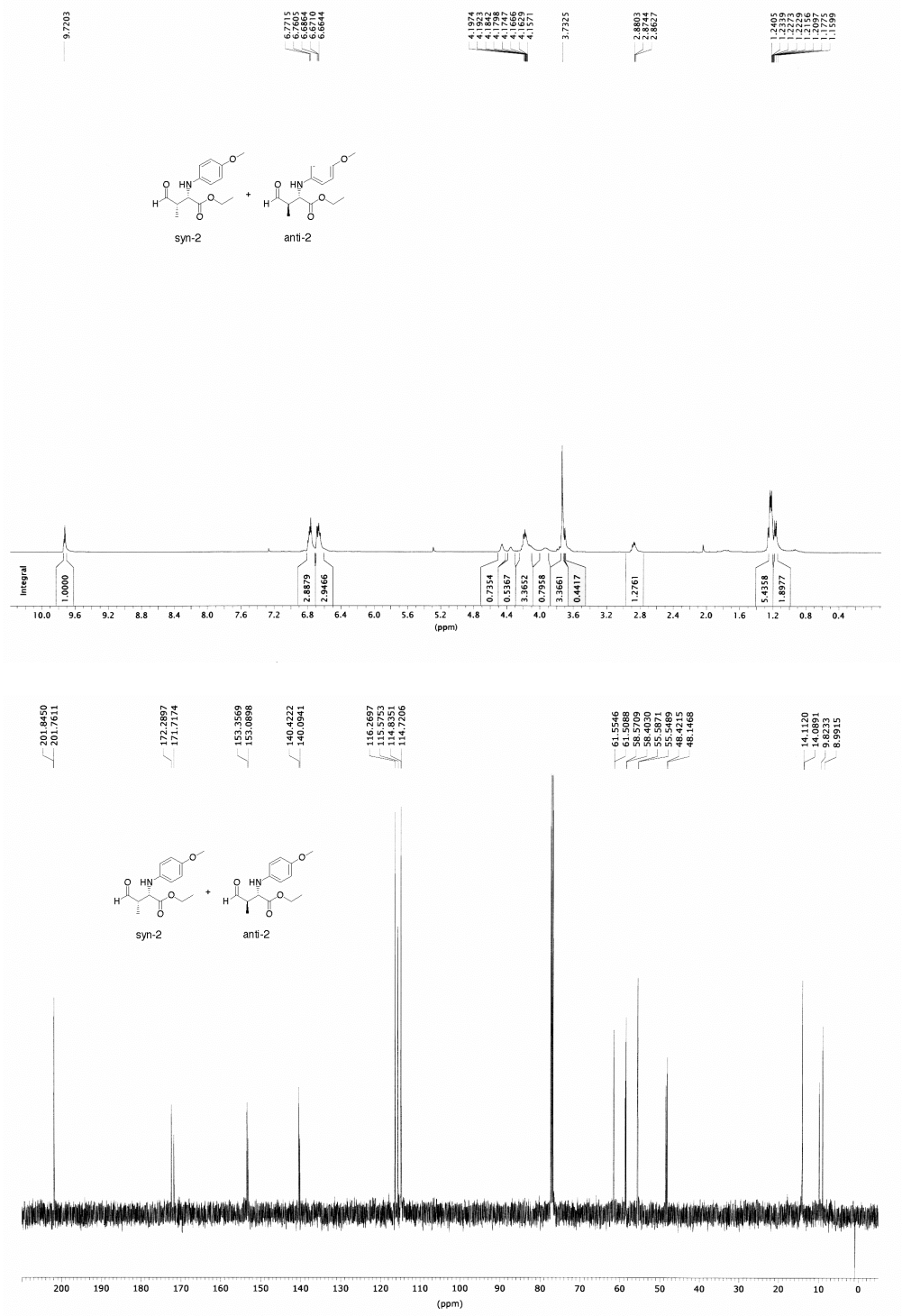
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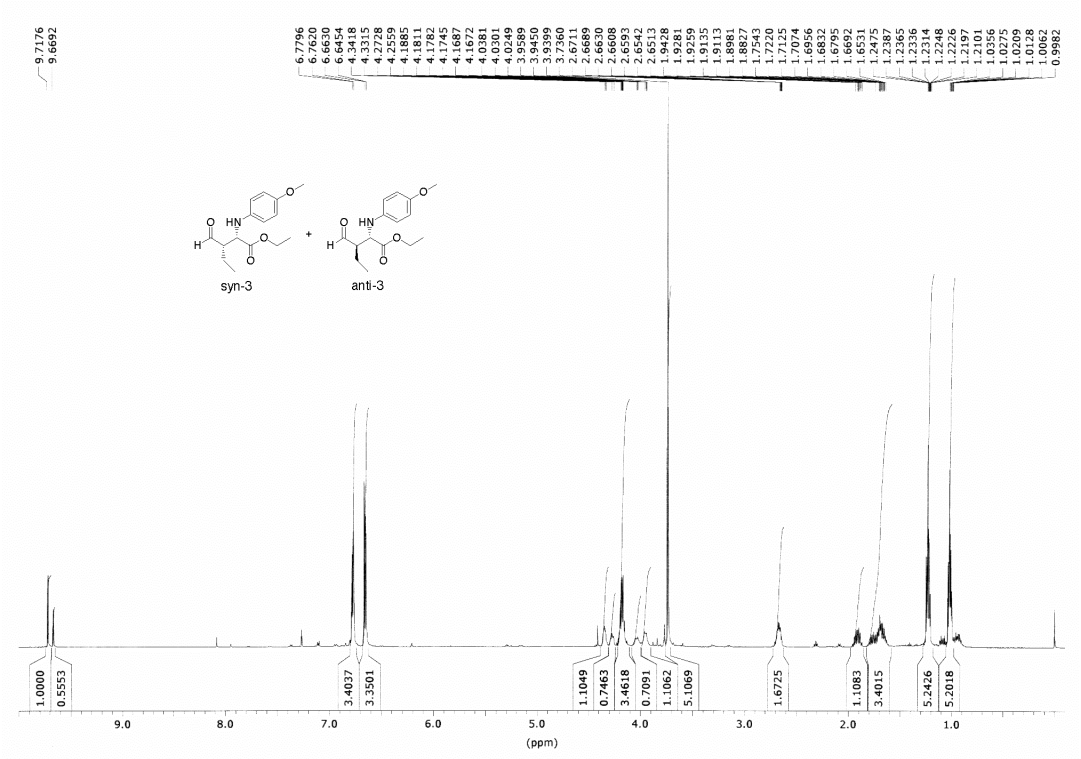
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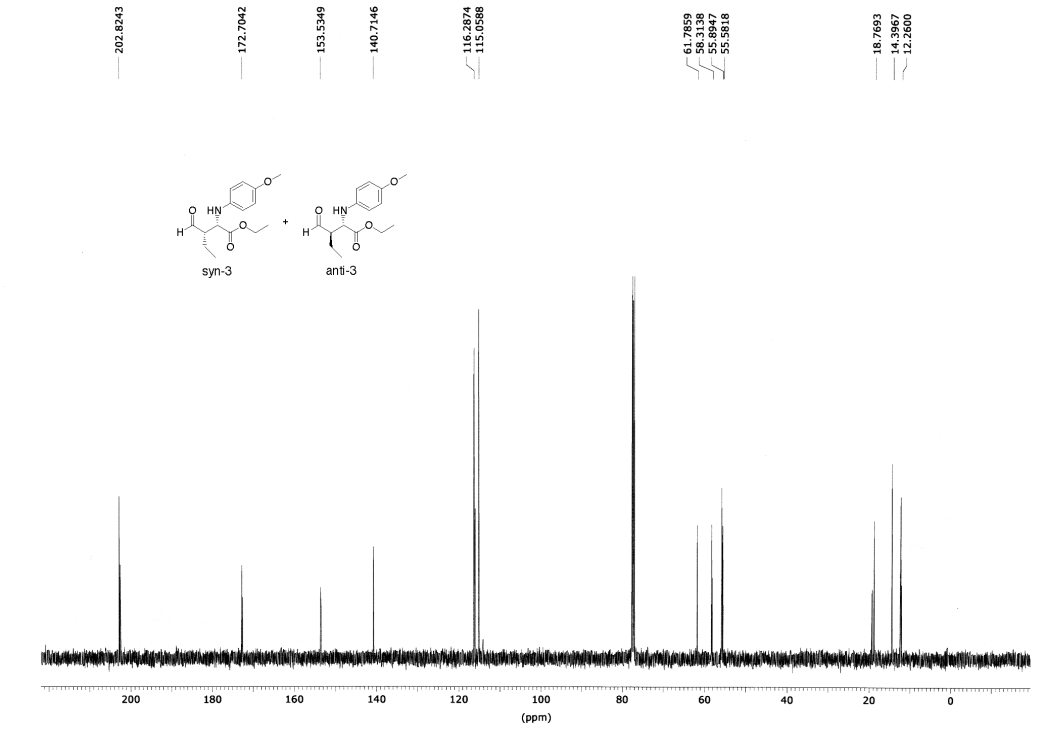
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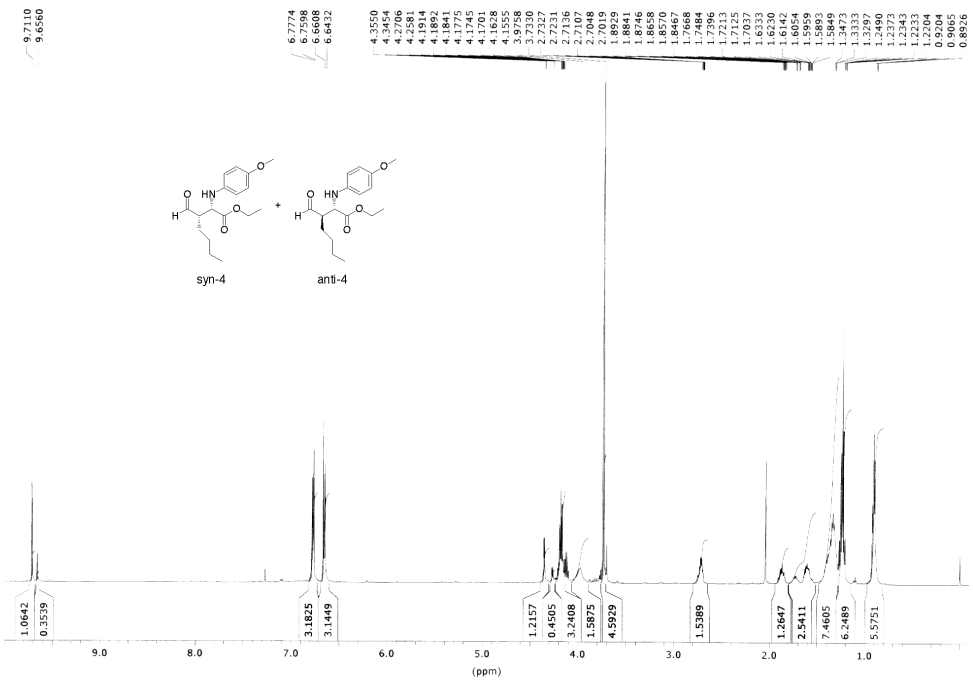
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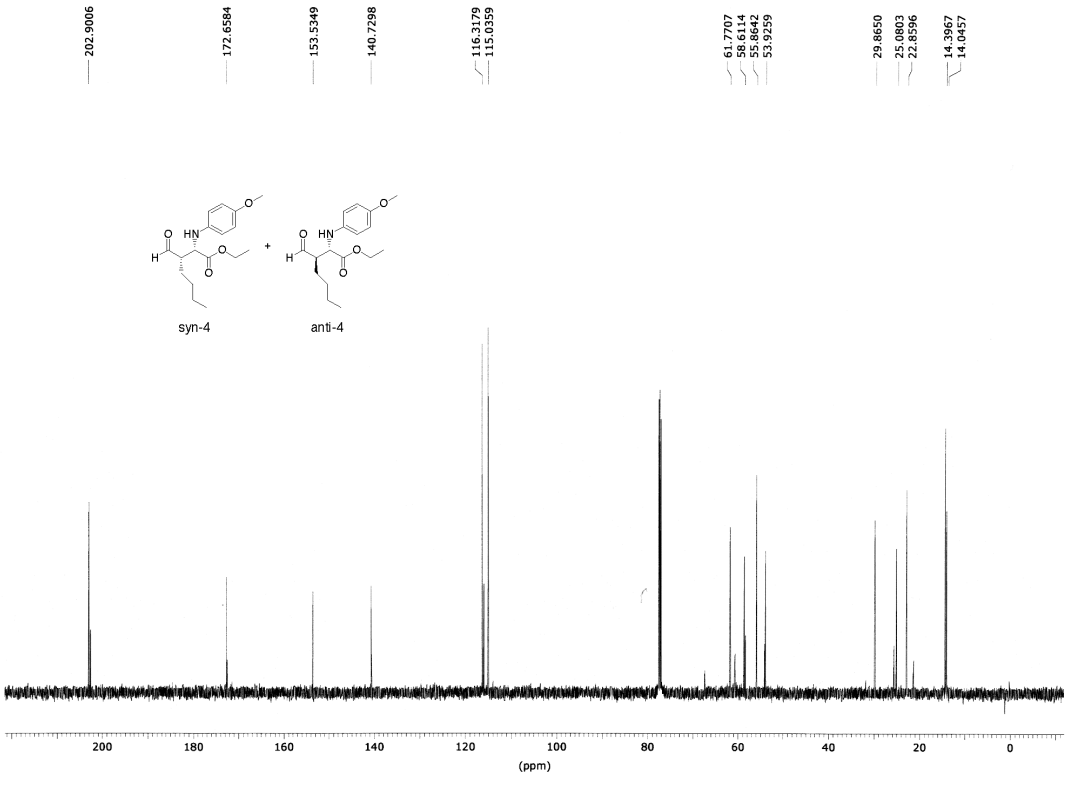
