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Supporting Information

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[Pd(PPh$_3$)$_4$]-Catalyzed Diastereoselective Synthesis of *trans*-1,2-Diazetidines from 2,3-Allenyl Hydrazines and Aryl Halides

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Supporting Information

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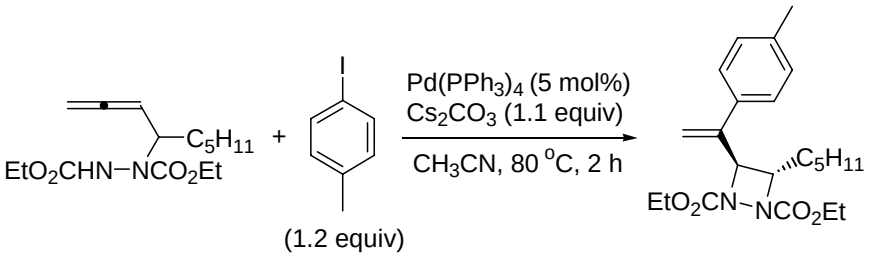
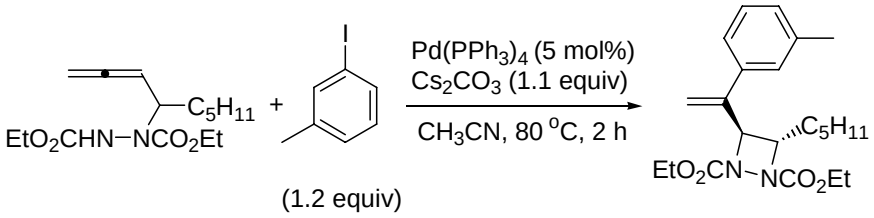
(1) 3-(*n*-Pentyl)-4-(1'-*p*-tolylvinyl)-1,2-bis(ethoxycarbonyl)-1,2-diazetidine (3a) (entry 1, Table 2).



1a **3a**

Under an atmosphere of argon, Cs_2CO_3 (90 mg, 0.28 mmol), 4-iodotoluene (65 mg, 0.30 mmol), $Pd(PPh_3)_4$ (15 mg, 0.013 mmol), **1a** (76 mg, 0.25 mmol), and CH_3CN (3 mL) were added sequentially to an oven-dried Schlenk tube equipped with a stirring bar. The reaction mixture was stirred at 80 °C for 2 h, at which time the reaction was complete as determined by TLC analysis. The resulting mixture was concentrated and the residue was purified by flash chromatography on silica gel (eluent: petroleum ether/ether = 10:1) to afford 7 mg of an unidentified mixture and 76 mg (77%) of **3a**: Liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.23 (d, *J* = 8.3 Hz, 2 H), 7.13 (d, *J* = 8.3 Hz, 2 H), 5.60 (s, 1 H), 5.44 (s, 1 H), 4.81 (d, *J* = 5.2 Hz, 1 H), 4.34-4.17 (m, 4 H), 3.85 (dt, *J* = 5.2 and 6.9 Hz, 1 H), 2.33 (s, 3 H), 1.86-1.62 (m, 2 H), 1.33-1.12 (m, 12 H), 0.81 (t, *J* = 6.8 Hz, 3 H); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 161.1, 160.6, 144.2, 137.9, 134.5, 129.1, 126.3, 113.7, 69.4, 69.1, 62.4, 62.2, 34.5, 31.2, 24.0, 22.3, 21.0, 14.3, 14.2, 13.8; MS (EI) *m/z* (%) 388 (M^+, 23.29), 144 (100); IR (neat) 2932, 1752, 1713, 1626, 1513, 1466, 1322, 1099 cm^{-1}; HRMS (EI) calcd for $C_{22}H_{32}N_2O_4$ (M^+) 388.2362. Found 388.2365.

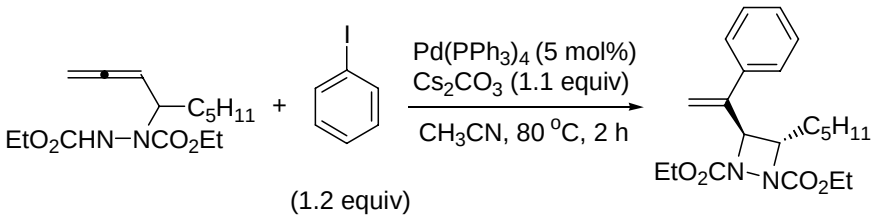
(2) 3-(*n*-Pentyl)-4-(1'-*m*-tolylvinyl)-1,2-bis(ethoxycarbonyl)-1,2-diazetidine (3b) (entry 2, Table 2).



1a **3b**

The reaction of **1a** (75 mg, 0.25 mmol), 3-iodotoluene (65 mg, 0.30 mmol), $Pd(PPh_3)_4$ (15 mg, 0.013 mmol), and Cs_2CO_3 (91 mg, 0.28 mmol) in 3 mL of CH_3CN afforded 69 mg (71%) of **3b**: Liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.22 (t, *J* = 7.5 Hz, 1 H), 7.18-7.08 (m, 3 H), 5.62 (s, 1 H), 5.46 (s, 1 H), 4.81 (d, *J* = 5.1 Hz, 1 H), 4.34-4.16 (m, 4 H), 3.90-3.81 (m, 1 H), 2.34 (s, 3 H), 1.85-1.66 (m, 2 H), 1.35-1.17 (m, 12 H), 0.82 (t, *J* = 6.6 Hz, 3 H); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 161.1, 160.6, 144.6, 138.0, 137.5, 128.8, 128.3, 127.3, 123.6, 114.3, 69.4, 69.2, 62.5, 62.3, 34.5, 31.2, 24.0, 22.4, 21.3, 14.4, 14.3, 13.8; MS (ESI) *m/z* (%) 389 (M^++1), 411 (M^++Na); IR (neat) 2932, 1753, 1713, 1599, 1579, 1466, 1322, 1101 cm^{-1}; HRMS (ESI) calcd. for $C_{22}H_{32}N_2O_4Na$ (M^++Na) 411.2254; Found: 411.2257.

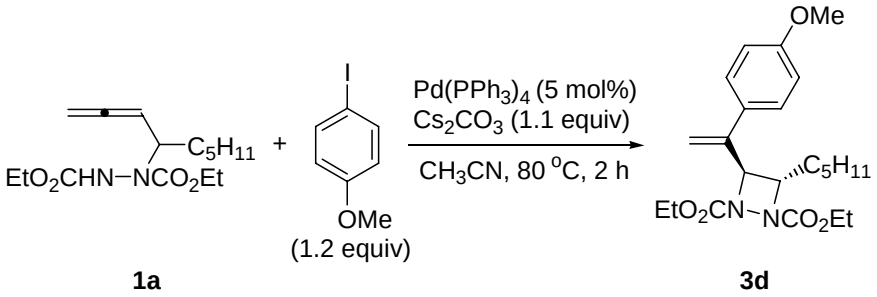
(3) 3-(*n*-Pentyl)-4-(1'-phenylvinyl)-1,2-bis(ethoxycarbonyl)-1,2-diazetidine (3c) (entry 3, Table 2).



1a **3c**

The reaction of **1a** (76 mg, 0.25 mmol), iodobenzene (63 mg, 0.31 mmol), $Pd(PPh_3)_4$ (15 mg, 0.013 mmol), and Cs_2CO_3 (90 mg, 0.28 mmol) in 3 mL of CH_3CN afforded 67 mg (70%) of **3c**: Liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.36-7.25 (m, 5 H), 5.65 (s, 1 H), 5.47 (s, 1 H), 4.81 (d, *J* = 5.3 Hz, 1 H), 4.35-4.15 (m, 4 H), 3.86 (dt, *J* = 5.3 and 7.2 Hz, 1 H), 1.83-1.66 (m, 2 H), 1.35-1.16 (m, 12 H), 0.81 (t, *J* = 6.5 Hz, 3 H); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 161.1, 160.6, 144.6, 137.6, 128.5, 128.1, 126.6, 114.7, 69.4, 69.2, 62.5, 62.3, 34.5, 31.2, 24.0, 22.4, 14.4, 14.3, 13.8; MS (EI) *m/z* (%) 374 (M^+, 9.50), 130 (100); IR (neat) 2932, 1752, 1713, 1466, 1322, 1100 cm^{-1}; HRMS (EI) calcd for $C_{21}H_{30}N_2O_4$ (M^+) 374.2206. Found 374.2197.

(4) 3-(*n*-Pentyl)-4-(1'-(*p*-methoxyphenyl)vinyl)-1,2-bis(ethoxycarbonyl)-1,2-diazetidine (3d) (entry 4, Table 2).

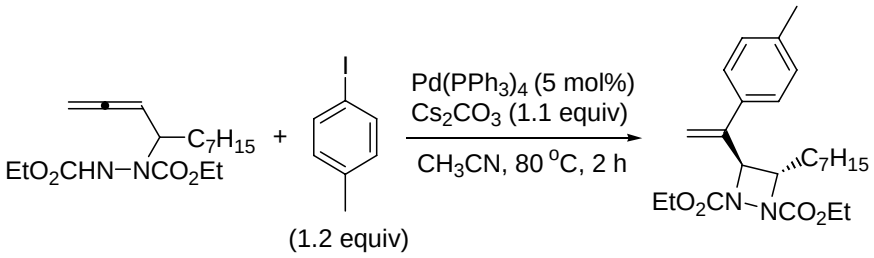


1a **3d**

The reaction of **1a** (75 mg, 0.25 mmol), 4-iodoanisole (71 mg, 0.30 mmol), $Pd(PPh_3)_4$ (15 mg, 0.013 mmol), and Cs_2CO_3 (90 mg, 0.28 mmol) in 3 mL of CH_3CN afforded 74 mg (73%) of **3d**: Liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.27 (d, *J* = 8.7 Hz, 2 H), 6.85 (d, *J* = 8.7 Hz, 2 H), 5.56 (s, 1 H), 5.39 (s, 1 H), 4.78 (d, *J* = 5.3 Hz, 1 H), 4.33-4.15 (m, 4 H), 3.84 (dt, *J* = 5.3 and 6.9 Hz, 1 H), 3.79 (s, 3 H), 1.83-1.66 (m, 2 H), 1.34-1.16 (m, 12 H), 0.81 (t, *J* = 6.6 Hz, 3 H); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ

161.1, 160.6, 159.5, 143.9, 129.9, 127.7, 113.8, 113.2, 69.5, 69.2, 62.5, 62.3, 55.2, 34.5, 31.3, 24.1, 22.4, 14.4, 14.3, 13.8; MS (EI) *m/z* (%) 404 (M^+, 14.73), 160 (100); IR (neat) 2933, 1751, 1712, 1608, 1513, 1465, 1322, 1099 cm^{-1}; HRMS (EI) calcd for $C_{22}H_{32}N_2O_5$ (M^+) 404.2311. Found 404.2321.

(5) 3-(*n*-Heptyl)-4-(1'-*p*-tolylvinyl)-1,2-bis(ethoxycarbonyl)-1,2-diazetidine (3e) (entry 5, Table 2).

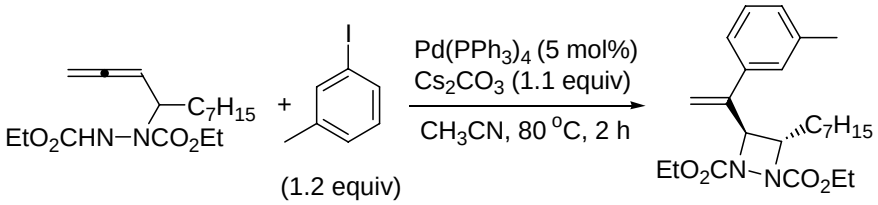


1b **3e**

The reaction of **1b** (66 mg, 0.20 mmol), 4-iodotoluene (52 mg, 0.24 mmol), $Pd(PPh_3)_4$ (12 mg, 0.010 mmol), and Cs_2CO_3 (72 mg, 0.22 mmol) in 2.5 mL of CH_3CN afforded 63 mg (75%) of **3e**: Liquid; ^1H NMR (300 MHz, $CDCl_3$) δ 7.23 (d, *J* = 8.0 Hz, 2 H), 7.14 (d, *J* = 8.0 Hz, 2 H), 5.60 (s, 1 H), 5.44 (s, 1 H), 4.81 (d, *J* = 5.1 Hz, 1 H), 4.35-4.16 (m, 4 H), 3.89-3.81 (m, 1 H), 2.34 (s, 3 H), 1.83-1.67 (m, 2 H), 1.35-1.17 (m, 16 H), 0.85 (t, *J* = 6.8 Hz, 3 H); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 161.1, 160.7, 144.4, 138.0, 134.6, 129.1, 126.4, 113.8, 69.5, 69.2, 62.5, 62.3, 34.6, 31.6, 29.1, 29.0, 24.4, 22.5, 21.1, 14.4, 14.3, 14.0; MS (EI) *m/z* (%) 416 (M^+, 31.08), 144 (100); IR (neat) 2929, 1753, 1713, 1626, 1513, 1466, 1322, 1099 cm^{-1}; HRMS (EI) calcd for $C_{24}H_{36}N_2O_4$ (M^+) 416.2675. Found 416.2689.

(6) 3-(*n*-Heptyl)-4-(1'-*m*-tolylvinyl)-1,2-bis(ethoxycarbonyl)-1,2-diazetidine (3f)

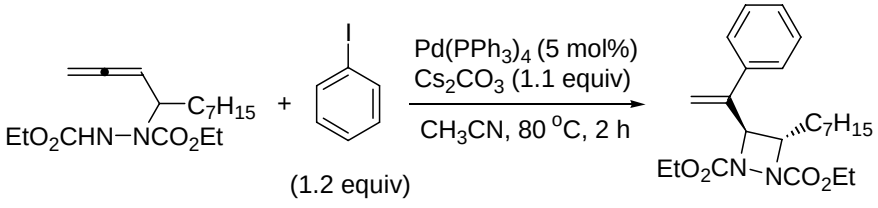
(entry 6, Table 2).



1b **3f**

The reaction of **1b** (82 mg, 0.25 mmol), 3-iodotoluene (67 mg, 0.31 mmol), $Pd(PPh_3)_4$ (15 mg, 0.013 mmol), and Cs_2CO_3 (91 mg, 0.28 mmol) in 3 mL of CH_3CN afforded 73 mg (70%) of **3f**: Liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.22 (t, *J* = 7.7 Hz, 1 H), 7.18-7.08 (m, 3 H), 5.62 (s, 1 H), 5.45 (s, 1 H), 4.81 (d, *J* = 5.5 Hz, 1 H), 4.35-4.16 (m, 4 H), 3.86 (dt, *J* = 5.5 and 6.6 Hz, 1 H), 2.34 (s, 3 H), 1.84-1.66 (m, 2 H), 1.35-1.16 (m, 16 H), 0.85 (t, *J* = 6.8 Hz, 3 H); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 161.1, 160.7, 144.6, 138.1, 137.6, 128.8, 128.3, 127.4, 123.6, 114.4, 69.4, 69.2, 62.5, 62.3, 34.5, 31.6, 29.04, 28.99, 24.4, 22.5, 21.3, 14.4, 14.3, 14.0; MS (ESI) *m/z* (%) 417 (M^++1), 439 (M^++Na); IR (neat) 2929, 1753, 1713, 1599, 1579, 1466, 1322, 1102 cm^{-1}; HRMS (ESI) calcd. for $C_{24}H_{36}N_2O_4Na$ (M^++Na) 439.2567; Found: 439.2566.

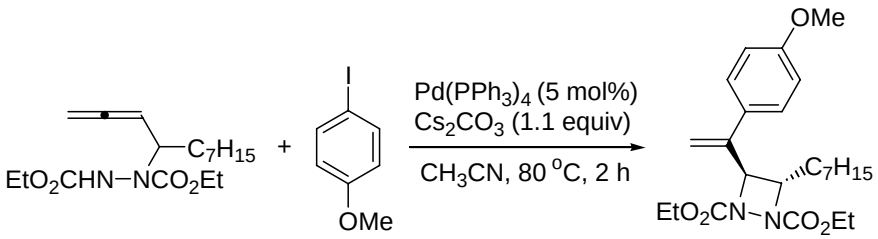
(7) 3-(*n*-Heptyl)-4-(1'-phenylvinyl)-1,2-bis(ethoxycarbonyl)-1,2-diazetidine (3g) (entry 7, Table 2).



1b **3g**

The reaction of **1b** (82 mg, 0.25 mmol), iodobenzene (63 mg, 0.31 mmol), $Pd(PPh_3)_4$ (15 mg, 0.013 mmol), and Cs_2CO_3 (90 mg, 0.28 mmol) in 3 mL of CH_3CN afforded 64 mg (63%) of **3g**: Liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.36-7.25 (m, 5 H), 5.65 (s, 1 H), 5.47 (s, 1 H), 4.81 (d, *J* = 5.3 Hz, 1 H), 4.35-4.15 (m, 4 H), 3.86 (dt, *J* = 5.3 and 6.9 Hz, 1 H), 1.83-1.66 (m, 2 H), 1.35-1.15 (m, 16 H), 0.85 (t, *J* = 6.8 Hz, 3 H); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 161.1, 160.6, 144.6, 137.6, 128.5, 128.1, 126.6, 114.7, 69.4, 69.2, 62.6, 62.3, 34.6, 31.6, 29.1, 29.0, 24.4, 22.5, 14.4, 14.3, 14.0; MS (ESI) *m/z* (%) 403 (M^++1), 425 (M^++Na); IR (neat) 2929, 1753, 1713, 1466, 1322, 1101 cm^{-1}; HRMS (ESI) calcd. for $C_{23}H_{34}N_2O_4Na$ (M^++Na) 425.2411; Found: 425.2416.

(8) 3-(*n*-Heptyl)-4-(1'-(*p*-methoxyphenyl)vinyl)-1,2-bis(ethoxycarbonyl)-1,2-diazetidine (3h) (entry 8, Table 2).

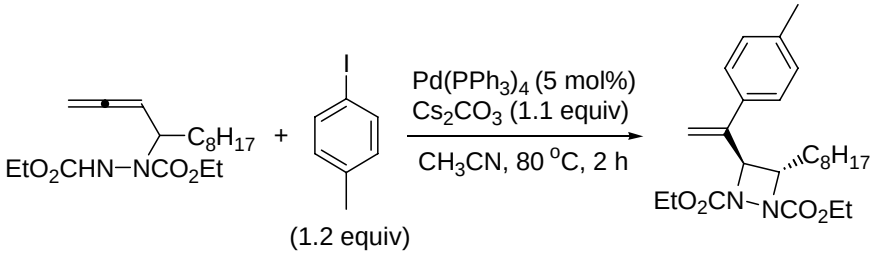


1b **3h**

The reaction of **1b** (66 mg, 0.20 mmol), 4-iodoanisole (56 mg, 0.24 mmol), $Pd(PPh_3)_4$ (12 mg, 0.010 mmol), and Cs_2CO_3 (72 mg, 0.22 mmol) in 2.5 mL of CH_3CN afforded 66 mg (75%) of **3h**: Liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.27 (d, *J* = 8.6 Hz, 2 H), 6.86 (d, *J* = 8.6 Hz, 2 H), 5.56 (s, 1 H), 5.39 (s, 1 H), 4.78 (d, *J* = 5.2 Hz, 1 H), 4.33-4.15 (m, 4 H), 3.84 (dt, *J* = 5.2 and 7.2 Hz, 1 H), 3.79 (s, 3 H),

1.83-1.66 (m, 2 H), 1.34-1.16 (m, 16 H), 0.84 (t, J = 6.8 Hz, 3 H); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 161.1, 160.7, 159.5, 143.9, 129.9, 127.7, 113.8, 113.2, 69.5, 69.2, 62.5, 62.3, 55.2, 34.6, 31.6, 29.1, 29.0, 24.4, 22.5, 14.4, 14.3, 14.0; MS (ESI) *m/z* (%) 433 (M^++1); IR (neat) 2930, 1752, 1713, 1608, 1513, 1465, 1322, 1100 cm^{-1}; HRMS (MALDI/DHB) calcd. for $C_{24}H_{36}N_2O_5Na$ (M^++Na) 455.2516; Found: 455.2512.

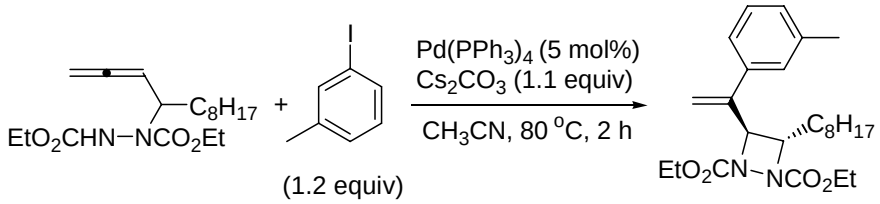
(9) 3-(*n*-Octyl)-4-(1'-*p*-tolylvinyl)-1,2-bis(ethoxycarbonyl)-1,2-diazetidine (3i) (entry 9, Table 2).