anti-4
1.0642
0.3539
3.1825
3.1449
1.2157
0.4505
3.2408
1.5875
4.5929
1.5389
1.2647
2.5411
7.4605
6.2489
5.5751
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
(ppm)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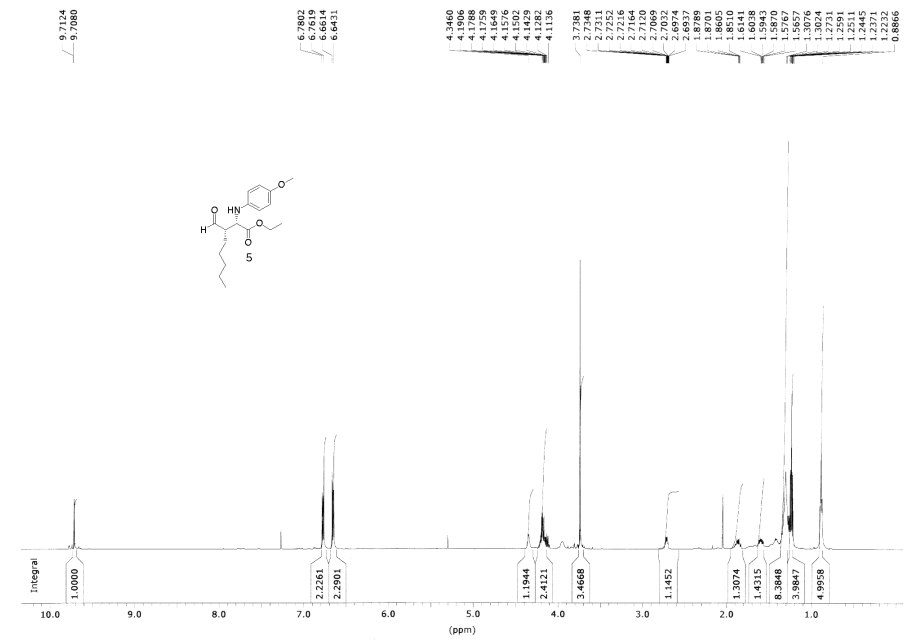
172.6584
153.5349
140.7298
116.3179
115.0359
61.7707
58.6114
55.8642