1c **3i**

The reaction of **1c** (69 mg, 0.20 mmol), 4-iodotoluene (52 mg, 0.24 mmol), $Pd(PPh_3)_4$ (12 mg, 0.010 mmol), and Cs_2CO_3 (73 mg, 0.22 mmol) in 2.5 mL of CH_3CN afforded 63 mg (72%) of **3i**: Liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.23 (d, J = 7.8 Hz, 2 H), 7.14 (d, J = 7.8 Hz, 2 H), 5.61 (s, 1 H), 5.44 (s, 1 H), 4.81 (d, J = 5.3 Hz, 1 H), 4.35-4.16 (m, 4 H), 3.85 (dt, J = 5.3 and 7.2 Hz, 1 H), 2.34 (s, 3 H), 1.83-1.67 (m, 2 H), 1.35-1.17 (m, 18 H), 0.86 (t, J = 6.8 Hz, 3 H); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 161.2, 160.7, 144.4, 138.0, 134.6, 129.2, 126.4, 113.8, 69.5, 69.2, 62.5, 62.3, 34.6, 31.8, 29.3, 29.11, 29.06, 24.4, 22.6, 21.1, 14.4, 14.3, 14.0; MS (EI) *m/z* (%) 430 (M^+, 25.35), 144 (100); IR (neat) 2927, 1753, 1713, 1626, 1513, 1466, 1322, 1099 cm^{-1}; HRMS (EI) calcd for $C_{25}H_{38}N_2O_4$ (M^+) 430.2832. Found 430.2828.

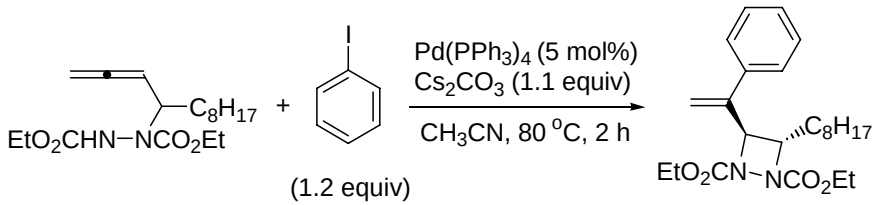
(10) 3-(*n*-Octyl)-4-(1'-*m*-tolylvinyl)-1,2-bis(ethoxycarbonyl)-1,2-diazetidine (3j) (entry 10, Table 2).



1c **3j**

The reaction of **1c** (68 mg, 0.20 mmol), 3-iodotoluene (54 mg, 0.25 mmol), $Pd(PPh_3)_4$ (12 mg, 0.010 mmol), and Cs_2CO_3 (73 mg, 0.22 mmol) in 2.5 mL of CH_3CN afforded 61 mg (71%) of **3j**: Liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.22 (t, *J* = 7.7 Hz, 1 H), 7.18-7.08 (m, 3 H), 5.62 (s, 1 H), 5.46 (s, 1 H), 4.80 (d, *J* = 4.8 Hz, 1 H), 4.35-4.16 (m, 4 H), 3.90-3.81 (m, 1 H), 2.35 (s, 3 H), 1.84-1.66 (m, 2 H), 1.35-1.16 (m, 18 H), 0.86 (t, *J* = 6.8 Hz, 3 H); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 161.1, 160.7, 144.7, 138.1, 137.6, 128.9, 128.3, 127.4, 123.6, 114.4, 69.4, 69.3, 62.5, 62.3, 34.6, 31.7, 29.3, 29.11, 29.05, 24.4, 22.5, 21.4, 14.4, 14.3, 14.0; MS (ESI) *m/z* (%) 431 (M^++1), 453 (M^++Na); IR (neat) 2927, 1753, 1713, 1599, 1576, 1466, 1322, 1101 cm^{-1}; HRMS (ESI) calcd. for $C_{25}H_{38}N_2O_4Na$ (M^++Na) 453.2724; Found: 453.2723.

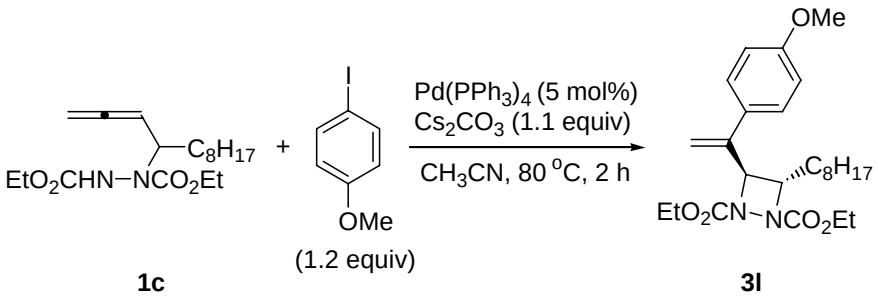
(11) 3-(*n*-Octyl)-4-(1'-phenylvinyl)-1,2-bis(ethoxycarbonyl)-1,2-diazetidine (3k) (entry 11, Table 2).



1c **3k**

The reaction of **1c** (68 mg, 0.20 mmol), iodobenzene (50 mg, 0.25 mmol), $Pd(PPh_3)_4$ (12 mg, 0.010 mmol), and Cs_2CO_3 (72 mg, 0.22 mmol) in 2.5 mL of CH_3CN afforded 55 mg (66%) of **3k**: Liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.36-7.25 (m, 5 H), 5.65 (s, 1 H), 5.47 (s, 1 H), 4.82 (d, *J* = 5.4 Hz, 1 H), 4.36-4.15 (m, 4 H), 3.86 (dt, *J* = 5.4 and 6.9 Hz, 1 H), 1.84-1.66 (m, 2 H), 1.35-1.15 (m, 18 H), 0.86 (t, *J* = 6.6 Hz, 3 H); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 161.1, 160.6, 144.6, 137.6, 128.5, 128.1, 126.6, 114.7, 69.4, 69.2, 62.6, 62.3, 34.6, 31.7, 29.3, 29.11, 29.06, 24.4, 22.6, 14.4, 14.3, 14.0; MS (ESI) *m/z* (%) 417 (M^++1), 439 (M^++Na); IR (neat) 2928, 1753, 1713, 1466, 1322, 1100 cm^{-1}; HRMS (ESI) calcd. for $C_{24}H_{36}N_2O_4Na$ (M^++Na) 439.2567; Found: 439.2562.

(12) 3-(*n*-Octyl)-4-(1'-(*p*-methoxyphenyl)vinyl)-1,2-bis(ethoxycarbonyl)-1,2-diazetidine (3l) (entry 12, Table 2).

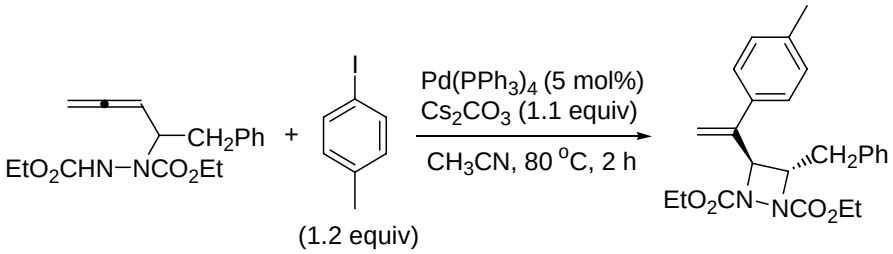


1c **3l**

The reaction of **1c** (69 mg, 0.20 mmol), 4-iodoanisole (57 mg, 0.24 mmol), $Pd(PPh_3)_4$ (12 mg, 0.010 mmol), and Cs_2CO_3 (72 mg, 0.22 mmol) in 2.5 mL of CH_3CN afforded 67 mg (74%) of **3l**: Liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.27 (d, *J* = 9.0 Hz, 2 H), 6.86 (d, *J* = 9.0 Hz, 2 H), 5.56 (s, 1 H), 5.39 (s, 1 H), 4.78 (d, *J* = 5.3 Hz, 1 H), 4.34-4.15 (m, 4 H), 3.84 (dt, *J* = 5.3 and 6.9 Hz, 1 H), 3.79 (s, 3 H),

1.83-1.65 (m, 2 H), 1.34-1.15 (m, 18 H), 0.85 (t, J = 6.8 Hz, 3 H); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 161.1, 160.7, 159.5, 143.9, 129.9, 127.7, 113.8, 113.2, 69.5, 69.2, 62.5, 62.3, 55.2, 34.6, 31.7, 29.3, 29.12, 29.07, 24.4, 22.5, 14.4, 14.3, 14.0; MS (ESI) *m/z* (%) 447 (M^++1), 469 (M^++Na); IR (neat) 2928, 1752, 1713, 1608, 1513, 1465, 1322, 1100 cm^{-1}; HRMS (MALDI/DHB) calcd. for $C_{25}H_{38}N_2O_5Na$ (M^++Na) 469.2673; Found: 469.2667.

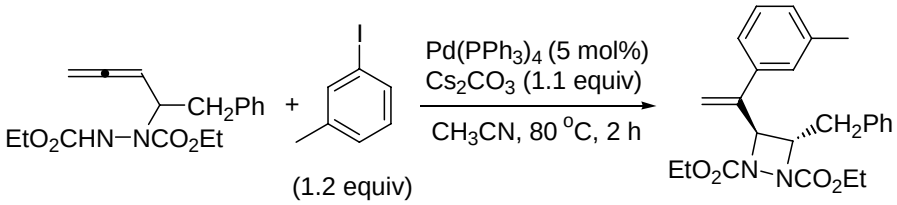
(13) 3-Benzyl-4-(1'-*p*-tolylvinyl)-1,2-bis(ethoxycarbonyl)-1,2-diazetidine (3m) (entry 13, Table 2).



1d **3m**

The reaction of **1d** (81 mg, 0.25 mmol), 4-iodotoluene (66 mg, 0.30 mmol), $Pd(PPh_3)_4$ (15 mg, 0.013 mmol), and Cs_2CO_3 (91 mg, 0.28 mmol) in 3 mL of CH_3CN afforded 71 mg (68%) of **3m**: Liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.29-7.19 (m, 5 H), 7.17-7.07 (m, 4 H), 5.55 (s, 1 H), 5.42 (s, 1 H), 4.78 (d, J = 5.1 Hz, 1 H), 4.25-4.02 (m, 5 H), 3.10 (dd, J = 5.2 and 14.4 Hz, 1 H), 2.94 (dd, J = 5.2 and 14.4 Hz, 1 H), 2.35 (s, 3 H), 1.23 (t, J = 7.1 Hz, 3 H), 1.21 (t, J = 7.1 Hz, 3 H); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 160.8, 160.2, 144.2, 137.8, 135.1, 134.4, 129.8, 129.1, 128.3, 126.9, 126.3, 113.8, 69.2, 67.2, 62.4, 62.3, 39.3, 21.1, 14.34, 14.27; MS (ESI) *m/z* (%) 409 (M^++1); IR (neat) 2981, 1751, 1713, 1626, 1513, 1465, 1322, 1099 cm^{-1}; HRMS (ESI) calcd for $C_{24}H_{29}N_2O_4$ (M^++1) 409.2122. Found 409.2108.

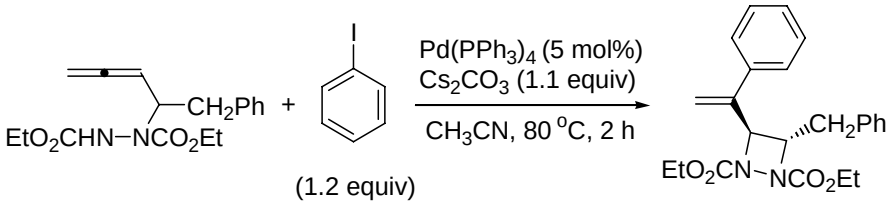
(14) 3-Benzyl-4-(1'-*m*-tolylvinyl)-1,2-bis(ethoxycarbonyl)-1,2-diazetidine (3n) (entry 14, Table 2).



1d **3n**

The reaction of **1d** (81 mg, 0.25 mmol), 3-iodotoluene (67 mg, 0.31 mmol), $Pd(PPh_3)_4$ (15 mg, 0.013 mmol), and Cs_2CO_3 (91 mg, 0.28 mmol) in 3 mL of CH_3CN afforded 71 mg (68%) of **3n**: Liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.30-7.15 (m, 6 H), 7.14-7.00 (m, 3 H), 5.57 (s, 1 H), 5.43 (s, 1 H), 4.77 (d, *J* = 4.8 Hz, 1 H), 4.25-4.02 (m, 5 H), 3.10 (dd, *J* = 5.3 and 14.4 Hz, 1 H), 2.93 (dd, *J* = 5.3 and 14.4 Hz, 1 H), 2.35 (s, 3 H), 1.23 (t, *J* = 7.1 Hz, 3 H), 1.22 (t, *J* = 7.2 Hz, 3 H); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 160.8, 160.2, 144.4, 138.0, 137.3, 135.1, 129.8, 128.8, 128.3, 128.2, 127.2, 126.9, 123.4, 114.4, 69.1, 67.2, 62.4, 62.3, 39.3, 21.4, 14.34, 14.27; MS (ESI) *m/z* (%) 409 (M^++1), 431 (M^++Na); IR (neat) 2981, 1752, 1713, 1599, 1579, 1465, 1321, 1103 cm^{-1}; HRMS (ESI) calcd. for $C_{24}H_{28}N_2O_4Na$ (M^++Na) 431.1941; Found: 431.1939.

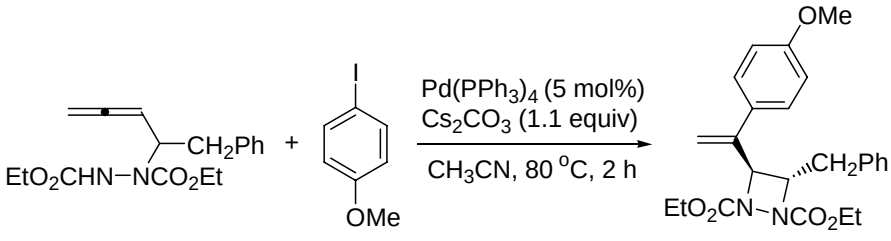
(15) 3-Benzyl-4-(1'-phenylvinyl)-1,2-bis(ethoxycarbonyl)-1,2-diazetidine (3o) (entry 15, Table 2).



1d **3o**

The reaction of **1d** (81 mg, 0.25 mmol), iodobenzene (63 mg, 0.31 mmol), $Pd(PPh_3)_4$ (15 mg, 0.013 mmol), and Cs_2CO_3 (91 mg, 0.28 mmol) in 3 mL of CH_3CN afforded 62 mg (62%) of **3o**: Liquid; ^1H NMR (300 MHz, $CDCl_3$) δ 7.33-7.16 (m, 10 H), 5.59 (s, 1 H), 5.44 (s, 1 H), 4.79 (d, *J* = 4.8 Hz, 1 H), 4.25-4.02 (m, 5 H), 3.10 (dd, *J* = 5.3 and 14.3 Hz, 1 H), 2.94 (dd, *J* = 5.3 and 14.3 Hz, 1 H), 1.23 (t, *J* = 7.1 Hz, 3 H), 1.21 (t, *J* = 7.2 Hz, 3 H); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 160.8, 160.2, 144.4, 137.3, 135.0, 129.8, 128.4, 128.3, 128.0, 126.9, 126.4, 114.7, 69.1, 67.2, 62.4, 62.3, 39.4, 14.34, 14.27; MS (ESI) *m/z* (%) 395 (M^++1), 417 (M^++Na); IR (neat) 2981, 1752, 1713, 1599, 1569, 1495, 1455, 1321, 1101 cm^{-1}; HRMS (ESI) calcd. for $C_{23}H_{26}N_2O_4Na$ (M^++Na) 417.1785; Found: 417.1787.

(16) 3-Benzyl-4-(1'-(*p*-methoxyphenyl)vinyl)-1,2-bis(ethoxycarbonyl)-1,2-diazetidine (3p) (entry 16, Table 2).



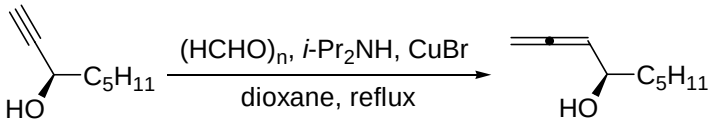
1d **3p**

The reaction of **1d** (64 mg, 0.20 mmol), 4-iodoanisole (57 mg, 0.24 mmol), $Pd(PPh_3)_4$ (12 mg, 0.010 mmol), and Cs_2CO_3 (73 mg, 0.22 mmol) in 2.5 mL of

CH_3CN afforded 61 mg (71%) of **3p**: Liquid; ^1H NMR (300 MHz, $CDCl_3$) δ 7.30-7.16 (m, 5 H), 7.17 (d, *J* = 8.9 Hz, 2 H), 6.81 (d, *J* = 8.9 Hz, 2 H), 5.51 (s, 1 H), 5.36 (s, 1 H), 4.76 (d, *J* = 4.8 Hz, 1 H), 4.25-4.00 (m, 5 H), 3.81 (s, 3 H), 3.10 (dd, *J* = 5.3 and 14.4 Hz, 1 H), 2.95 (dd, *J* = 5.3 and 14.4 Hz, 1 H), 1.23 (t, *J* = 6.9 Hz, 3 H), 1.21 (t, *J* = 7.2 Hz, 3 H); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 160.8, 160.2, 159.4, 143.7, 135.1, 129.8, 129.7, 128.3, 127.6, 126.9, 113.8, 113.2, 69.2, 67.3, 62.4, 62.3, 55.2, 39.4, 14.34, 14.27; MS (EI) *m/z* (%) 424 (M^+, 55.78), 160 (100); IR (neat) 2980, 1752, 1711, 1608, 1513, 1465, 1322, 1009 cm^{-1}; HRMS (EI) calcd for $C_{24}H_{28}N_2O_5$ (M^+) 424.1998. Found 424.2009.

Synthesis of Optically Active 2,3-Allenylic Hydrazines:

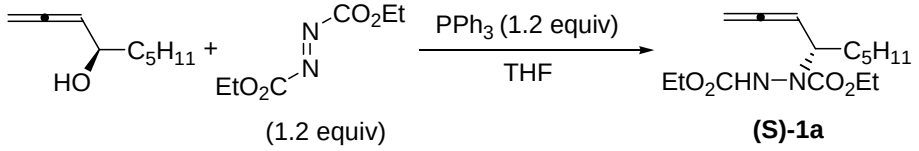
Synthesis of (R)-1,2-nonadien-4-ol



In a reaction flask containing CuBr (128 mg, 0.89 mmol) and paraformaldehyde (135 mg, 4.5 mmol) were added subsequently *i*-Pr_2NH (0.45 mL, 323 mg, 3.2 mmol), (*R*)-1-octyn-3-ol (224 mg, 1.77 mmol, 99.9% ee), and 1,4-dioxane (6 mL). The mixture was heated at 110 °C for 2.5 h. After cooling, the mixture was evaporated and the crude product was submitted to chromatography on silica gel (eluent: petroleum ether/ether = 10:1) to afford 164 mg (66%) of (*R*)-1,2-nonadien-4-ol: $[\alpha]^{20}_D$ = - 7.2 (*c* = 1.05, $CHCl_3$); Liquid; ^1H NMR (300 MHz, $CDCl_3$) δ 5.23 (q, *J* = 6.2 Hz, 1 H), 4.87-4.81 (m, 2 H), 4.22-4.10 (m, 1 H), 1.72 (s, 1 H), 1.63-1.48 (m, 2 H), 1.47-1.21

(m, 6 H), 0.88 (t, J = 7.1 Hz, 3 H).

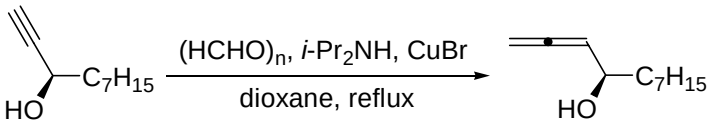
Synthesis of 4-(*N, N*-Bis(ethoxycarbonyl)hydrazino)-1,2-nonadiene ((S)-1a)



(1.2 equiv) **(S)-1a**

A solution of diethyl azodicarboxylate (213 mg, 1.22 mmol) in 2 mL of THF was added dropwise to a solution of triphenyl phosphine (318 mg, 1.21 mmol) and (*R*)-1,2-nonadien-4-ol (141 mg, 1.01 mmol) in 4 mL of THF at 0 °C with stirring. After being stirred at 0 °C for 1 h and then at room temperature for additional 1 h, the reaction mixture was evaporated and the residue was purified by flash chromatography on silica gel (eluent: petroleum ether/ether = 5:1) to afford 221 mg (74%) of (S)-**1a**: $[\alpha]^{20}_{D}$ = - 61.9 (c = 1.00, $CHCl_3$); Liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.47-6.20 (m, 1 H), 5.30-5.01 (m, 1 H), 4.84-4.56 (m, 3 H), 4.16 (q, J = 7.0 Hz, 4 H), 1.72-1.43 (m, 2 H), 1.42-1.14 (m, 12 H), 0.85 (t, J = 6.6 Hz, 3 H).

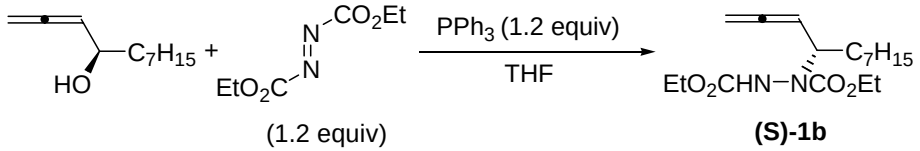
Synthesis of (R)-1,2-undecadien-4-ol



The reaction of CuBr (135 mg, 0.94 mmol), paraformaldehyde (140 mg, 4.7 mmol), *i*-Pr_2NH (0.47 mL, 337 mg, 3.3 mmol), and (*R*)-1-decyn-3-ol (286 mg, 1.85 mmol, 99.4% ee) in 1,4-dioxane (6 mL) afforded 243 mg (78%) of (*R*)-1,2-undecadien-4-ol: $[\alpha]^{20}_{D}$ = - 6.2 (c = 1.10, $CHCl_3$); $[\alpha]^{20}_{D}$ = + 22.9 (c = 1.20, MeOH);[1] Liquid; ^{1}H NMR

(300 MHz, $CDCl_3$) δ 5.23 (q, *J* = 6.6 Hz, 1 H), 4.87-4.81 (m, 2 H), 4.21-4.11 (m, 1 H), 1.72 (s, 1 H), 1.62-1.48 (m, 2 H), 1.47-1.19 (m, 10 H), 0.887 (t, *J* = 6.6 Hz, 3 H).

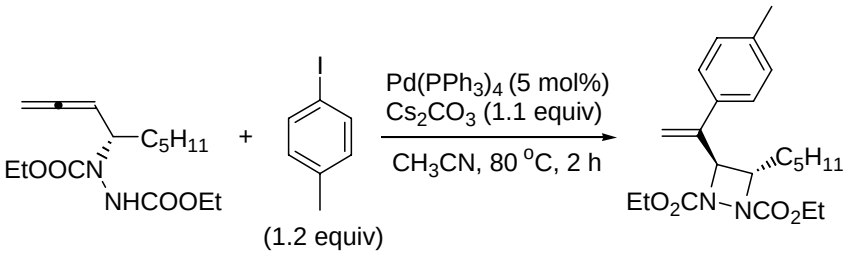
Synthesis of 4-(*N, N*-Bis(ethoxycarbonyl)hydrazino)-1,2-undecadiene ((S)-1b)



(1.2 equiv) **(S)-1b**

The reaction of diethyl azodicarboxylate (212 mg, 1.22 mmol), triphenyl phosphine (318 mg, 1.21 mmol), and (*R*)-1,2-undecadien-4-ol (168 mg, 1.00 mmol) in THF (4+2 mL) afforded 227 mg (70%) of (S)-**1b**: $[\alpha]^{20}_{D}$ = - 55.6 (*c* = 1.00, $CHCl_3$); Liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.52-6.24 (m, 1 H), 5.26-5.03 (m, 1 H), 4.83-4.55 (m, 3 H), 4.15 (q, *J* = 7.1 Hz, 4 H), 1.71-1.43 (m, 2 H), 1.42-1.13 (m, 16 H), 0.83 (t, *J* = 6.6 Hz, 3 H).

(17) (3S, 4S)-3-(*n*-Pentyl)-4-(1'-*p*-tolylvinyl)-1,2-bis(ethoxycarbonyl)-1,2-diazetidine ((3S, 4S)-3a) (entry 1, Table 3).

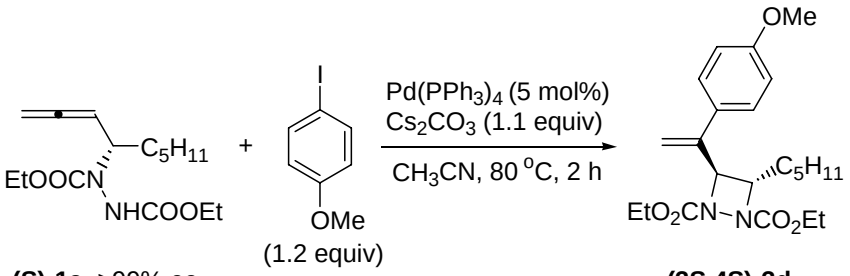


(S)-1a, >99% ee (1.2 equiv) **(3S,4S)-3a**

The reaction of (S)-**1a** (61 mg, 0.20 mmol), 4-iodotoluene (53 mg, 0.24 mmol), $Pd(PPh_3)_4$ (12 mg, 0.010 mmol), and Cs_2CO_3 (72 mg, 0.22 mmol) in 2.5 mL of CH_3CN afforded 59 mg (74%) of (3S, 4R)-**3a** with 99.4% ee as determined by HPLC analysis (Chiralcel OD-H, *n*-Hexane : *i*-PrOH = 100 : 1, 0.7 mL/min, 230 nm), t_r 14.0

(major), 19.5 (minor); $[\alpha]^{20}_{D}$ = + 40.1 (*c* = 1.05, $CHCl_3$).

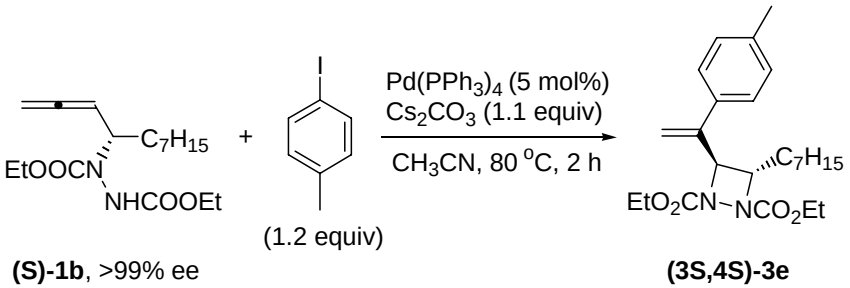
(18) (3S, 4S)-3-(*n*-Pentyl)-4-(1'-(*p*-methoxyphenyl)vinyl)-1,2-bis(ethoxycarbonyl)-1,2-diazetidine ((3S, 4S)-3d) (entry 2, Table 3).



(S)-1a, >99% ee **(3S,4S)-3d**

The reaction of (S)-**1a** (62 mg, 0.21 mmol), 4-iodoanisole (57 mg, 0.24 mmol), $Pd(PPh_3)_4$ (12 mg, 0.010 mmol), and Cs_2CO_3 (73 mg, 0.22 mmol) in 2.5 mL of CH_3CN afforded 62 mg (74%) of (3S, 4R)-**3d** with 99.4% ee as determined by HPLC analysis (Chiralcel OD-H, *n*-Hexane : *i*-PrOH = 100 : 1, 0.7 mL/min, 254 nm), t_r 24.0 (major), 36.7 (minor); $[\alpha]^{20}_{D}$ = + 40.1 (*c* = 1.05, $CHCl_3$).

(19) (3S, 4S)-3-(*n*-Heptyl)-4-(1'-*p*-tolylvinyl)-1,2-bis(ethoxycarbonyl)-1,2-diazetidine ((3S, 4S)-3e) (entry 3, Table 3).

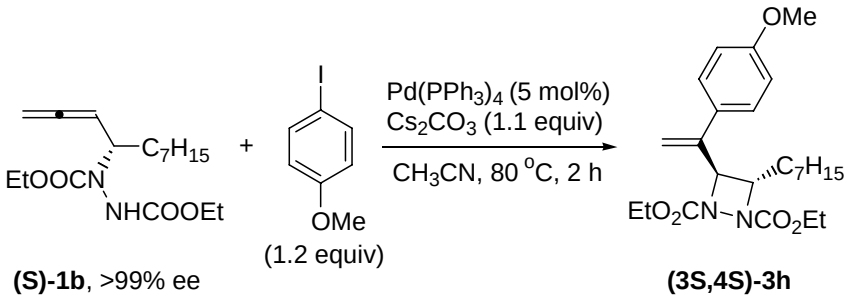


(S)-1b, >99% ee **(3S,4S)-3e**

The reaction of (S)-**1b** (66 mg, 0.20 mmol), 4-iodotoluene (52 mg, 0.24 mmol), $Pd(PPh_3)_4$ (12 mg, 0.010 mmol), and Cs_2CO_3 (73 mg, 0.22 mmol) in 2.5 mL of CH_3CN afforded 57 mg (68%) of (3S, 4R)-**3e** with 98.5% ee as determined by HPLC

analysis (Chiralcel OD-H, *n*-Hexane : *i*-PrOH = 95 : 5, 0.7 mL/min, 230 nm), t_r 7.3 (major), 9.2 (minor); $[\alpha]^{20}_D$ = + 35.5 (*c* = 1.05, $CHCl_3$).

(20) (3S, 4S)-3-(*n*-Heptyl)-4-(1'-(*p*-methoxyphenyl)vinyl)-1,2-bis(ethoxy-carbonyl)-1,2-diazetidine ((3S, 4S)-3h) (entry 4, Table 3).

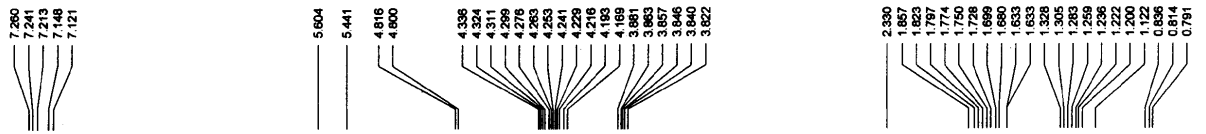


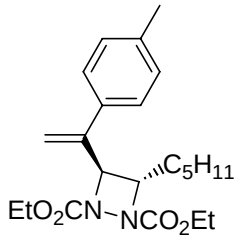
(S)-1b, >99% ee **(3S,4S)-3h**

The reaction of (S)-**1b** (65 mg, 0.21 mmol), 4-iodoanisole (56 mg, 0.24 mmol), $Pd(PPh_3)_4$ (12 mg, 0.010 mmol), and Cs_2CO_3 (72 mg, 0.22 mmol) in 2.5 mL of CH_3CN afforded 65 mg (75%) (3S, 4R)-**3h** of with 98.5% ee as determined by HPLC analysis (Chiralcel OD-H, *n*-Hexane : *i*-PrOH = 95 : 5, 0.7 mL/min, 230 nm), t_r 9.0 (major), 11.2 (minor); $[\alpha]^{20}_D$ = + 34.9 (*c* = 1.05, $CHCl_3$).

Reference:

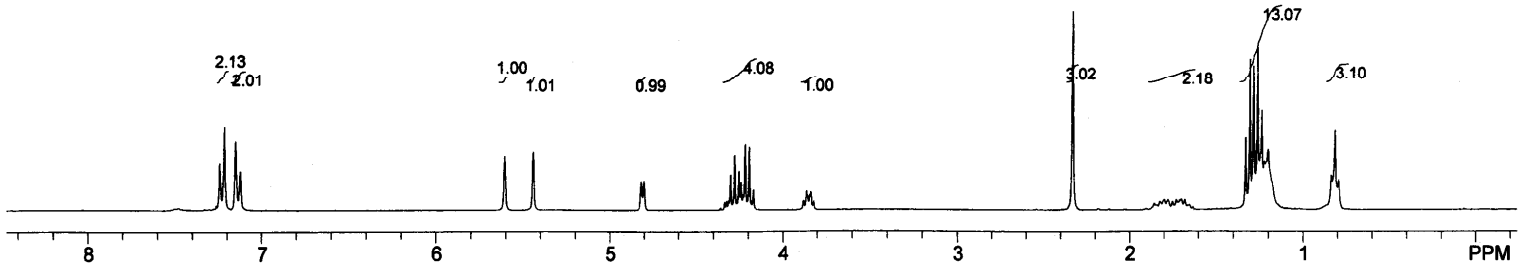
[1] S. Ma, H. Hou, S. Zhao, G. Wang, *Synthesis* **2002**, 1643.

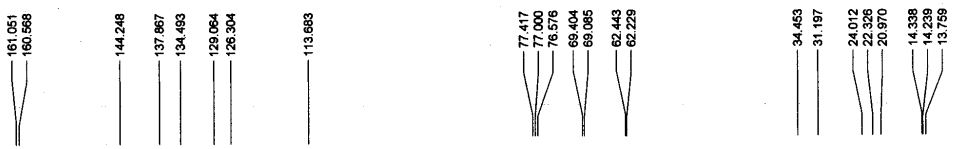


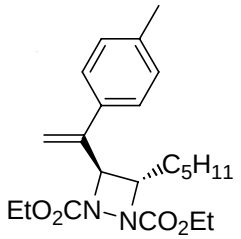


3a

cx-3-12202-h

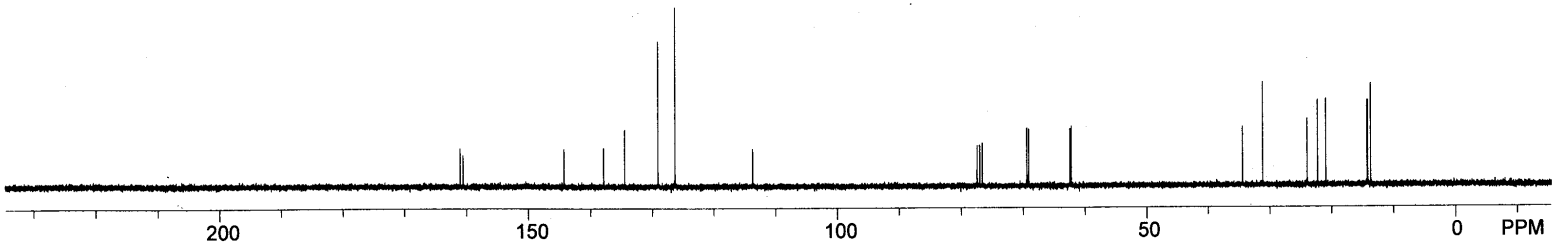


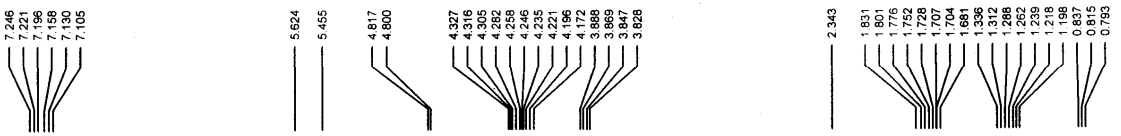




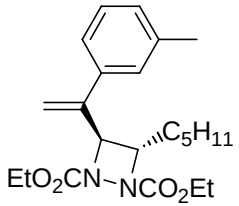
3a

cx-3-2601-1-c

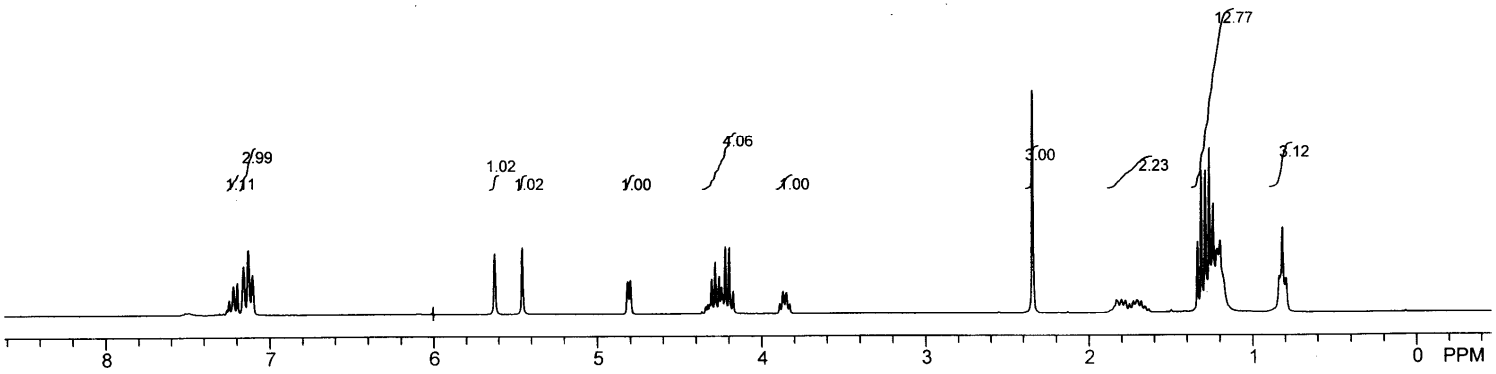


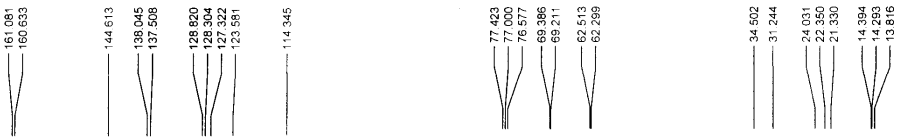


cx-3-12302-h

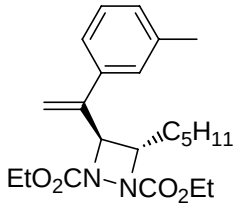


3b

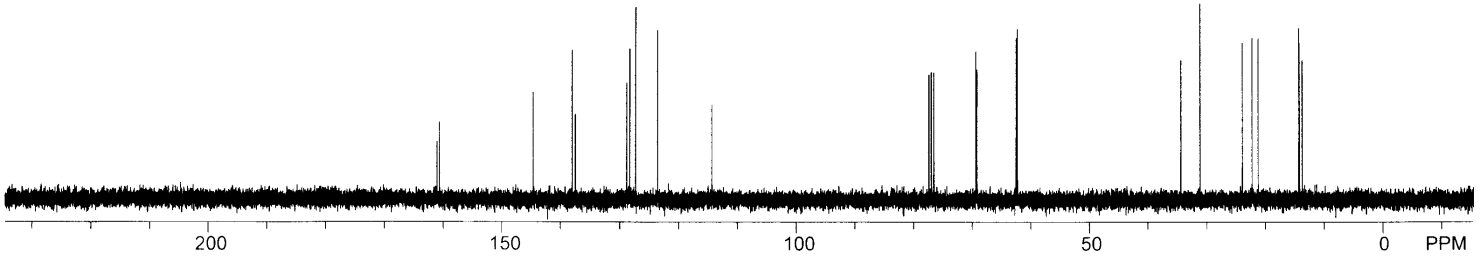


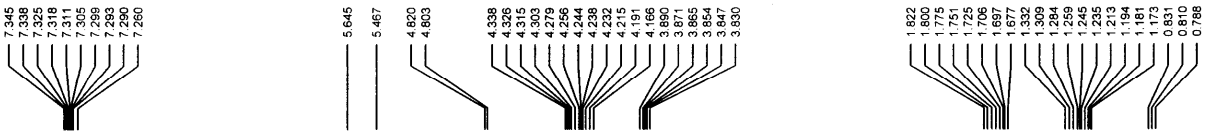


cx-3-12302-c



3b

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7.325
7.318
7.311
7.305
7.299
7.293
7.290
7.260
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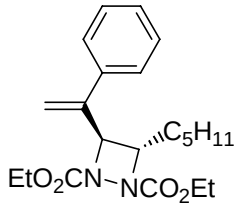
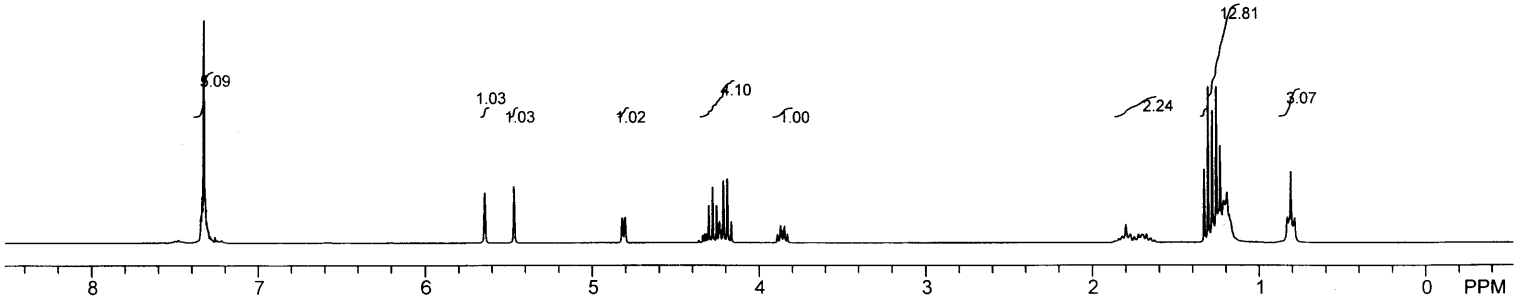
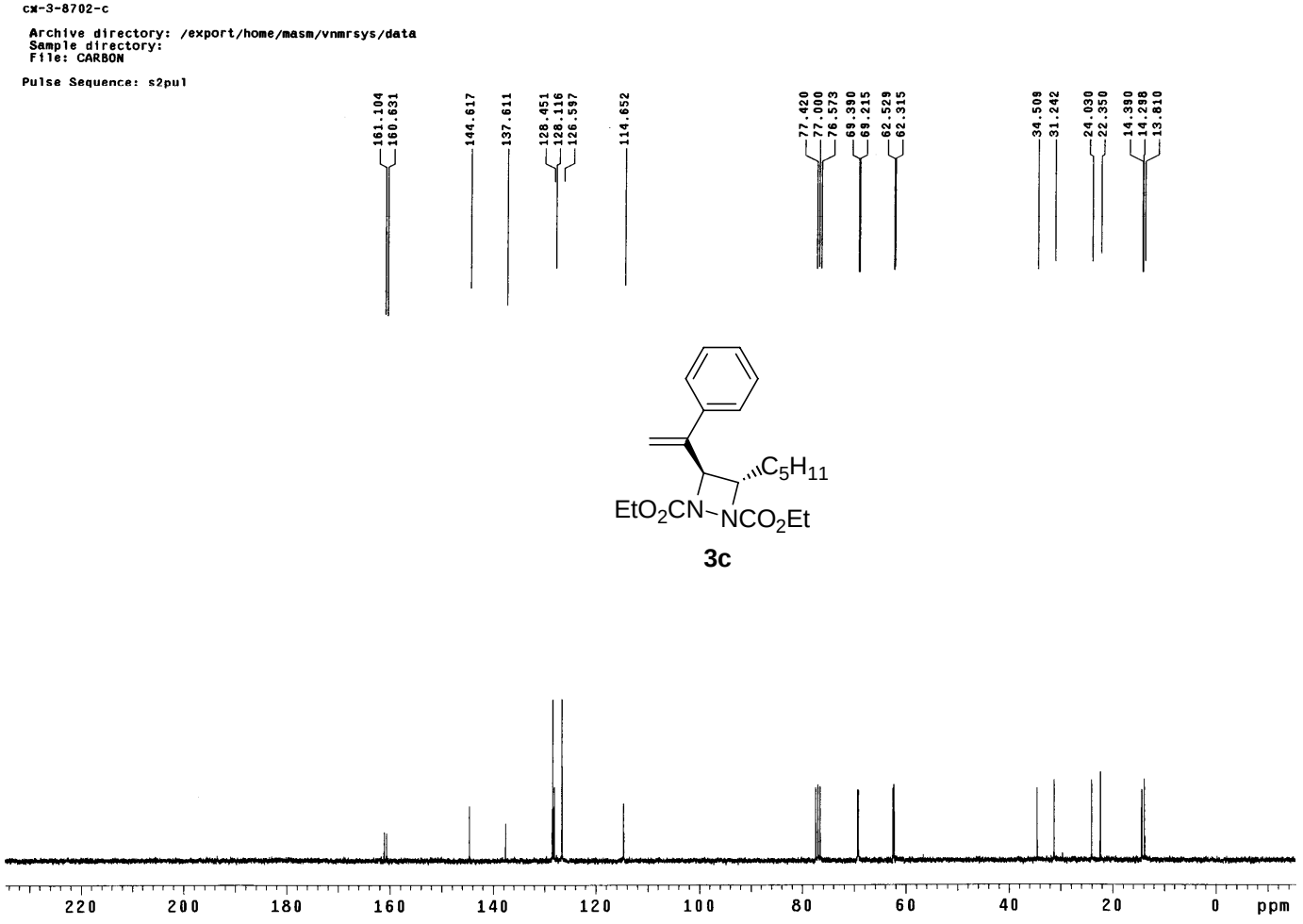
5.467
4.820
4.803
4.338
4.326
4.315
4.303
4.279
4.256
4.244
4.238
4.232
4.215
4.191
4.166
3.890
3.871
3.865
3.854
3.847
3.830
1.822
1.800
1.775
1.751
1.725
1.706
1.697
1.677
1.332
1.309
1.284
1.259
1.245
1.235
1.213
1.194
1.181
1.173
0.831
0.810
0.788