53.9259
29.8650
25.0803
22.8596
14.3967
14.0457
syn-4
anti-4
200
180
160
140
120
100
80
60
40
20
0
(ppm)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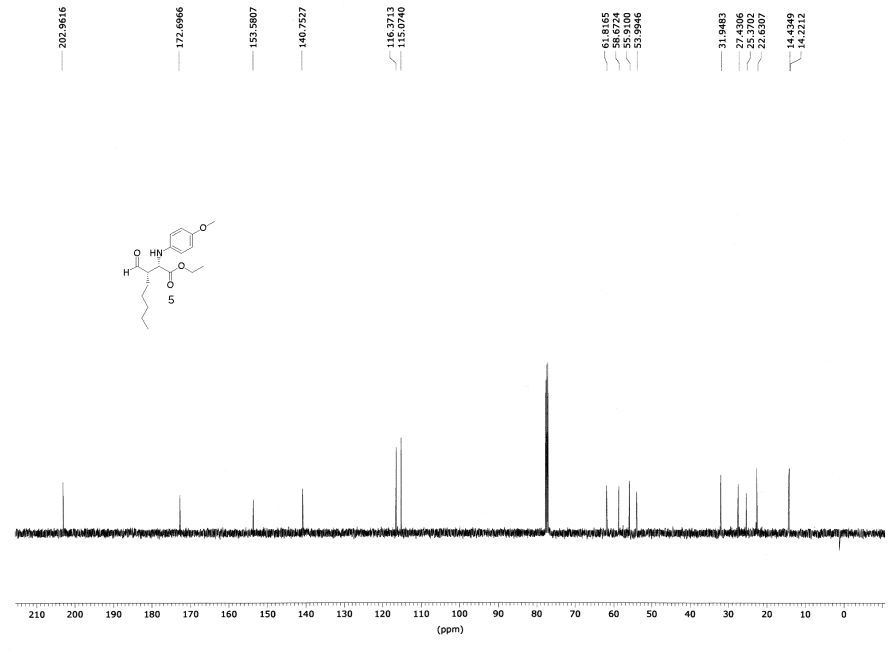
9.7080
6.7802
6.7619
6.6614
6.6431
4.3460
4.1906
4.1788
4.1759
4.1648
4.1576
4.1502
4.1429
4.1388
4.1136
3.7381
2.7348
2.7311
2.7252
2.7216
2.7164
2.7120
2.7069
2.7032
2.6974
2.6937