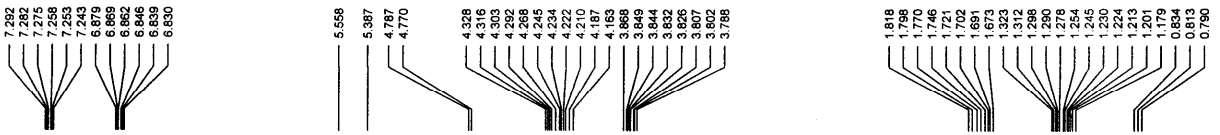
cx-3-8702-h


C_5H_{11}
EtO_2CN
NCO_2Et

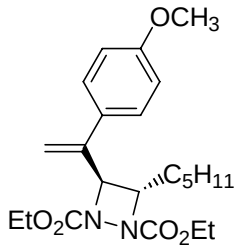
3c


5.09
1.03
1.03
1.02
4.10
1.00
2.24
12.81
3.07
8
7
6
5
4
3
2
1
0
PPM

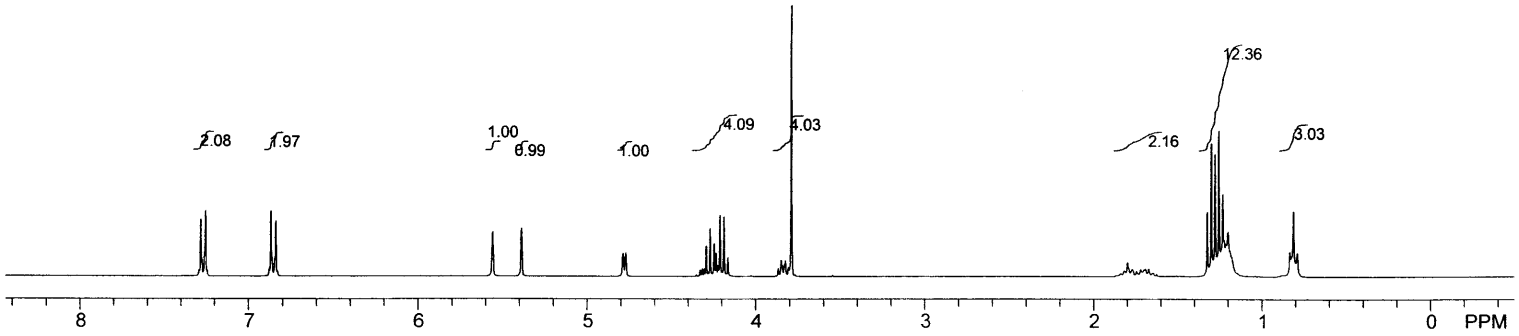

cx-3-8702-c
Archive directory: /export/home/masm/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
161.104
160.631
144.617
137.611
128.451
128.116
126.597
114.652
77.420
77.000
76.573
69.390
69.215
62.529
62.315
34.509
31.242
24.030
22.350
14.390
14.288
13.810
C5H11
EtO2CN
NCO2Et
3c
220
200
180
160
140
120
100
80
60
40
20
0
ppm

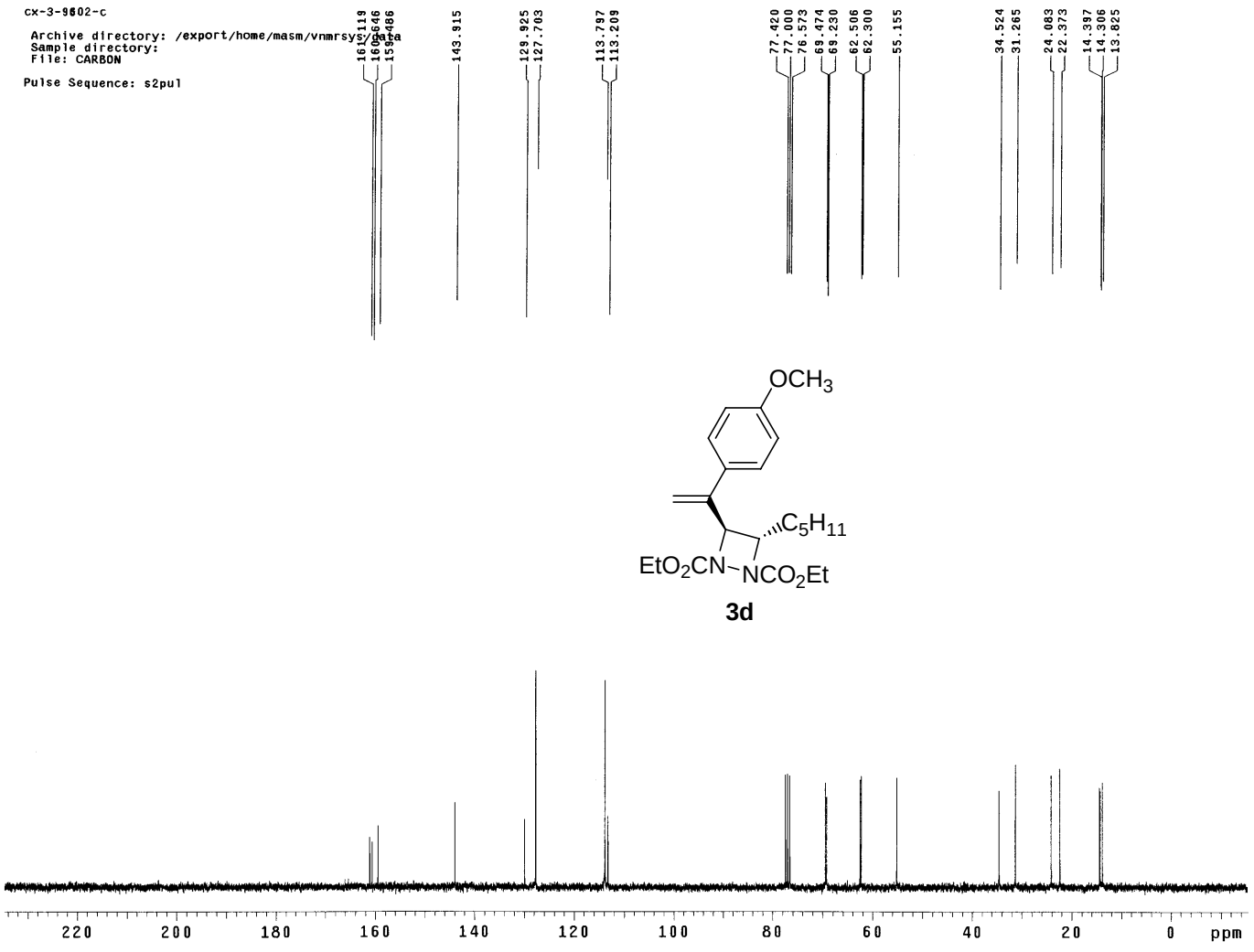

7.292
7.282
7.275
7.258
7.253
7.243
6.879
6.869
6.862
6.846
6.839
6.830
5.558
5.387
4.787
4.770
4.328
4.316
4.303
4.292
4.268
4.245
4.234
4.222
4.210
4.187
4.163
3.868
3.849
3.844
3.832
3.826
3.807
3.802
3.788
1.818
1.798
1.770
1.746
1.721
1.702
1.691
1.673
1.323
1.312
1.298
1.290
1.278
1.254
1.245
1.230
1.224
1.213
1.201
1.179
0.834
0.813
0.790

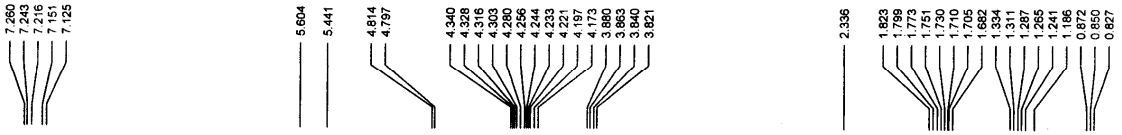
cx-3-9602-h


OCH3
C5H11
EtO2CN
NCO2Et

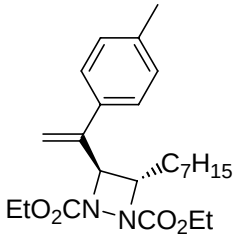
3d


2.08
1.97
1.00
0.99
1.00
4.09
4.03
2.16
12.36
3.03
8
7
6
5
4
3
2
1
0
PPM

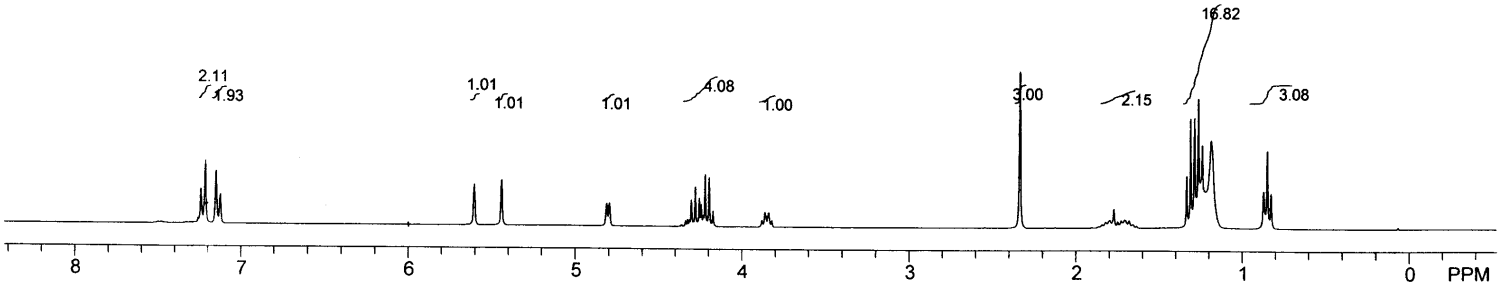

cx-3-9602-c
Archive directory: /export/home/masm/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
161.119
160.646
159.486
143.915
129.925
127.703
113.797
113.209
77.420
77.000
76.573
69.474
69.230
62.506
62.300
55.155
34.524
31.265
24.083
22.373
14.397
14.306
13.825
OCH3
C5H11
EtO2CN
NCO2Et
3d
220
200
180
160
140
120
100
80
60
40
20
0
ppm

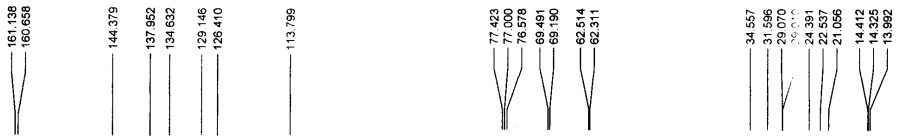


cx-3-10102-h

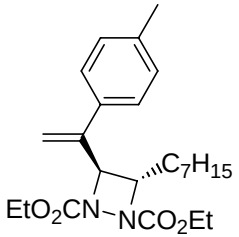


3e

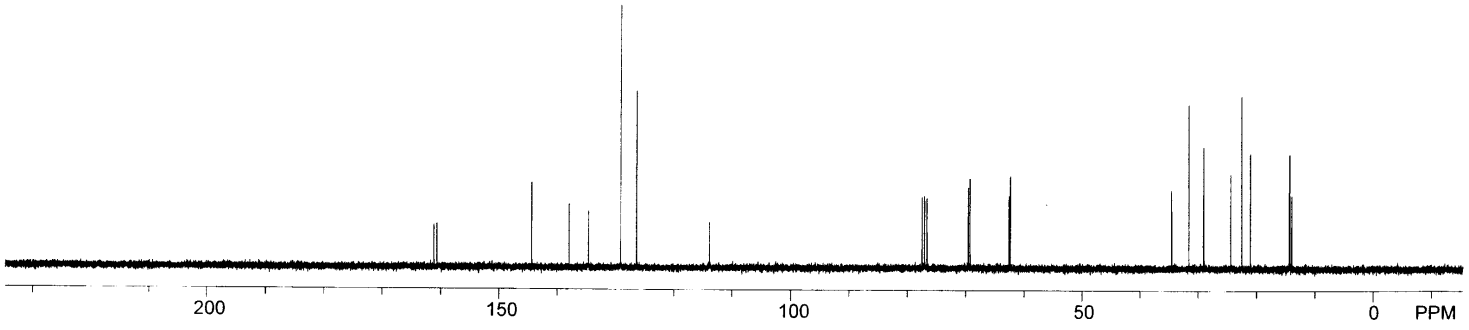


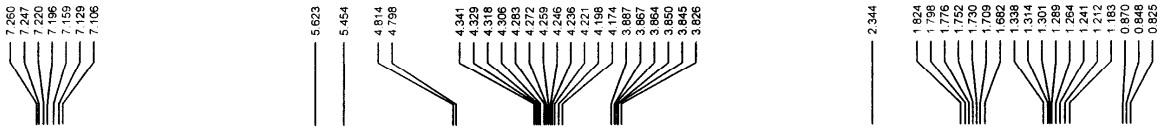


cx-3-10102-c

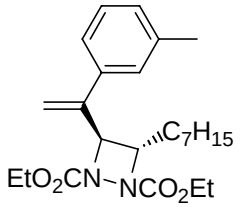


3e

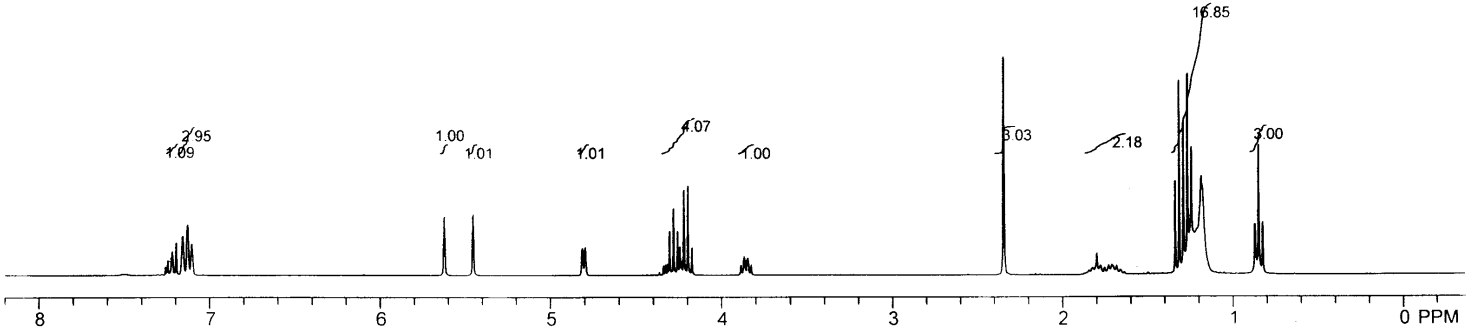


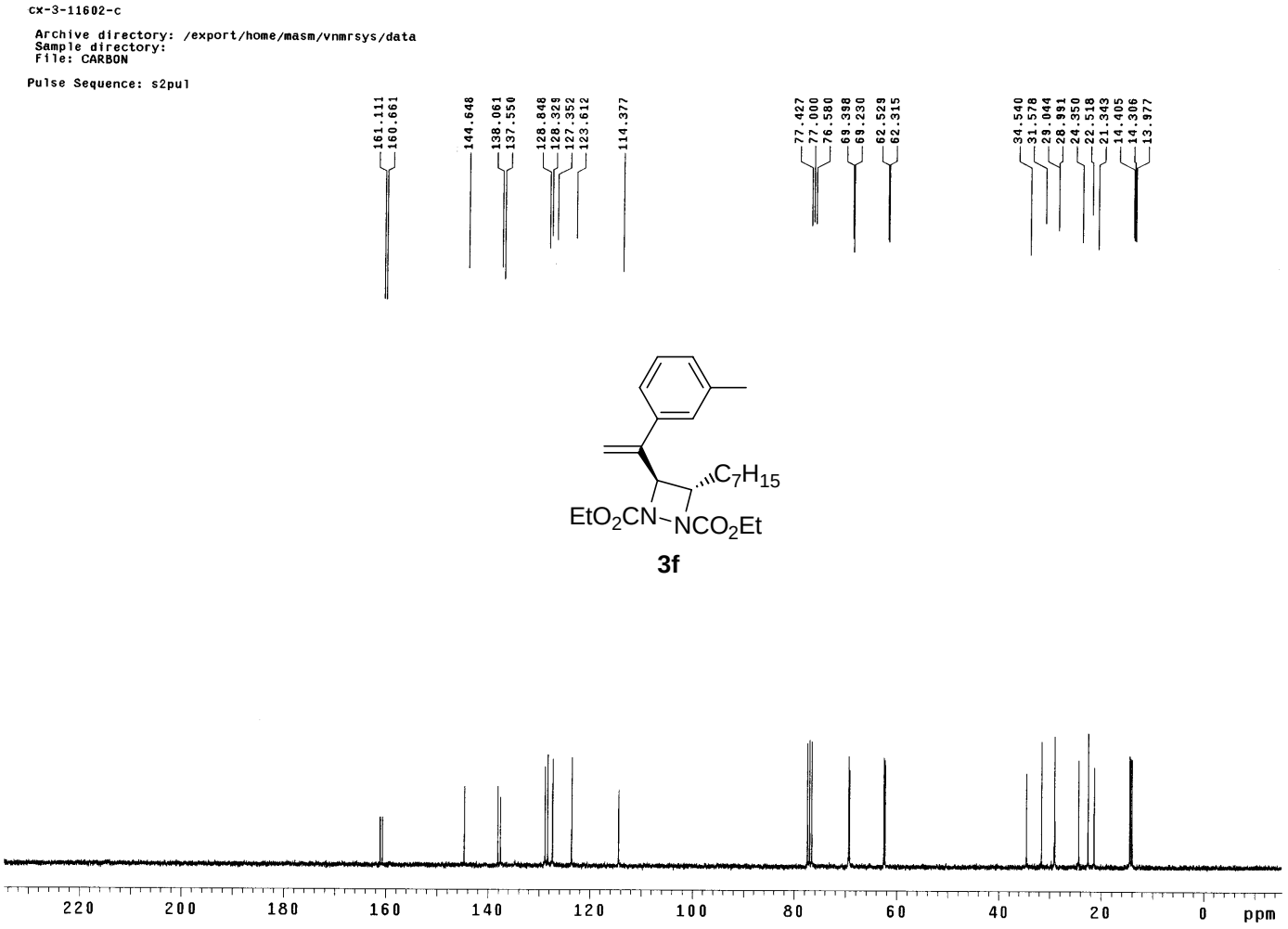


cx-3-11602-h

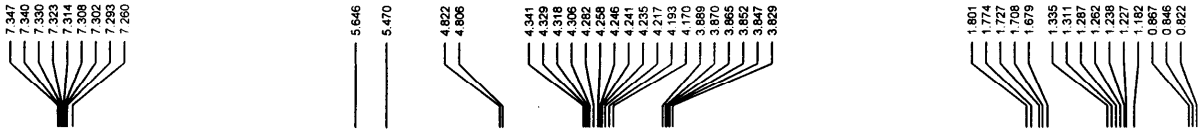


3f

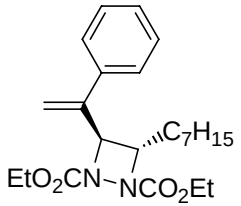



cx-3-11602-c
Archive directory: /export/home/masm/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
161.111
160.661
144.648
138.061
137.550
128.848
128.329
127.352
123.612
114.377
77.427
77.000
76.580
69.398
69.230
62.528
62.315
34.540
31.578
29.044
28.991
24.350
22.518
21.343
14.405
14.306
13.977
C_7H_{15}
EtO_2CN
NCO_2Et
220
200
180
160
140
120
100
80
60
40
20
0
ppm

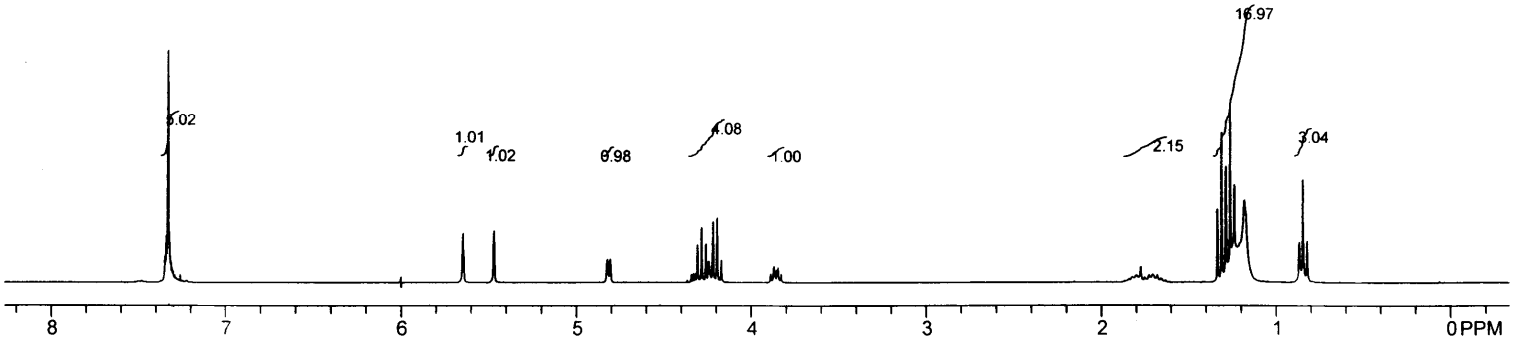
3f

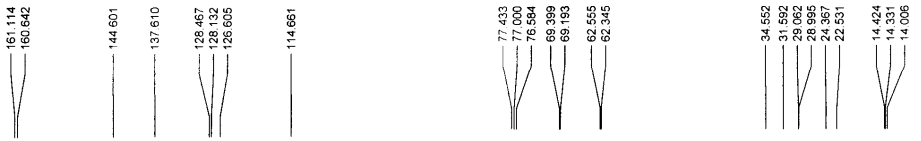


cx-3-11502-h

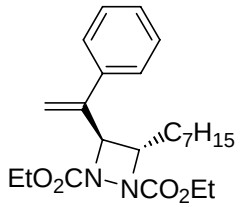


3g

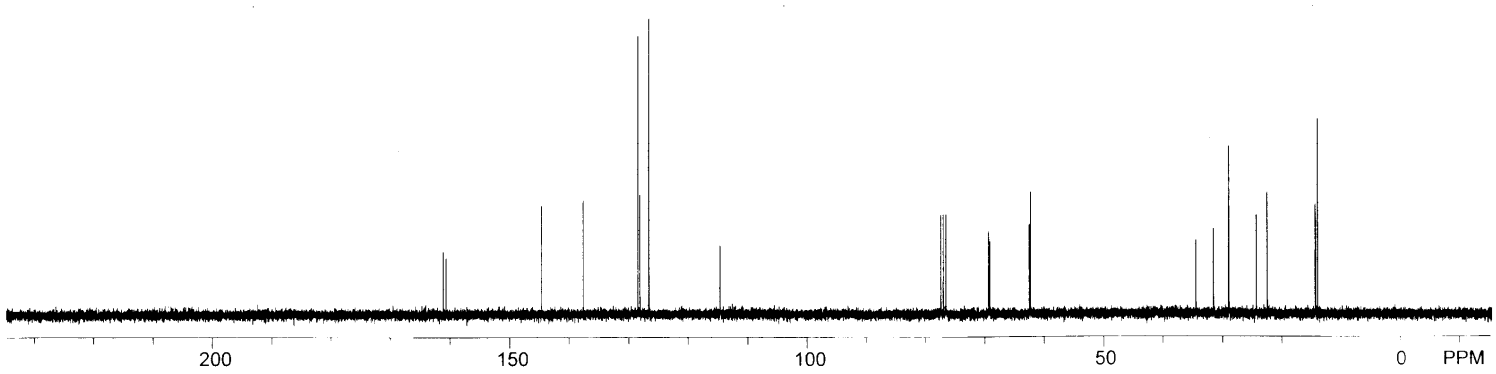


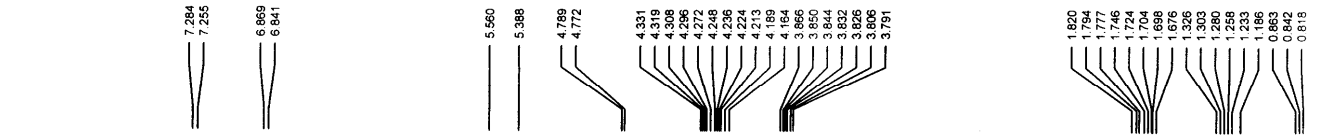


cx-3-11502-c

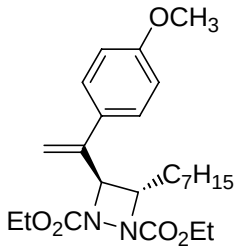


3g

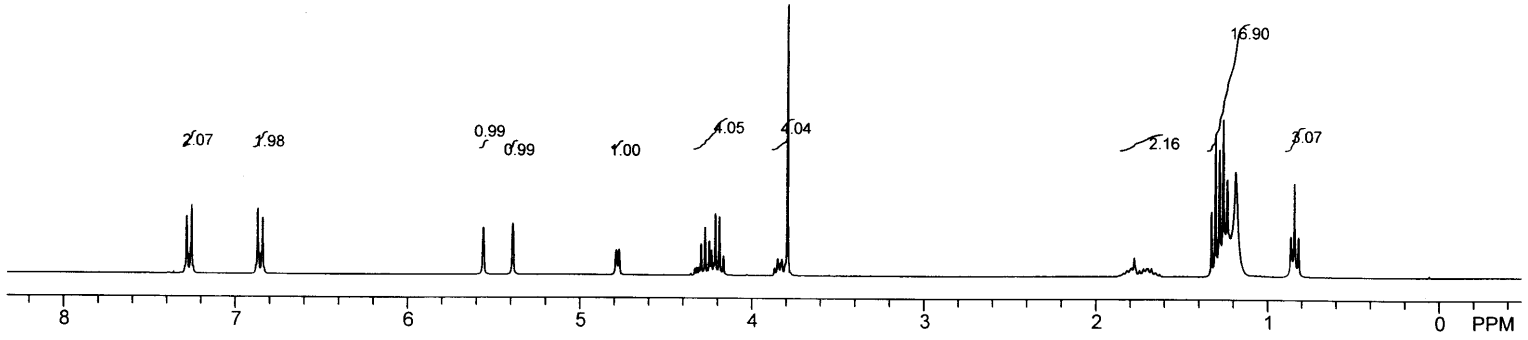


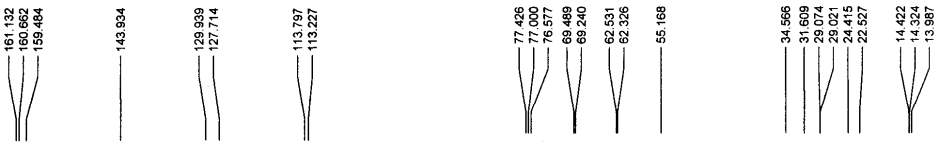


cx-3-10202-h

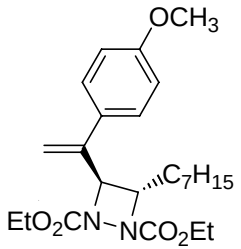


3h

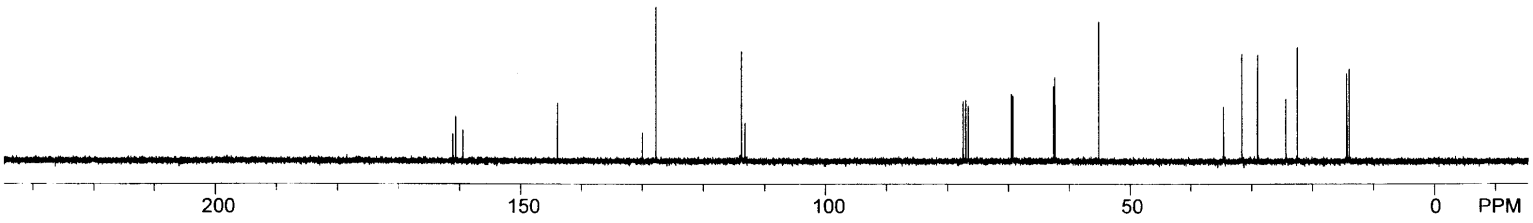


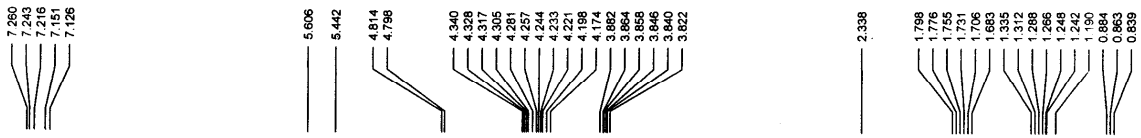


cx-3-10202-c



3h

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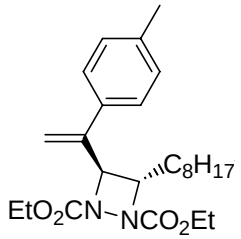
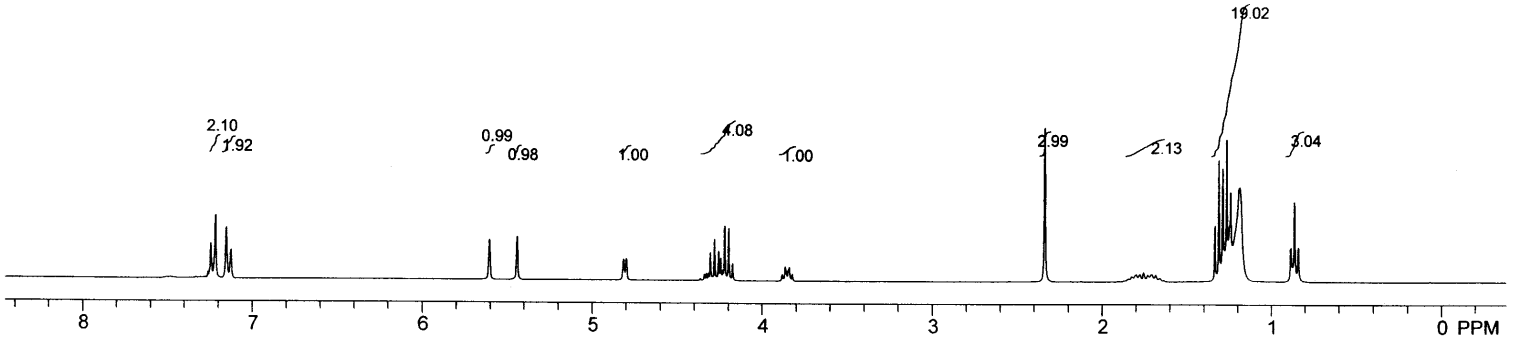
7.243
7.216
7.151
7.126
5.606
5.442
4.814
4.798
4.340
4.329
4.317
4.305
4.281
4.257
4.244
4.233
4.221
4.198
4.174
3.882
3.864
3.858
3.846
3.840
3.822
2.338
1.798
1.776
1.755
1.731
1.706
1.683
1.335
1.312
1.288
1.266
1.248
1.242
1.190
0.884
0.863
0.839

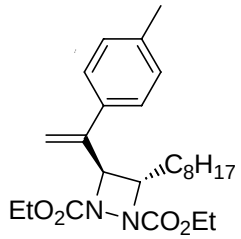
cx-3-9902-h


C8H17
EtO2CN
NCO2Et

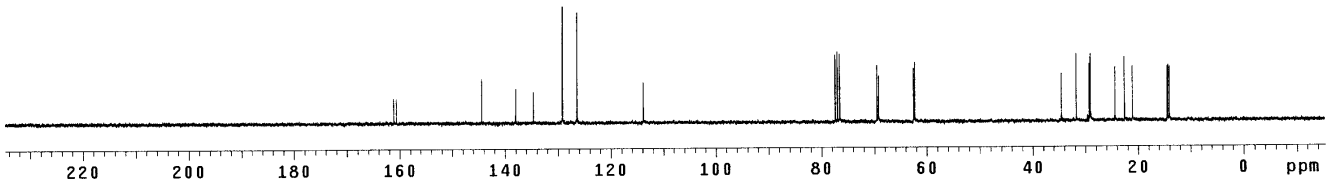
3i

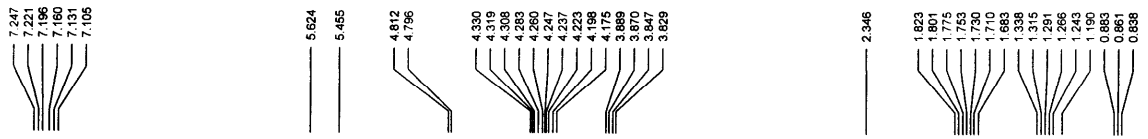

19.02
2.10
1.92
0.99
0.98
1.00
4.08
1.00
2.99
2.13
3.04
8
7
6
5
4
3
2
1
0 PPM


cx-3-9902-c
Archive directory: /export/home/masm/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
161.150
160.669
144.381
137.962
134.642
129.154
126.414
113.820
77.427
77.000
76.580
69.490
69.192
62.521
62.315
34.555
31.754
29.304
29.113
29.060
24.388
22.557
21.061
14.420
14.321
14.023