1.8789
1.8701
1.8655
1.8510
1.6141
1.6038
1.5943
1.5900
1.5767
1.5657
1.3076
1.3024
1.2734
1.2591
1.2511
1.2445
1.2277
1.2232
0.8866
O
HN
H
O
O
5
Integral
1.0000
2.2261
2.2901
1.1944
2.4121
3.4668
1.1452
1.3074
1.4315
8.3848
3.9847
4.9958
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
(ppm)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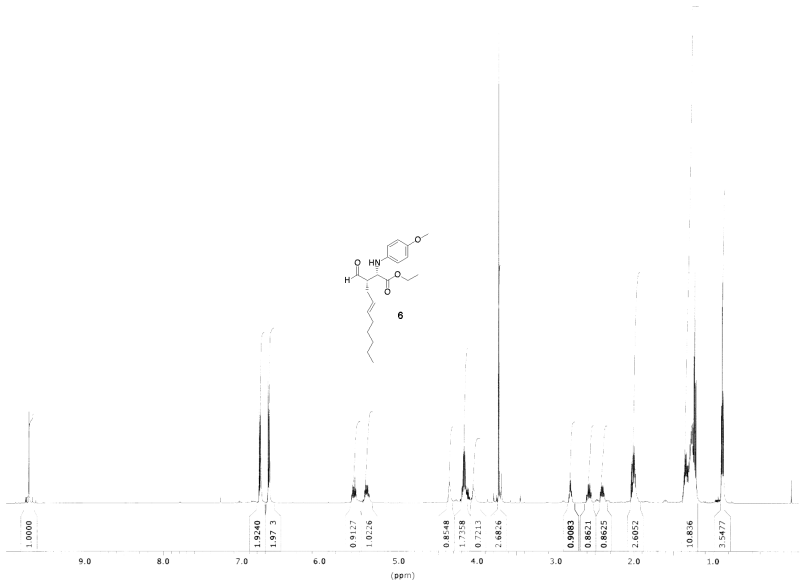
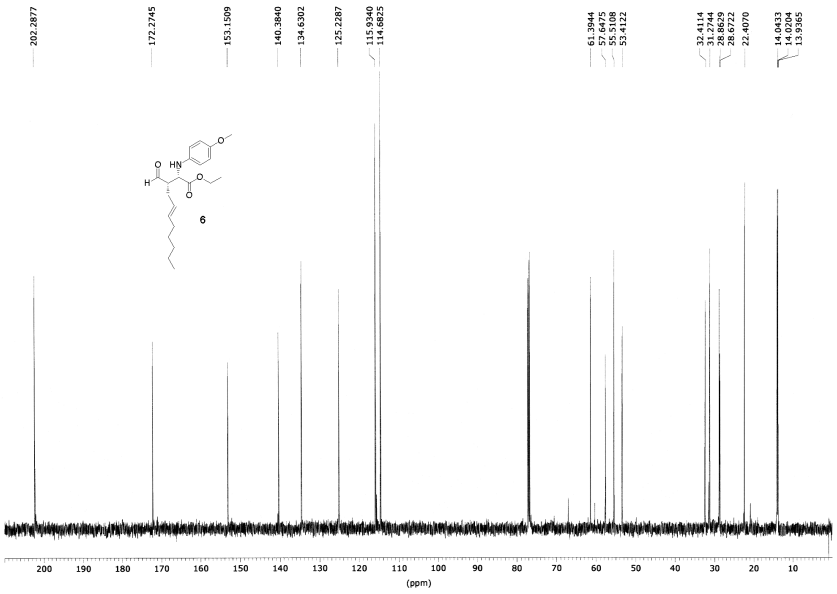
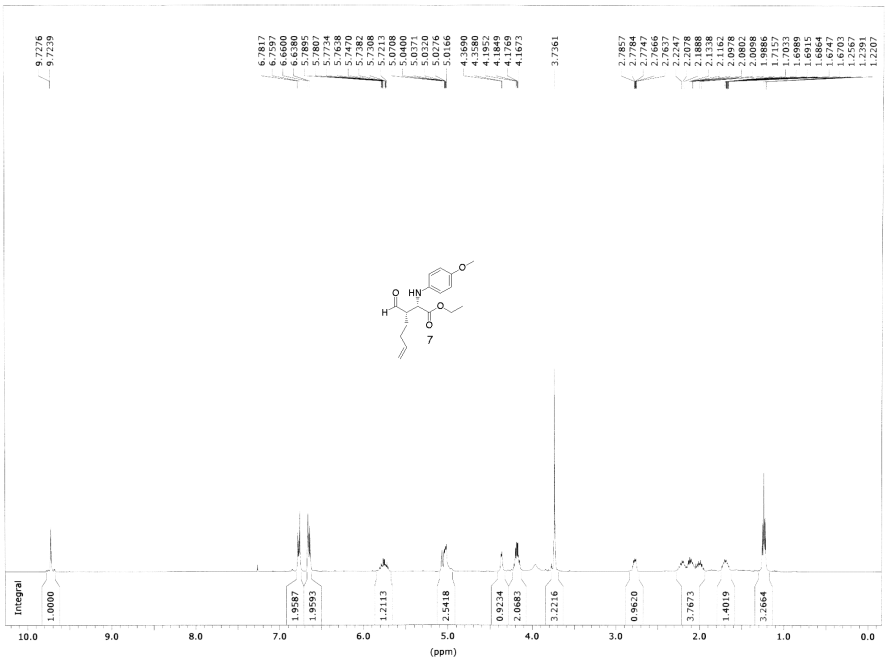
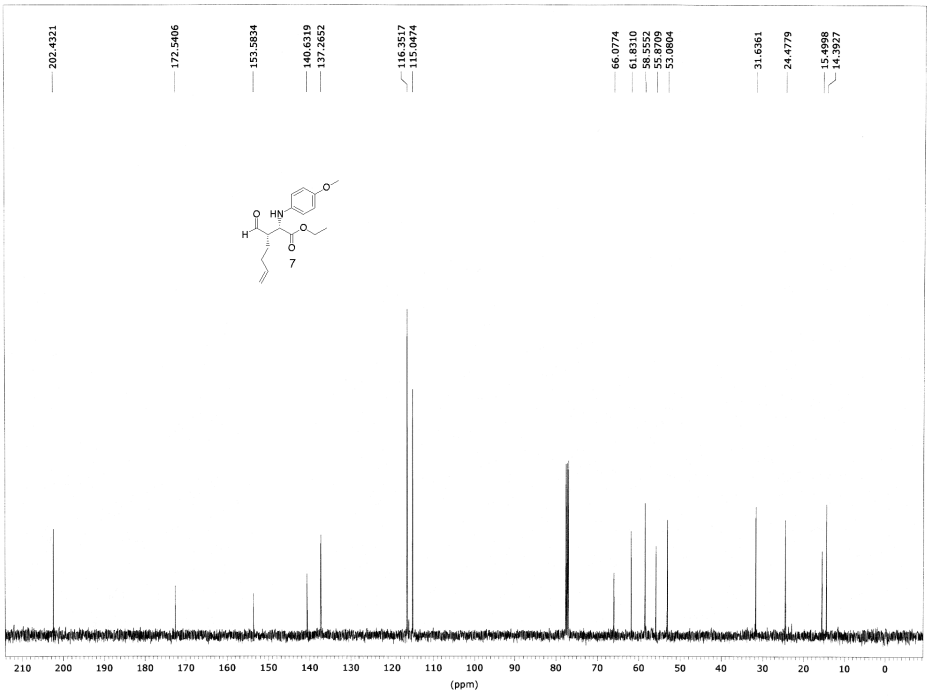
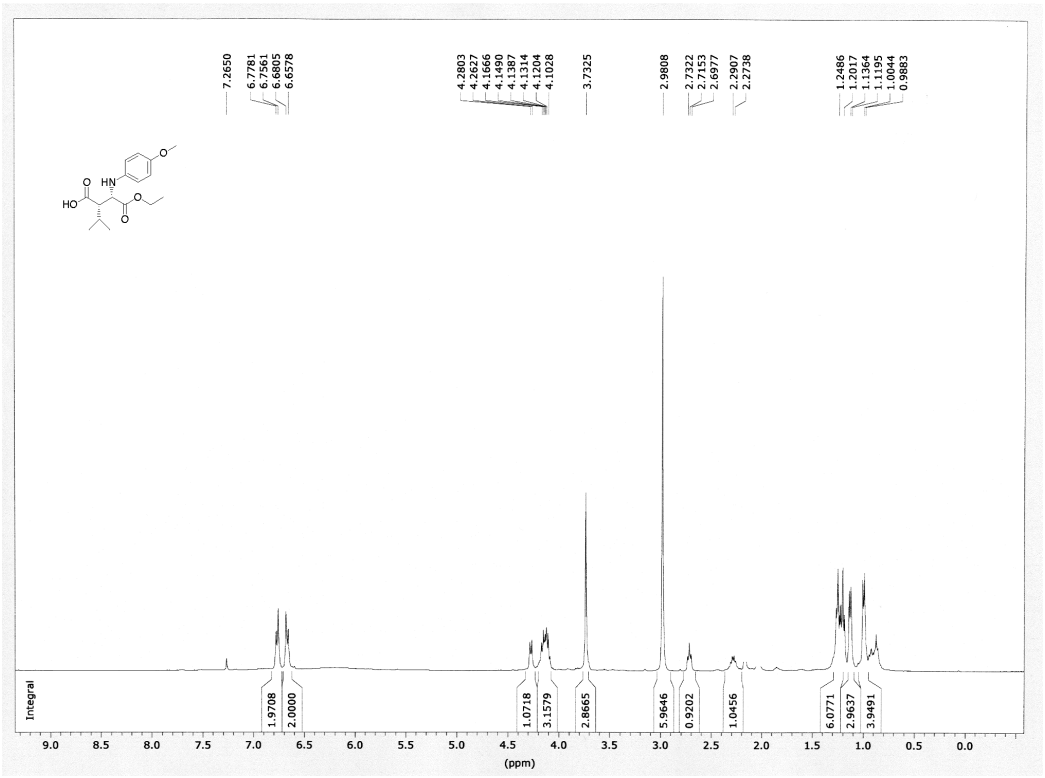
172.6966
153.5807
140.7527
116.3713
115.0740
61.8165
58.6724
55.9100
53.9946
31.9483
27.4306
25.3702
22.6307
14.4349
14.2212
O
HN
H
O
O
5
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
(ppm)