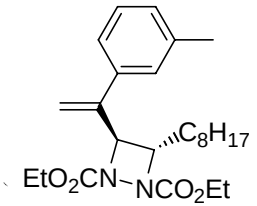

C_8H_{17}
EtO_2CN
NCO_2Et

3i

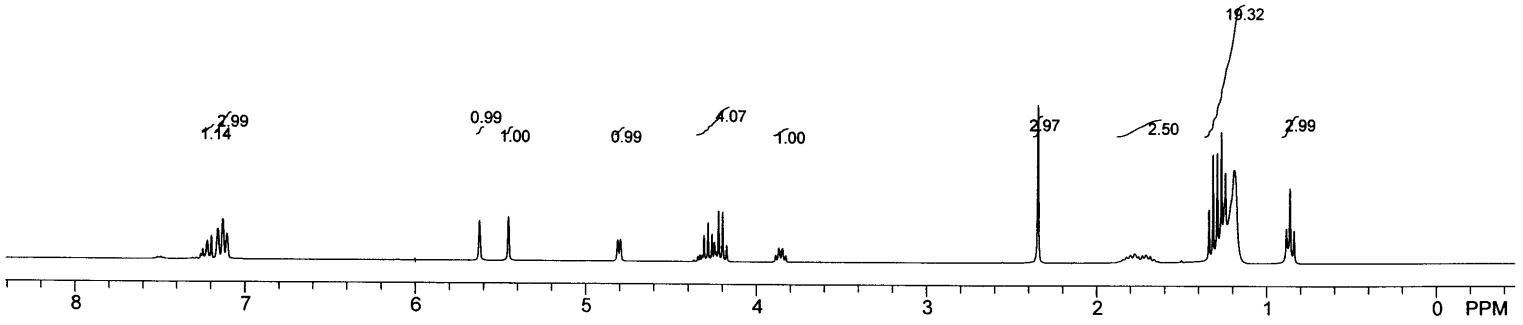

220
200
180
160
140
120
100
80
60
40
20
0
ppm

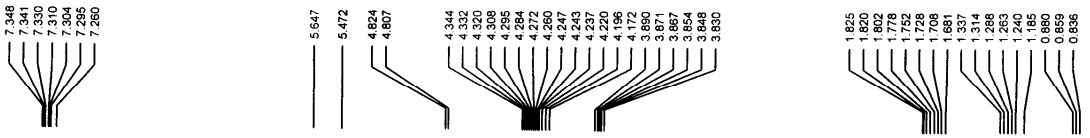


cx-3-11302-h

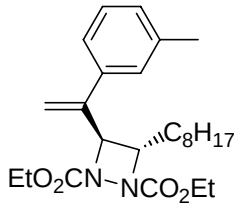


3j

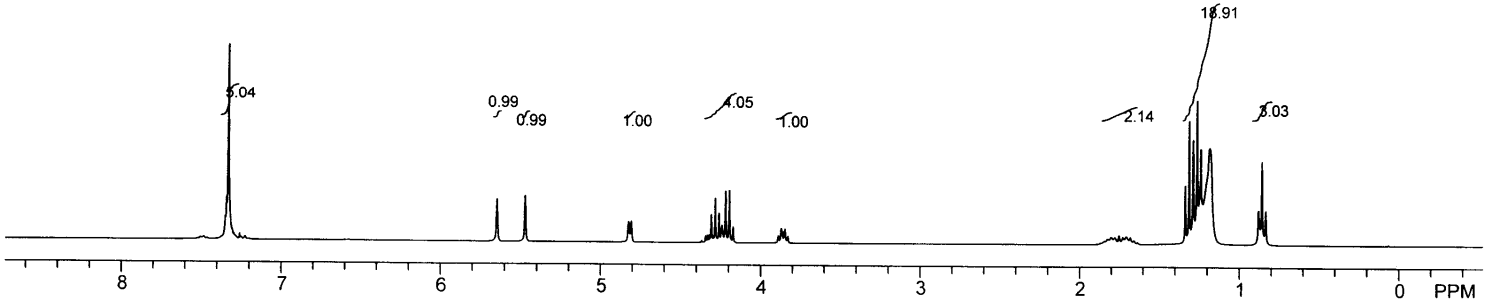


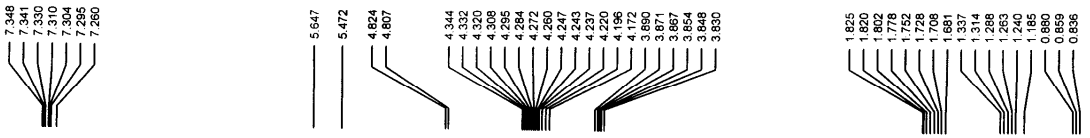


cx-3-12102-h

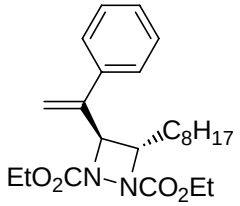


3j

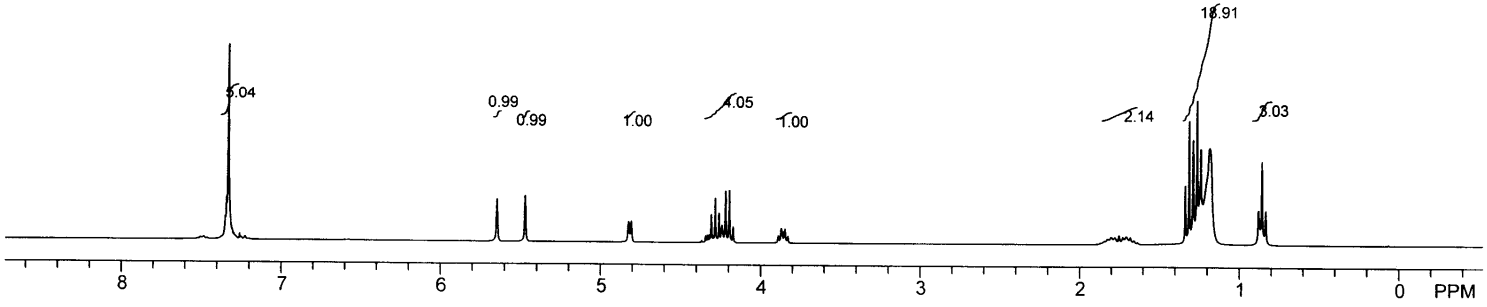


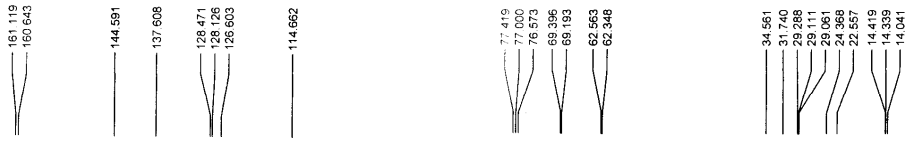


cx-3-12102-h

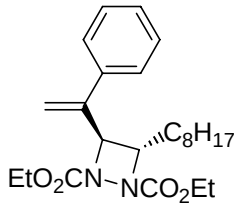


3k

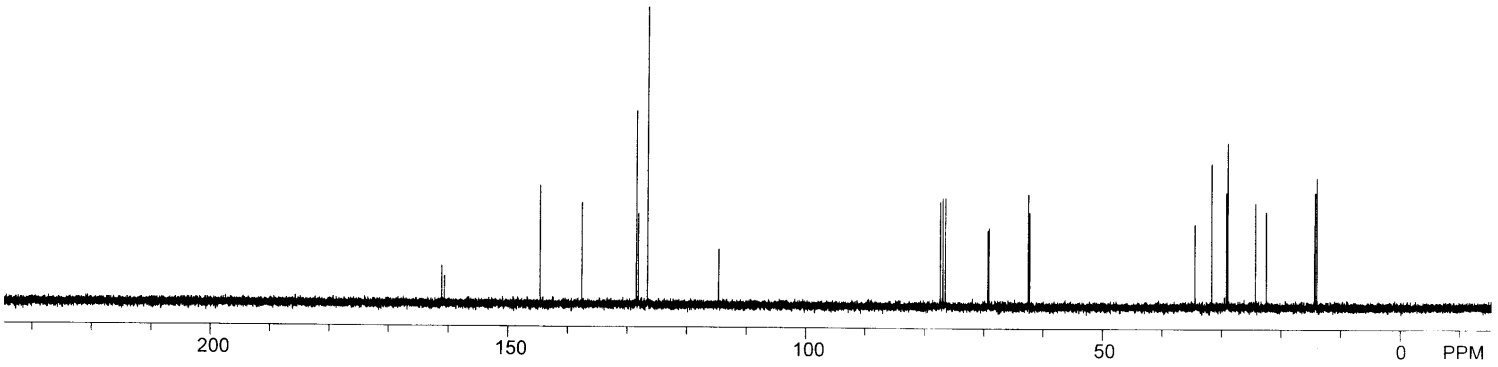


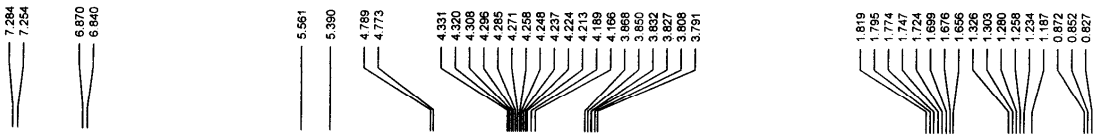


cx-3-12102-c

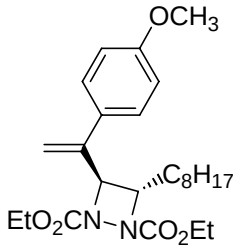


3k

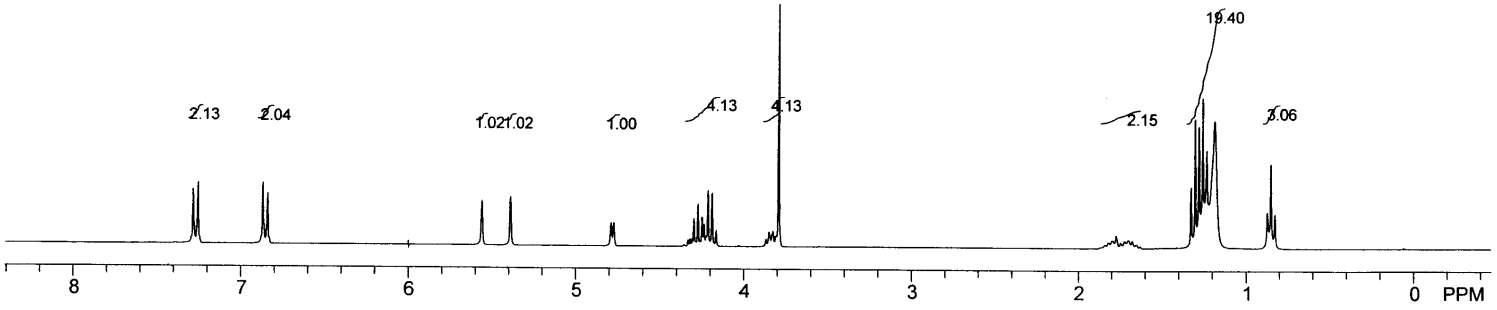


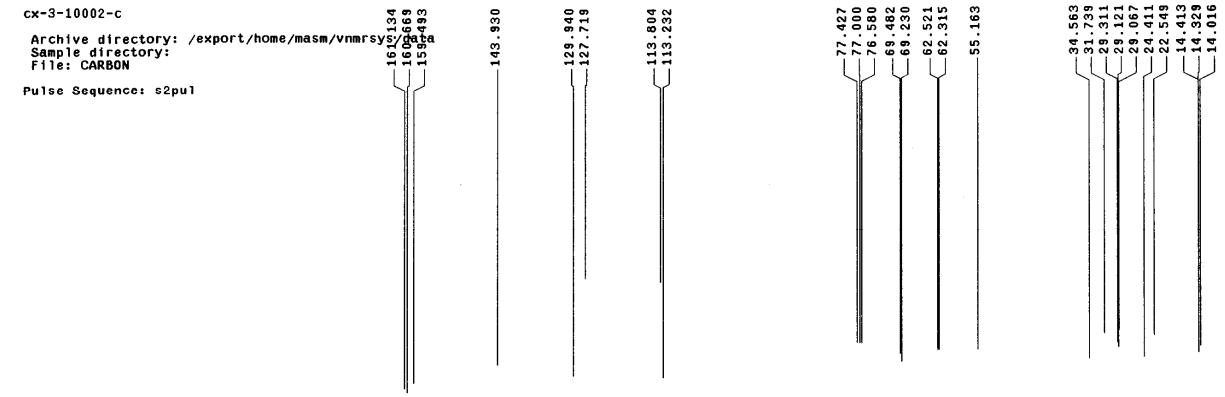


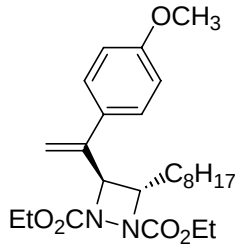
cx-3-10002-h



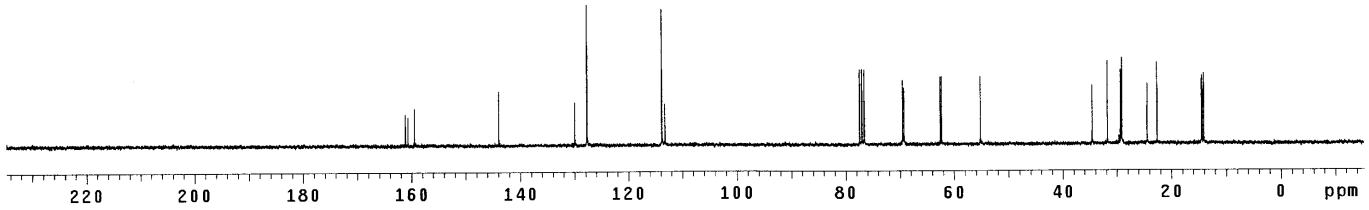
3l

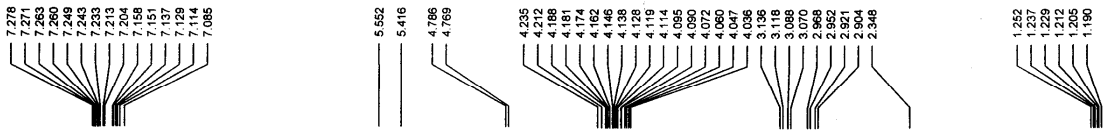



cx-3-10002-c
Archive directory: /export/home/masm/vnmrsys/exp
Sample directory:
File: CARBON
Pulse Sequence: s2pul
161.134
160.669
159.493
143.930
129.940
127.719
113.804
113.232
77.427
77.000
76.580
69.482
69.230
62.521
62.315
55.163
34.563
31.739
29.311
29.121
29.067
24.411
22.549
14.413
14.329
14.016

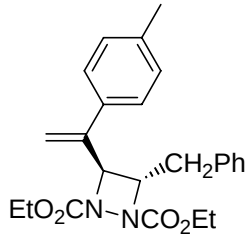

OCH3
C8H17
EtO2CN
NCO2Et

3l

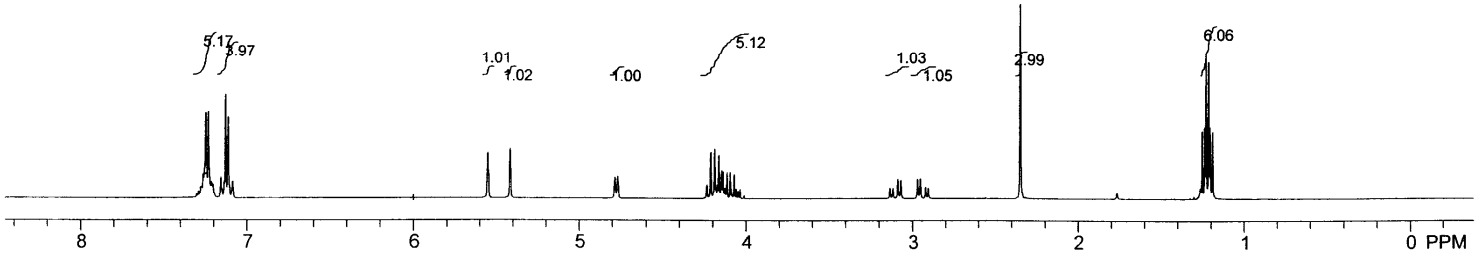

220
200
180
160
140
120
100
80
60
40
20
0
ppm

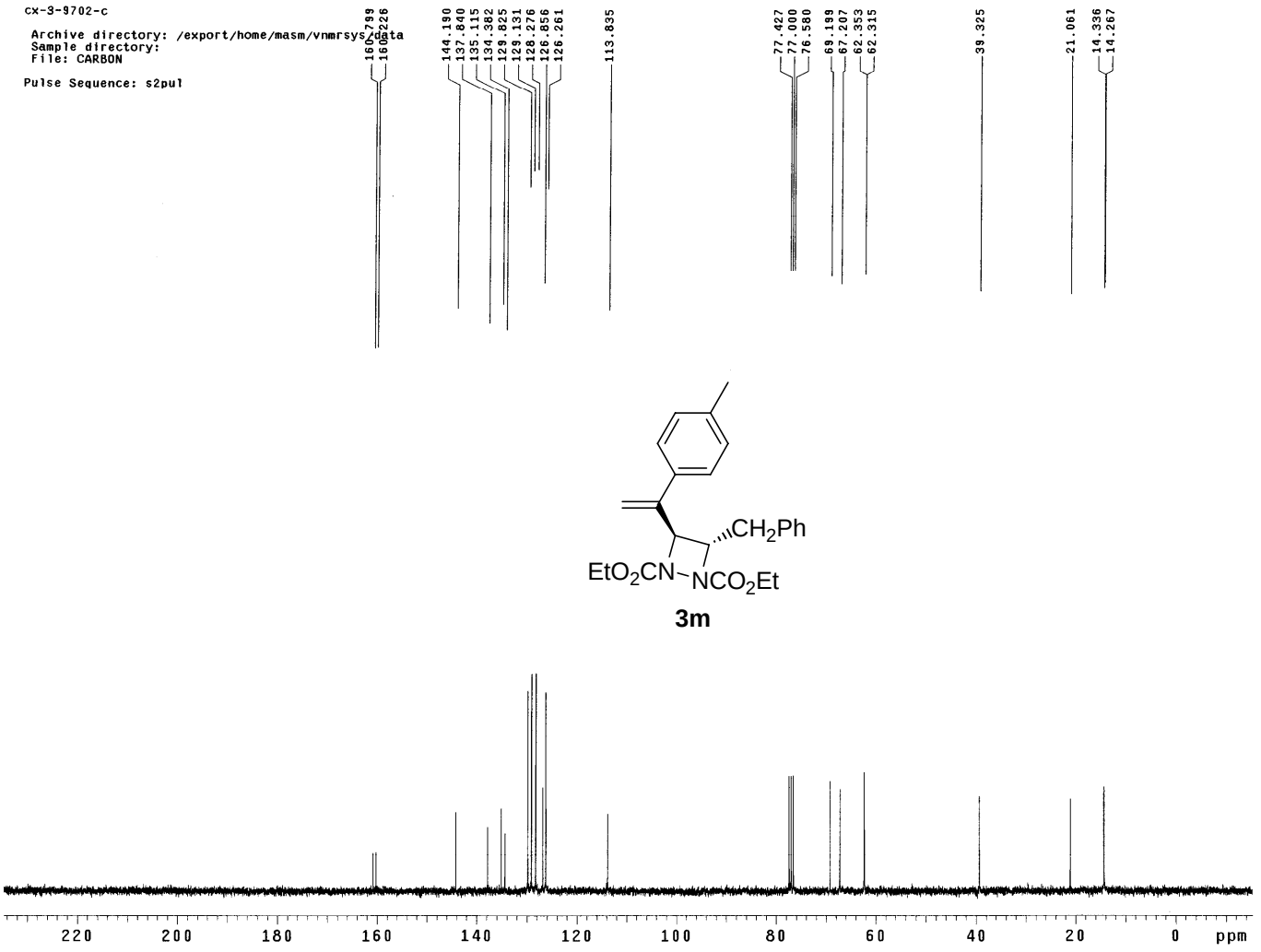

7.278
7.271
7.263
7.260
7.249
7.243
7.233
7.213
7.204
7.158
7.151
7.137
7.129
7.114
7.085
5.552
5.416
4.786
4.769
4.235
4.212
4.188
4.181
4.174
4.162
4.146
4.138
4.128
4.119
4.114
4.095
4.090
4.072
4.060
4.047
4.036
3.136
3.118
3.088
3.070
2.968
2.952
2.921
2.904
2.348
1.252
1.237
1.229
1.212
1.205
1.190

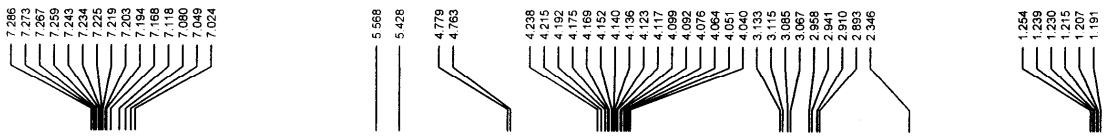
cx-3-9702-h


CH2Ph
EtO2CN
NCO2Et

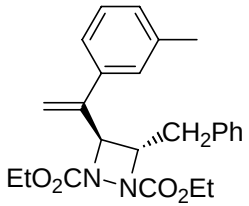
3m


5.17
2.97
1.01
1.02
1.00
5.12
1.03
1.05
2.99
6.06
8
7
6
5
4
3
2
1
0 PPM

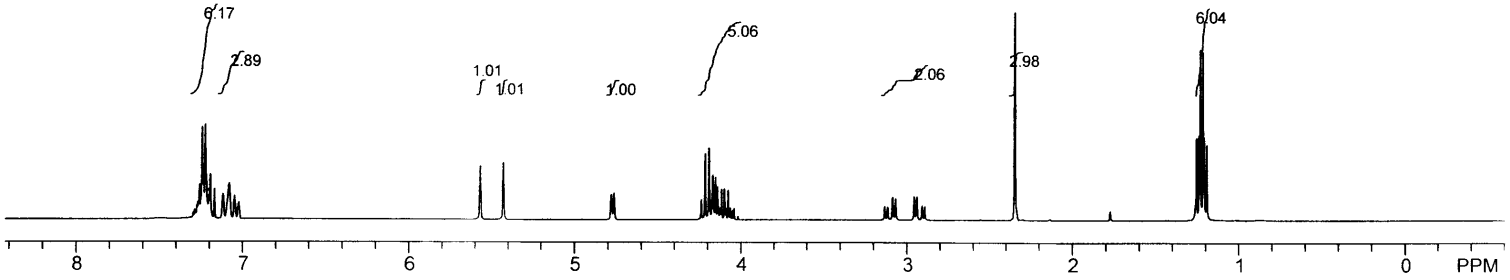

cx-3-9702-c
Archive directory: /export/home/masm/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
160.799
160.226
144.190
137.840
135.115
134.382
129.825
129.131
128.276
126.856
126.261
113.835
77.427
77.000
76.580
69.199
67.207
62.353
62.315
39.325
21.061
14.336
14.267
CH2Ph
EtO2CN
NCO2Et
3m
220
200
180
160
140
120
100
80
60
40
20
0
ppm

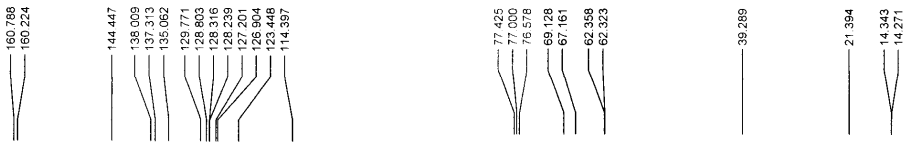


cx-3-11802-h

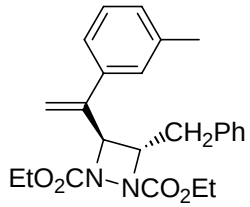


3n

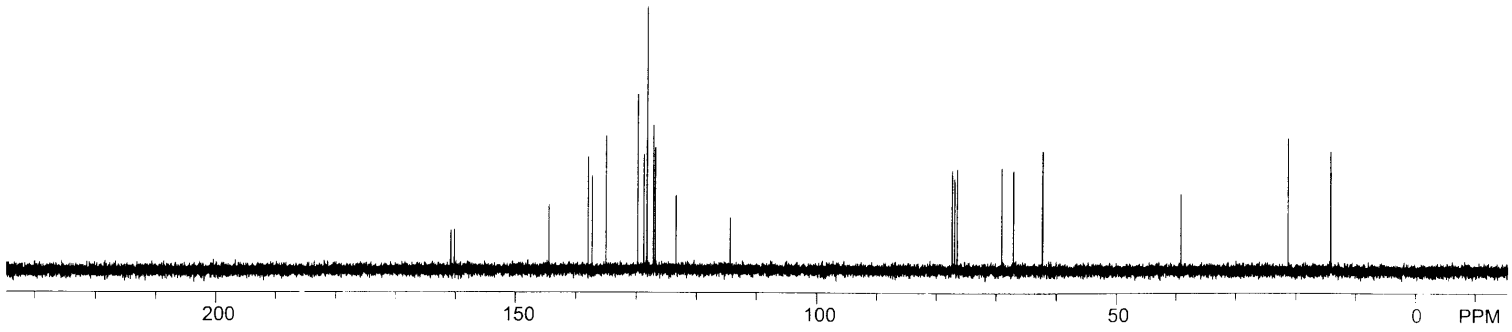


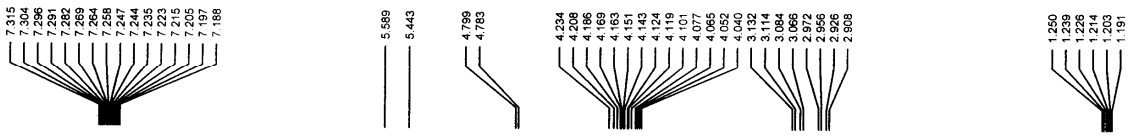


cx-3-11802-c

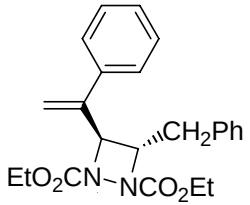


3n

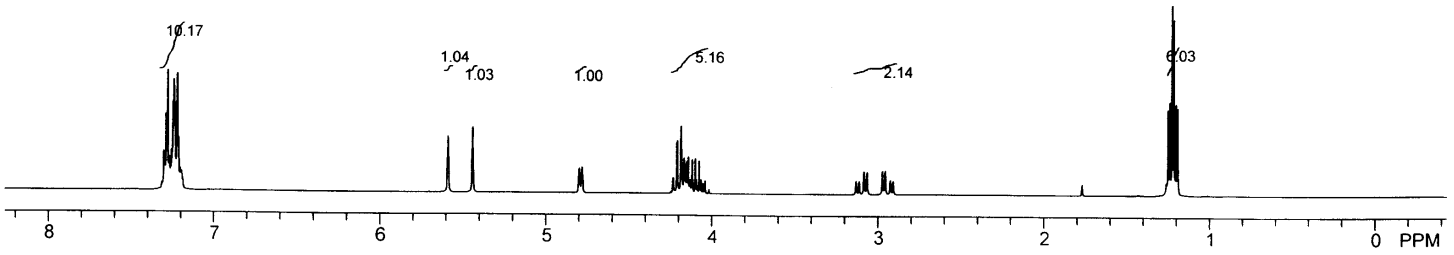


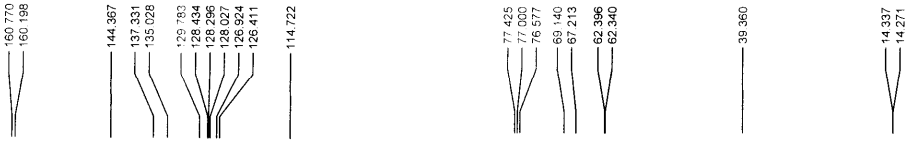


cx-3-11702-h

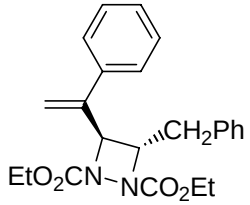


3o

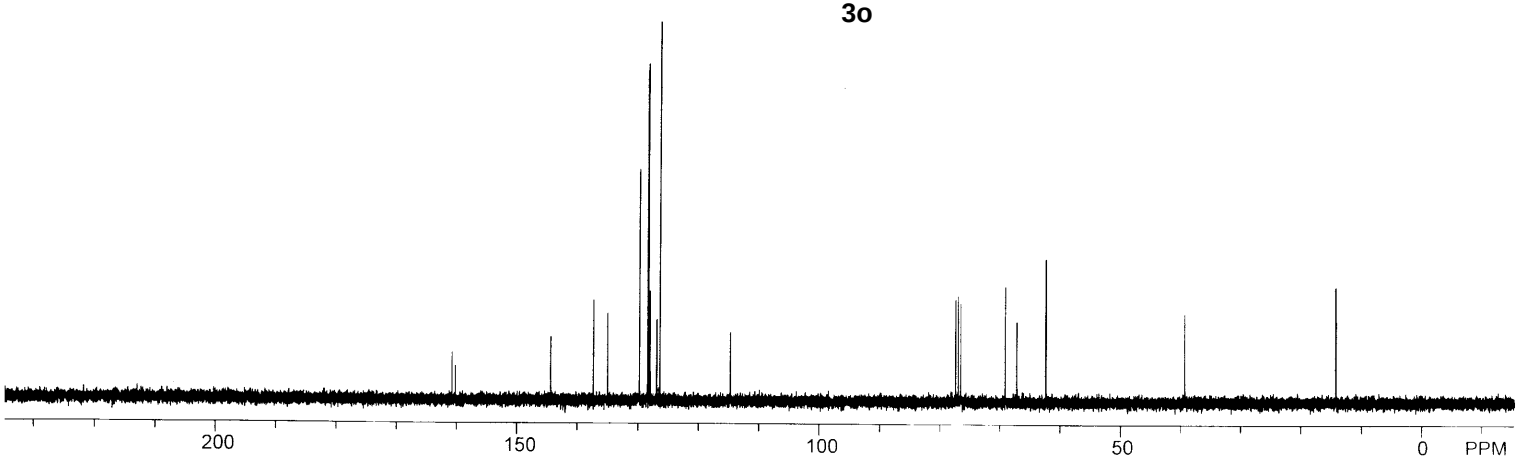


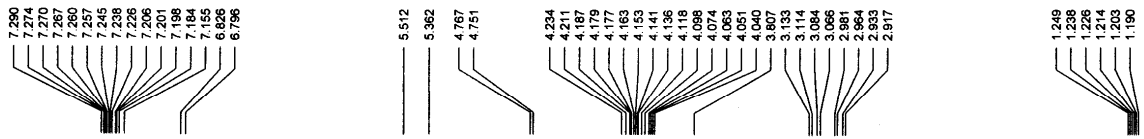


cx-3-11702-c

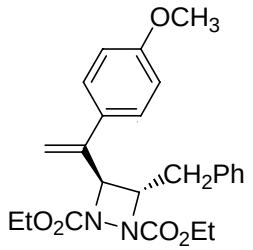


3o

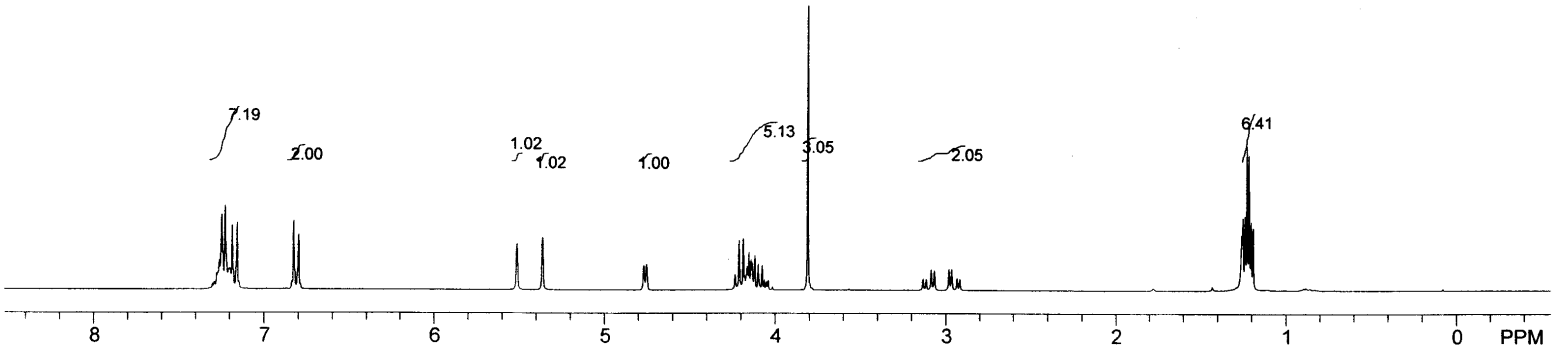


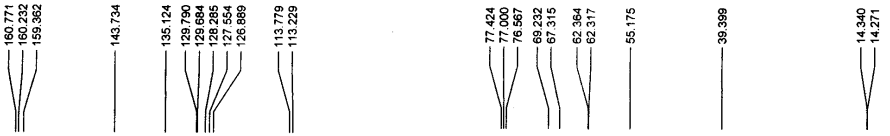

7.290
7.274
7.270
7.267
7.260
7.257
7.245
7.238
7.226
7.206
7.201
7.198
7.184
7.155
6.826
6.796
5.512
5.362
4.767
4.751
4.234
4.211
4.187
4.179
4.177
4.163
4.153
4.141
4.136
4.118
4.098
4.074
4.063
4.051
4.040
3.807
3.133
3.114
3.084
3.066
2.981
2.964
2.933
2.917
1.249
1.238
1.226
1.214
1.203
1.190

cx-3-9802-h

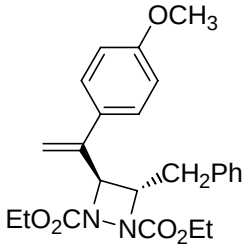

OCH3
CH2Ph
EtO2CN
NCO2Et

3p

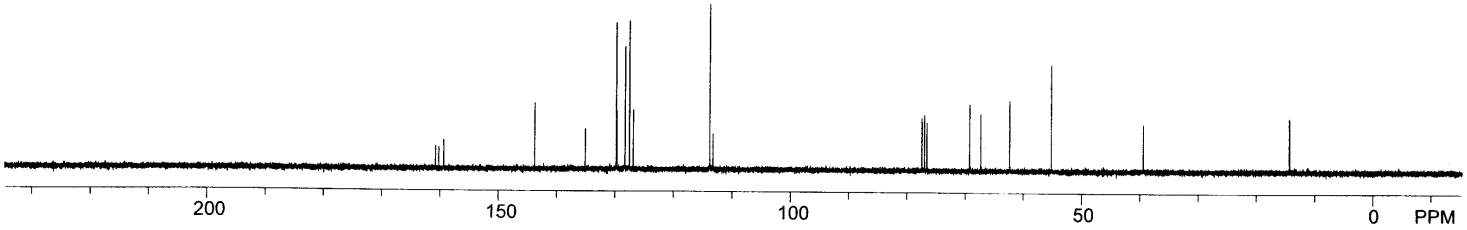

7.19
2.00
1.02
1.02
1.00
5.13
3.05
2.05
6.41
8
7
6
5
4
3
2
1
0
PPM

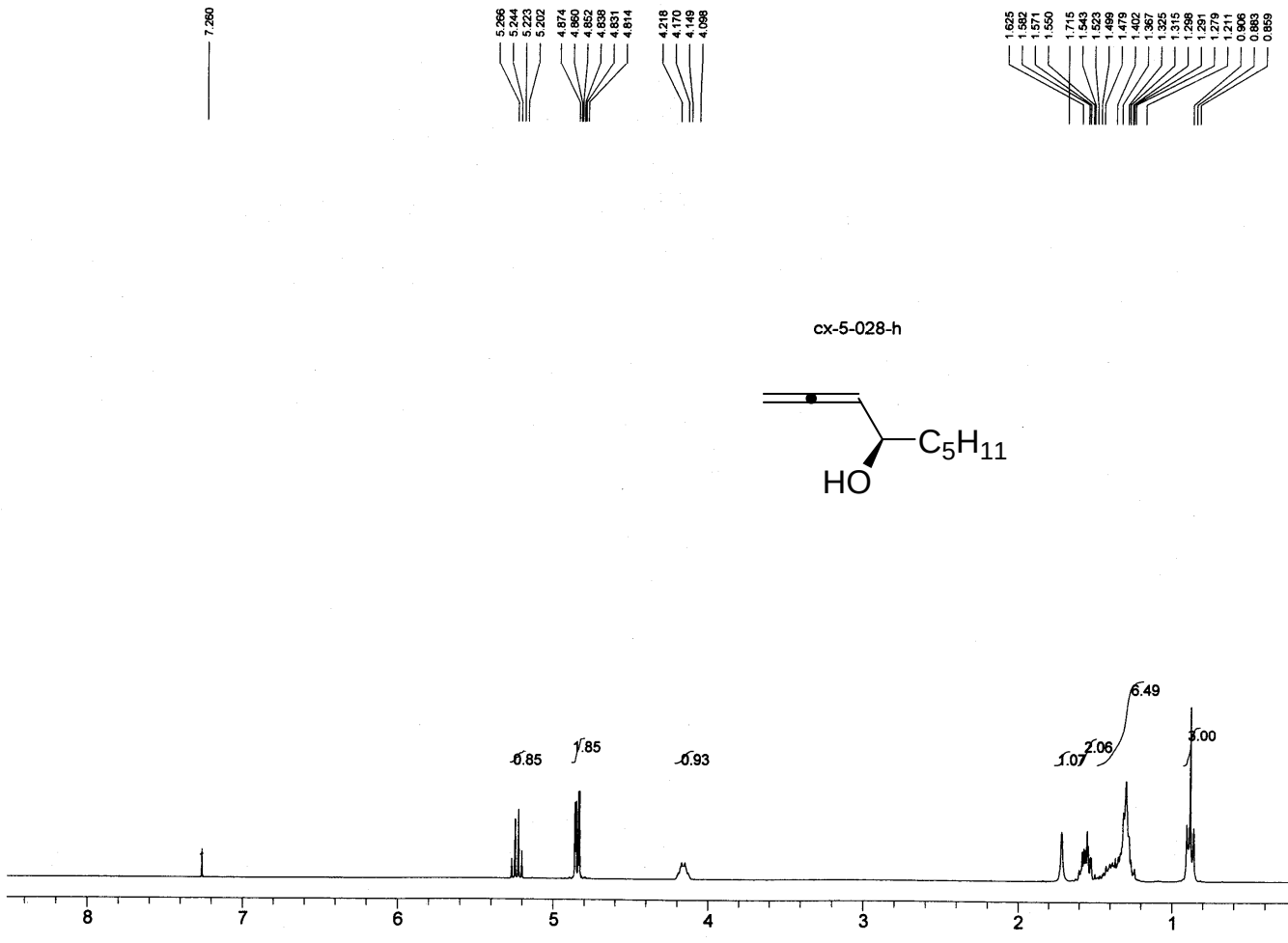

160.771
160.232
159.362
143.734
135.124
129.790
128.684
128.285
127.554
126.889
113.779
113.229
77.424
77.000
76.567
69.232
67.315
62.364
62.317
55.175
39.399
14.340
14.271

cx-3-9802-c


OCH3
CH2Ph
EtO2CN
NCO2Et

3p


200
150
100
50
0
PPM


7.260
5.266
5.244
5.223
5.202
4.874
4.860
4.852
4.838
4.831
4.814
4.218
4.170
4.149
4.098
1.625
1.592
1.571
1.550
1.715
1.543
1.523
1.499
1.479
1.402
1.367
1.325
1.315
1.298
1.291
1.279
1.211
0.906
0.883
0.859
cx-5-028-h
C5H11
HO
0.85
1.85
0.93
1.07
2.06
6.49
3.00
8
7
6
5
4
3
2
1
0
PPM

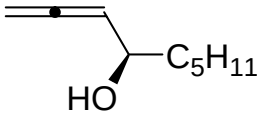
PERKIN–ELMER

Polarimeter 341
Serial No. 7413

RESULTS TABLE

Date: 11/09/2007 12:24:37

Operator: _____



Sample No.: 0001

Sample ID: _____

Sample Name: CX-5-028

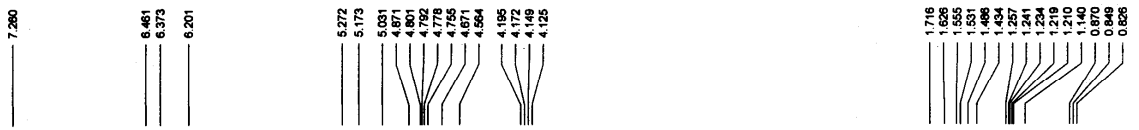
Comment: 21mg, c = 1.05
$CHCl_3$

Integration Time: 5.0 s

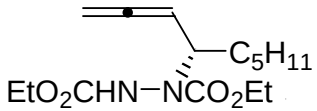
Cell Path: 100.00 mm

Sample	Wavelength	Temperature	Concentration	Rotation	Spec.Rotation
0001	589 nm	20.0	1.0500	-0.076	-7.2
0002	589 nm	20.0	1.0500	-0.076	-7.3
0003	589 nm	20.0	1.0500	-0.076	-7.2
0004	589 nm	20.0	1.0500	-0.076	-7.2
0005	589 nm	20.0	1.0500	-0.076	-7.2
0006	589 nm	20.0	1.0500	-0.076	-7.2
0007	589 nm	20.0	1.0500	-0.076	-7.2
0008	589 nm	20.0	1.0500	-0.074	-7.1
0009	589 nm	20.0	1.0500	-0.074	-7.1
0010	589 nm	20.0	1.0500	-0.075	-7.2

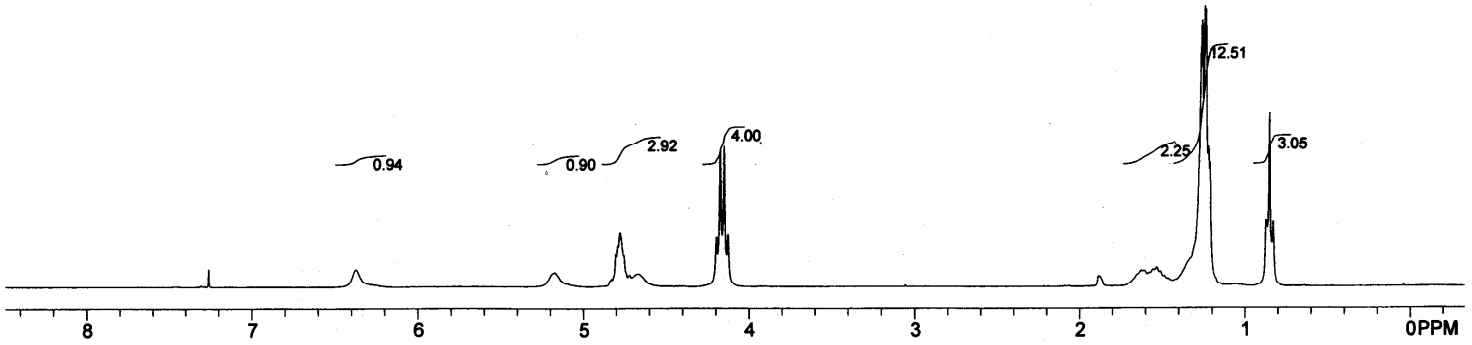
-7.2


7.260
6.461
6.373
6.201
5.272
5.173
5.031
4.871
4.801
4.792
4.778
4.755
4.671
4.584
4.195
4.172
4.149
4.125
1.716
1.626
1.555
1.531
1.486
1.434
1.257
1.241
1.234
1.219
1.210
1.140
0.870
0.849
0.826

cx-3-078-h


C5H11
EtO2CHN–NCO2Et

(S)-1a


0.94
0.90
2.92
4.00
2.25
12.51
3.05
8
7
6
5
4
3
2
1
0PPM

PERKIN–ELMER Polarimeter 341
Serial No. 7413

RESULTS TABLE Date: 10/31/2007 16:39:58

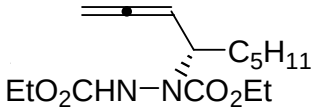
Operator: _____

Sample No.: 0001

Sample ID: _____

Sample Name: cx-3-078

Comment: $CHCl_3$



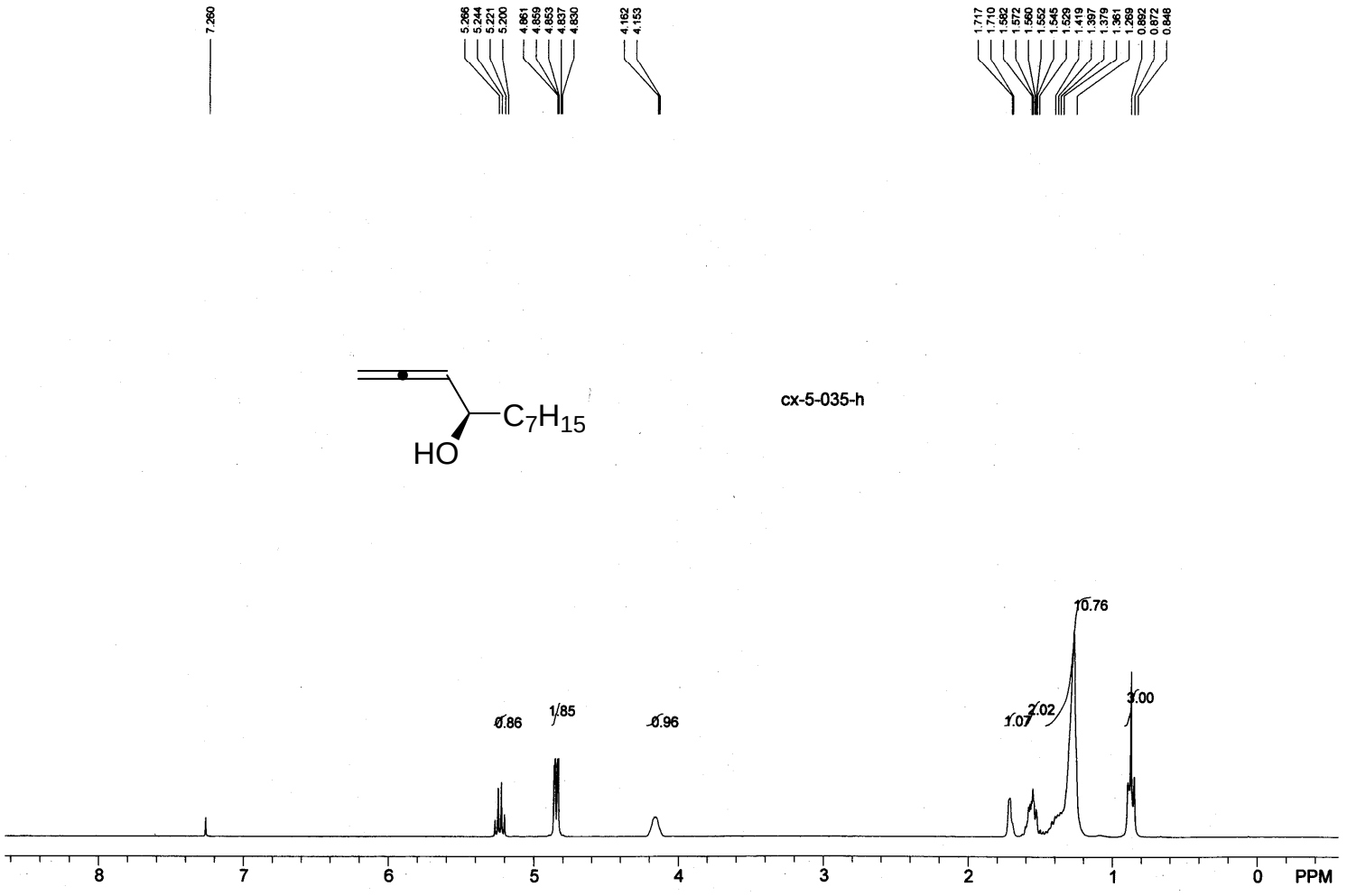
(S)-1a

Integration Time: 5.0 s

Cell Path: 100.00 mm

Sample	Wavelength	Temperature	Concentration	Rotation	Spec.Rotation
0001	589 nm	20.0	1.0000	-0.619	-61.9
0002	589 nm	20.0	1.0000	-0.620	-62.0
0003	589 nm	20.0	1.0000	-0.620	-62.0
0004	589 nm	20.0	1.0000	-0.620	-62.0
0005	589 nm	20.0	1.0000	-0.620	-62.0
0006	589 nm	20.0	1.0000	-0.618	-61.8
0007	589 nm	20.0	1.0000	-0.618	-61.8
0008	589 nm	20.0	1.0000	-0.618	-61.8
0009	589 nm	20.0	1.0000	-0.618	-61.8
0010	589 nm	20.0	1.0000	-0.618	-61.8

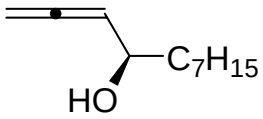
−61.9 c=1.00


7.260
5.266
5.244
5.221
5.200
4.861
4.859
4.853
4.837
4.830
4.162
4.153
1.717
1.710
1.582
1.572
1.560
1.552
1.545
1.529
1.419
1.397
1.379
1.361
1.269
0.892
0.872
0.848
C7H15
HO
cx-5-035-h
0.86
1.85
0.96
1.07
2.02
10.76
3.00
8
7
6
5
4
3
2
1
0
PPM