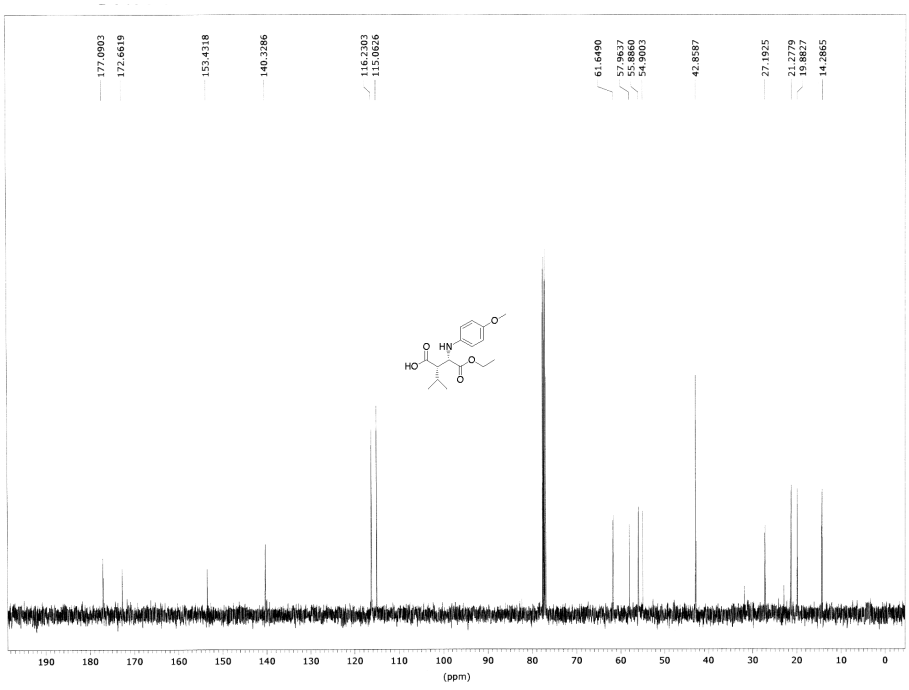

6
1.0000
1.9240
1.97 3
0.9127
1.0226
0.8548
1.7358
0.7213
2.6826
0.9083
0.8621
0.8625
2.6052
10.816
3.5477
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
(ppm)

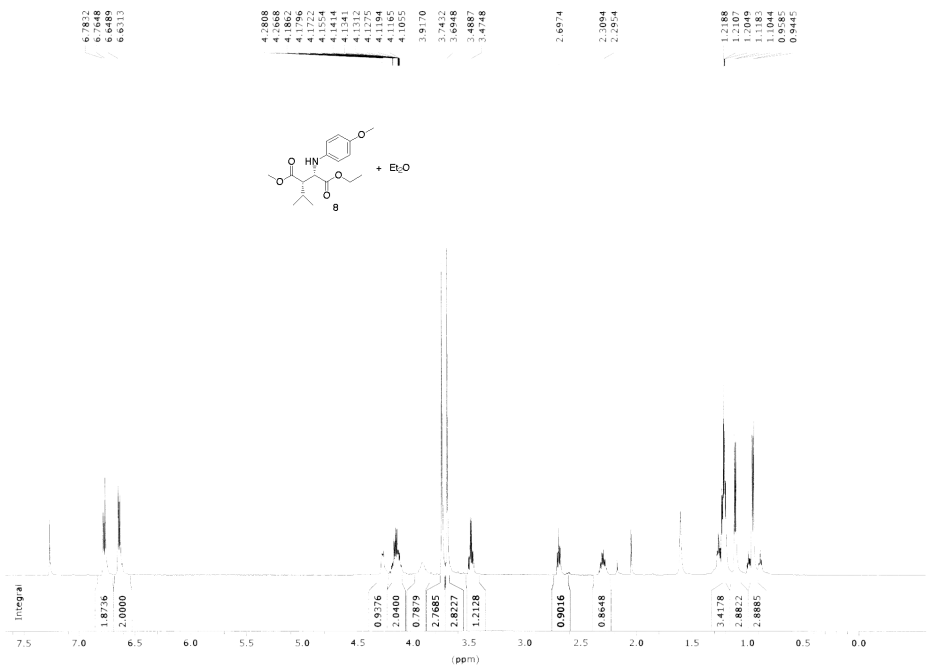

202.2877
172.2745
153.1509
140.3840
134.6302
125.2287
115.9340
114.6825
61.3944
57.6475
55.5108
53.4122
32.4114
31.7244
28.8629
28.6722
22.4070
14.0433
14.0204
13.9365
6
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
(ppm)

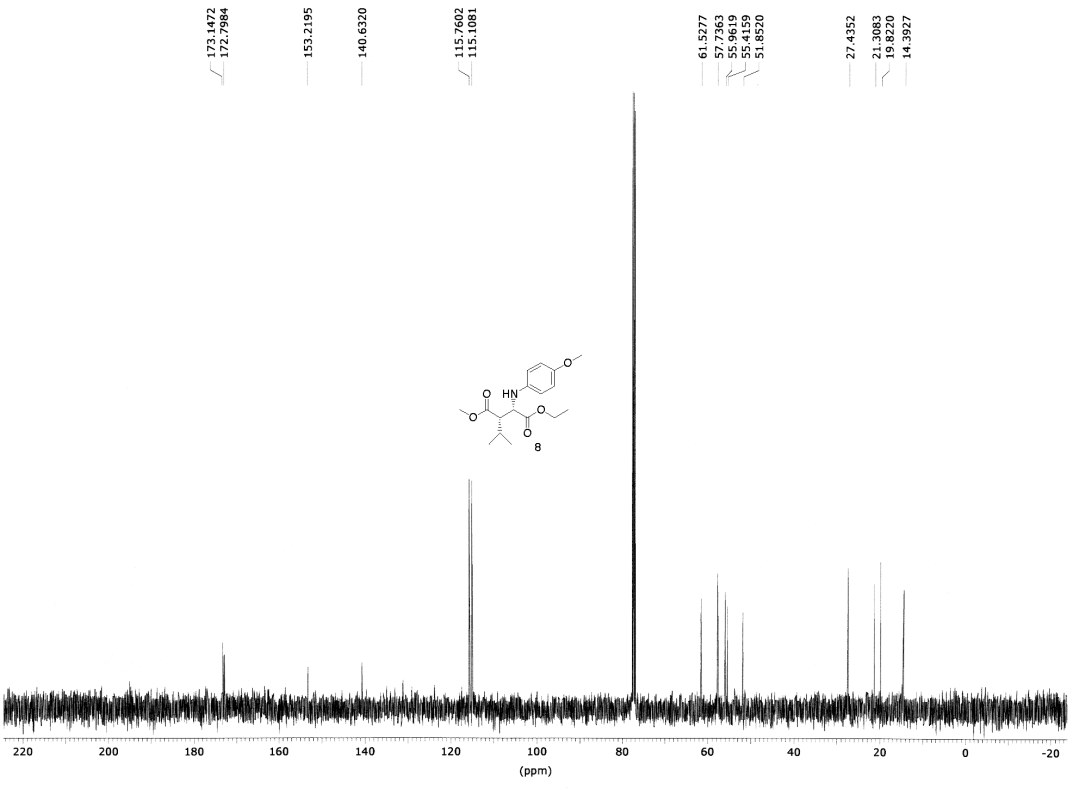

9.7276
9.7239
6.7817
6.7597
6.6600
6.6380
5.7885
5.7807
5.7734
5.7638
5.7470
5.7382
5.7308
5.7213
5.0708
5.0600
5.0371
5.0320
5.0276
5.0166
4.3690
4.3580
4.1952
4.1849
4.1769
4.1673
3.7361
2.7857
2.7784
2.7747
2.7666
2.7637
2.2247
2.2078
2.1888
2.1758
2.1162
2.0978
2.0802
2.0688
1.9886
1.7157
1.7033
1.6989
1.6851
1.6864
1.6747
1.6703
1.2797
1.2391
1.2207
Integral
1.0000
1.9587
1.9593
1.2113
2.5418
0.9234
2.0683
3.2216
0.9620
3.7673
1.4019
3.2664
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
(ppm)
HN
O
H
O
O
7

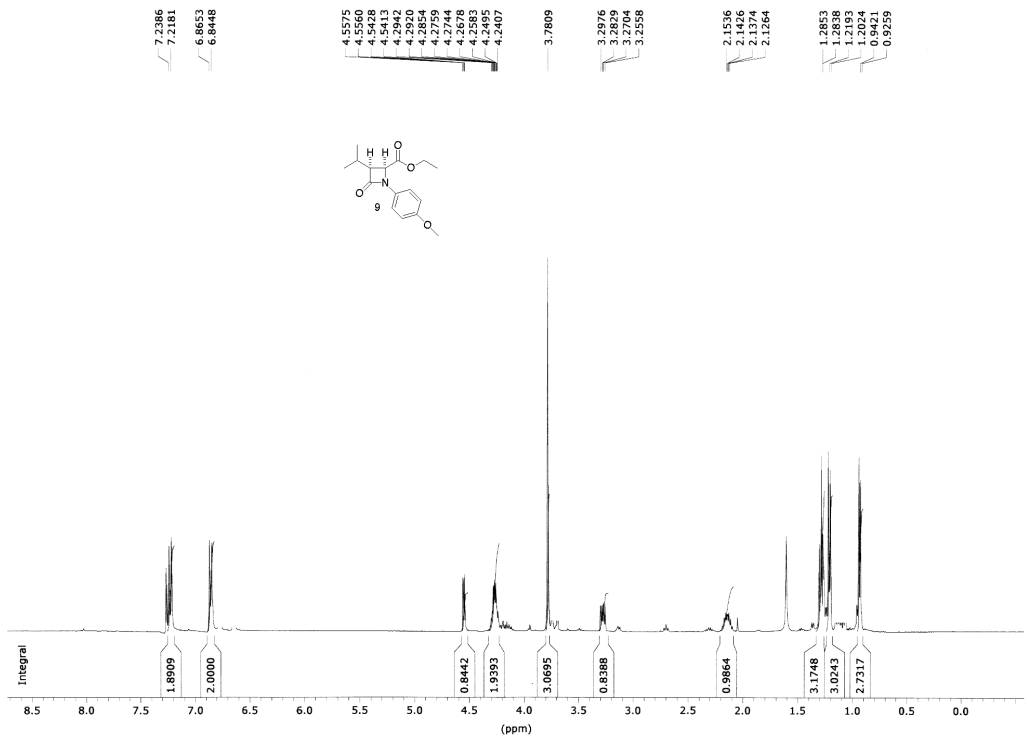

202.4321
172.5406
153.5834
140.6319
137.2652
116.3517
115.0474
66.0774
61.8310
58.5552
55.8709
53.0804
31.6361
24.4779
15.4998
14.3927
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
(ppm)
HN
O
H
O
O
7

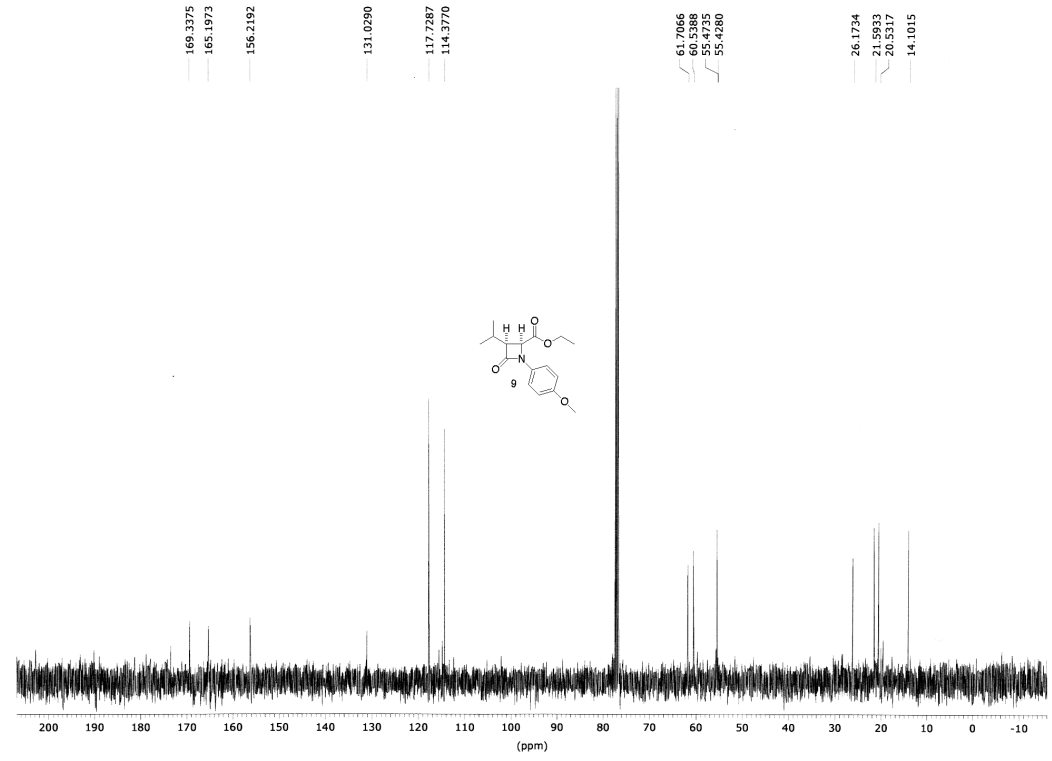

7.2650
6.7781
6.7561
6.6805
6.6578
4.2803
4.2627
4.1666
4.1490
4.1387
4.1314
4.1204
4.1028
3.7325
2.9808
2.7322
2.7153
2.6977
2.2907
2.2738
1.2486
1.2017
1.1364
1.1195
1.0044
0.9883
HO
HN
O
Integral
1.9708
2.0000
1.0718
3.1579
2.8665
5.9646
0.9202
1.0456
6.0771
2.9637
3.9491
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
(ppm)


177.0903
172.6619
153.4318
140.3286
116.2303
115.0626
61.6490
57.9637
55.8860
54.9003
42.8587
27.1925
21.2779
19.8827
14.2865
HO
HN
O
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
(ppm)


6.7832
6.7648
6.6489
6.6313
4.2808
4.2668
4.1962
4.1796
4.1722
4.1564
4.1414
4.1341
4.1312
4.1275
4.1194
4.1165
4.1055
3.9170
3.7432
3.6948
3.4887
3.4748
2.6974
2.3094
2.2954
1.2188
1.2107
1.2049
1.1183
1.1044
0.9585
0.9445
+ Et2O
8
Integral
1.8736
2.0000
0.9376
2.0400
0.7879
2.7685
2.8227
1.2128
0.9016
0.8648
3.4178
2.8822
2.8885
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
(ppm)


173.1472
172.7984
153.2195
140.6320
115.7602
115.1081
61.5277
57.7363
55.9619
55.4159
51.8520
27.4352
21.3083
19.8220
14.3927
8
220
200
180
160
140
120
100
80
60
40
20
0
-20
(ppm)


7.2386
7.2181
6.8653
6.8448
4.5575
4.5560
4.5428
4.5413
4.2942
4.2920
4.2854
4.2759
4.2744
4.2678
4.2583
4.2495
4.2407
3.7809
3.2976
3.2829
3.2704
3.2558
2.1536
2.1426
2.1374
2.1264
1.2853
1.2698
1.2193
1.2024
0.9421
0.9259
9
Integral
1.8909
2.0000
0.8442
1.9393
3.0695
0.8388
0.9864
3.1748
3.0243
2.7317
(ppm)


169.3375
165.1973
156.2192
131.0290
117.7287
114.3770
61.7066
60.5388
55.4735
55.4280
26.1734
21.5933
20.5317
14.1015
9
(ppm)