PERKIN–ELMER Polarimeter 341
Serial No. 7413

RESULTS TABLE Date: 11/13/2007 11:21:16

Operator: _____



Sample No.: 0001

Sample ID: _____

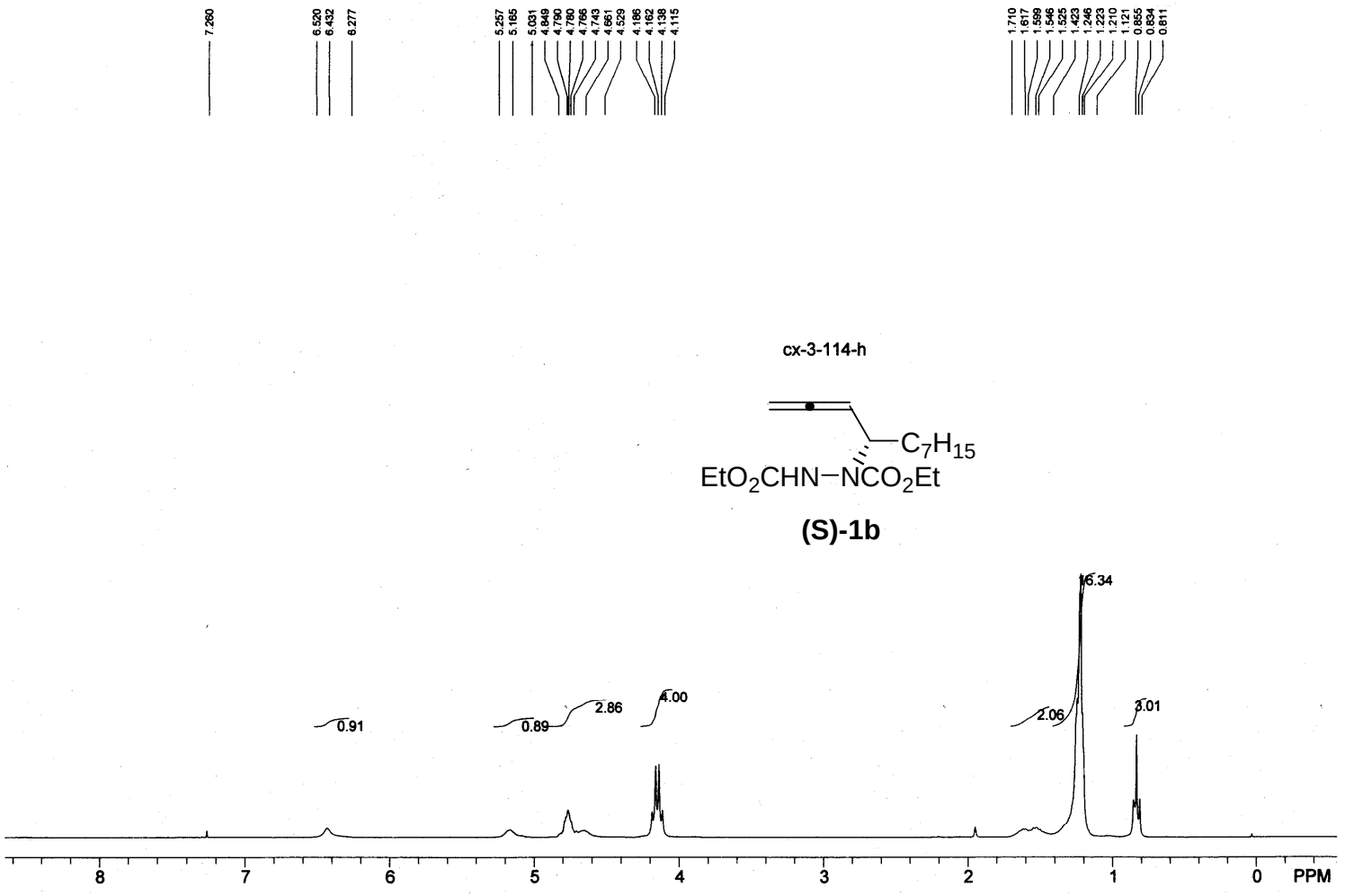
Sample Name: CX-6-035

Comment: 22mg, C=1.1, CHCl3

Integration Time: 5.0 s

Cell Path: 100.00 mm

Sample	Wavelength	Temperature	Concentration	Rotation	Spec.Rotation	
0001	589 nm	20.0	1.1000	-0.069	-6.2	
0002	589 nm	20.0	1.1000	-0.067	-6.0	
0003	589 nm	20.0	1.1000	-0.067	-6.0	
0004	589 nm	20.0	1.1000	-0.068	-6.2	
0005	589 nm	20.0	1.1000	-0.070	-6.4	−6.2
0006	589 nm	20.0	1.1000	-0.069	-6.2	
0007	589 nm	20.0	1.1000	-0.068	-6.1	
0008	589 nm	20.0	1.1000	-0.067	-6.1	
0009	589 nm	20.0	1.1000	-0.068	-6.2	
0010	589 nm	20.0	1.1000	-0.070	-6.3	


7.260
6.520
6.432
6.277
5.257
5.185
5.031
4.849
4.790
4.780
4.766
4.743
4.661
4.529
4.186
4.162
4.138
4.115
1.710
1.617
1.599
1.546
1.525
1.423
1.246
1.223
1.210
1.121
0.855
0.834
0.811
cx-3-114-h
C7H15
EtO2CHN−NCO2Et
(S)-1b
16.34
0.91
0.89
2.86
4.00
2.06
3.01
8
7
6
5
4
3
2
1
0
PPM

PERKIN–ELMER

Polarimeter 341
Serial No. 7413

RESULTS TABLE

Date: 10/31/2007 17:00:20

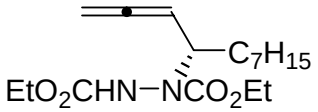
Operator: _____

Sample No.: 0002

Sample ID: _____

Sample Name: Ox-3-114

Comment: CHCl3



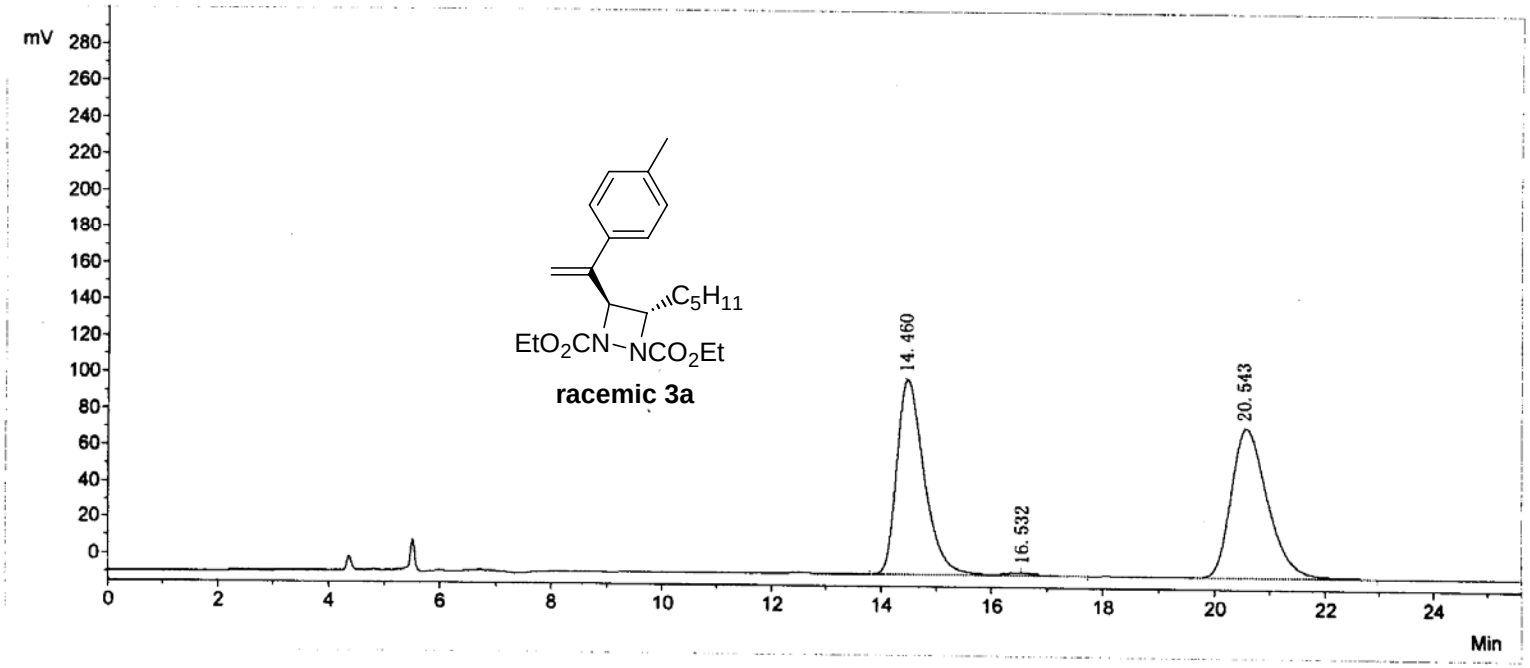
(S)-1b

Integration Time: 5.0 s

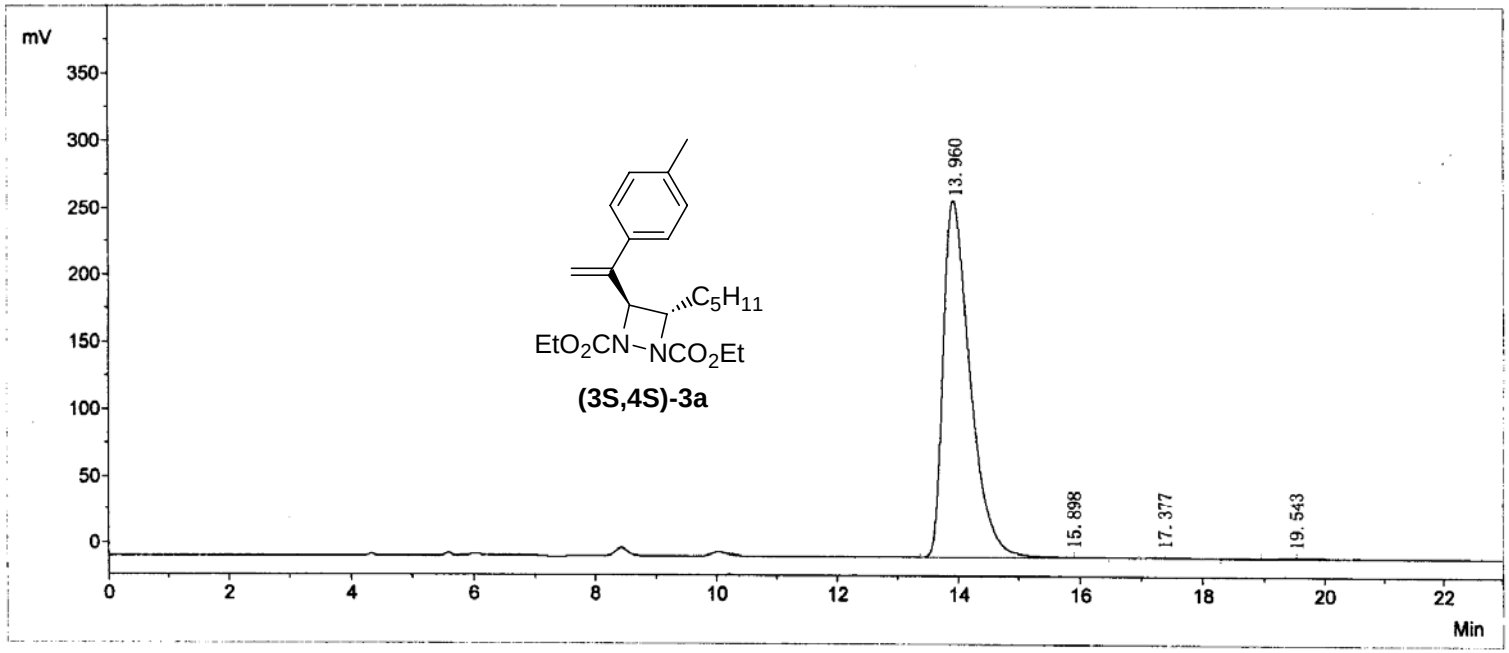
Cell Path: 100.00 mm

Sample	Wavelength	Temperature	Concentration	Rotation	Spec.Rotation
0002	589 nm	20.0	1.0000	-0.557	-55.7
0003	589 nm	20.0	1.0000	-0.556	-55.6
0004	589 nm	20.0	1.0000	-0.556	-55.6
0005	589 nm	20.0	1.0000	-0.556	-55.6
0006	589 nm	20.0	1.0000	-0.556	-55.6
0007	589 nm	20.0	1.0000	-0.556	-55.6
0008	589 nm	20.0	1.0000	-0.556	-55.6
0009	589 nm	20.0	1.0000	-0.556	-55.6
0010	589 nm	20.0	1.0000	-0.556	-55.6
0011	589 nm	20.0	1.0000	-0.556	-55.6

−55.6 c = 1.00



No.	R.Time	PeakHeight	PeakArea	PerCent
1	14.460	106056.4	3682125.9	49.2997
2	16.532	1319.8	48742.0	0.6526
3	20.543	81190.8	3737997.4	50.0477
Total		188567.0	7468865.3	100.0000



No.	R.Time	PeakHeight	PeakArea	PerCent
1	13.960	262453.1	8138530.1	99.3038 ✓
2	15.898	572.8	17795.3	0.2171
3	17.377	384.9	13020.7	0.1589
4	19.543	670.8	26239.9	0.3202 ✓
Total		264081.7	8195586.0	100.0000

99.4% ee

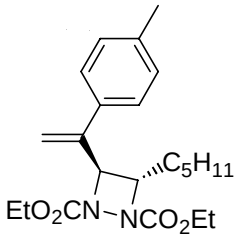
PERKIN–ELMER

Serial No. 7413

RESULTS TABLE

Date: 01/11/2007 11:14:19

Operator: _____



(3S,4S)-3a

Sample Name: cx-3-083

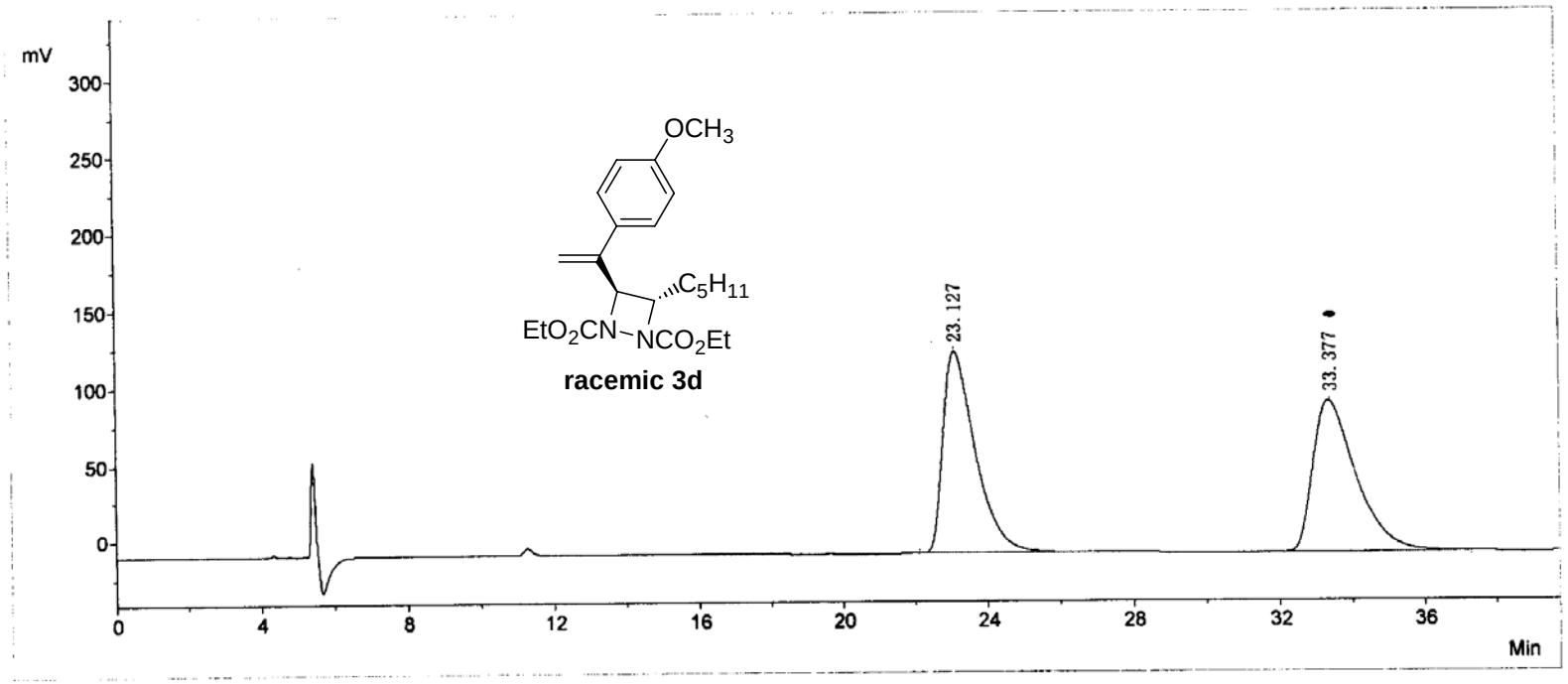
Comment: $CHCl_3$
c=1.05

Integration Time: 5.0 s

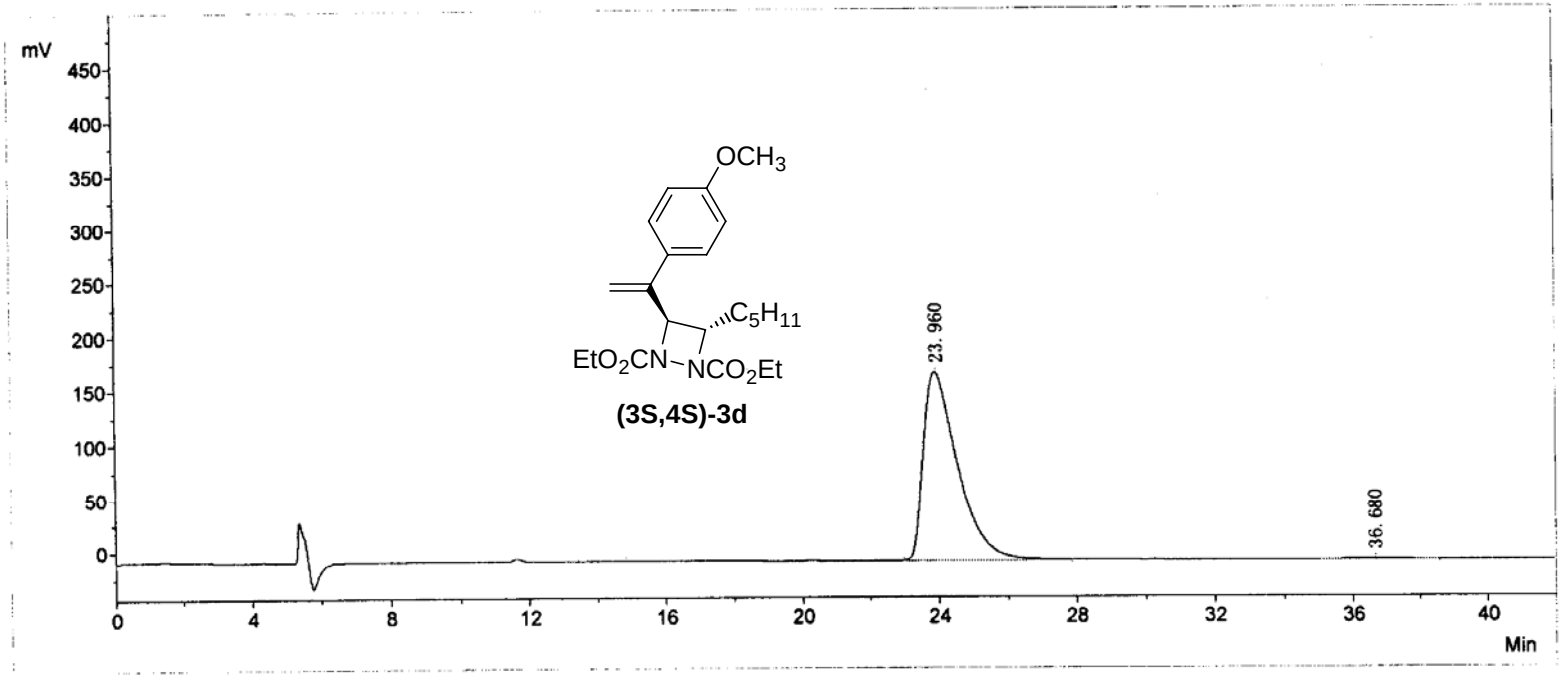
Cell Path: 100.00 mm

Sample	Wavelength	Temperature	Concentration	Rotation	Spec.Rotation
0009	589 nm	20.0°C	1.0500	+0.422°	+40.1 °
0010	589 nm	20.0°C	1.0500	+0.421°	+40.1 °
0011	589 nm	20.0°C	1.0500	+0.421°	+40.1 °
0012	589 nm	20.0°C	1.0500	+0.422°	+40.2 °
0013	589 nm	20.0°C	1.0500	+0.422°	+40.2 °

$[\alpha]_D^{20}$ +40.1°



No.	R.Time	PeakHeight	PeakArea	PerCent
1	23.127	130676.3	8109153.2	50.2699
2	33.377	97845.4	8022068.2	49.7301
Total		228521.7	16131221.4	100.0000

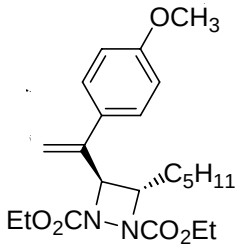


No.	R.Time	PeakHeight	PeakArea	PerCent	
1	23.960	173809.9	12078013.9	99.6832	
2	36.680	396.0	38385.1	0.3168	99.4%ee
Total		174205.9	12116399.0	100.0000	

Polarimeter 341

RESULTS TABLE

Operator: _____



(3S,4S)-3d

Sample ID: _____

Sample Name: CX-3-103

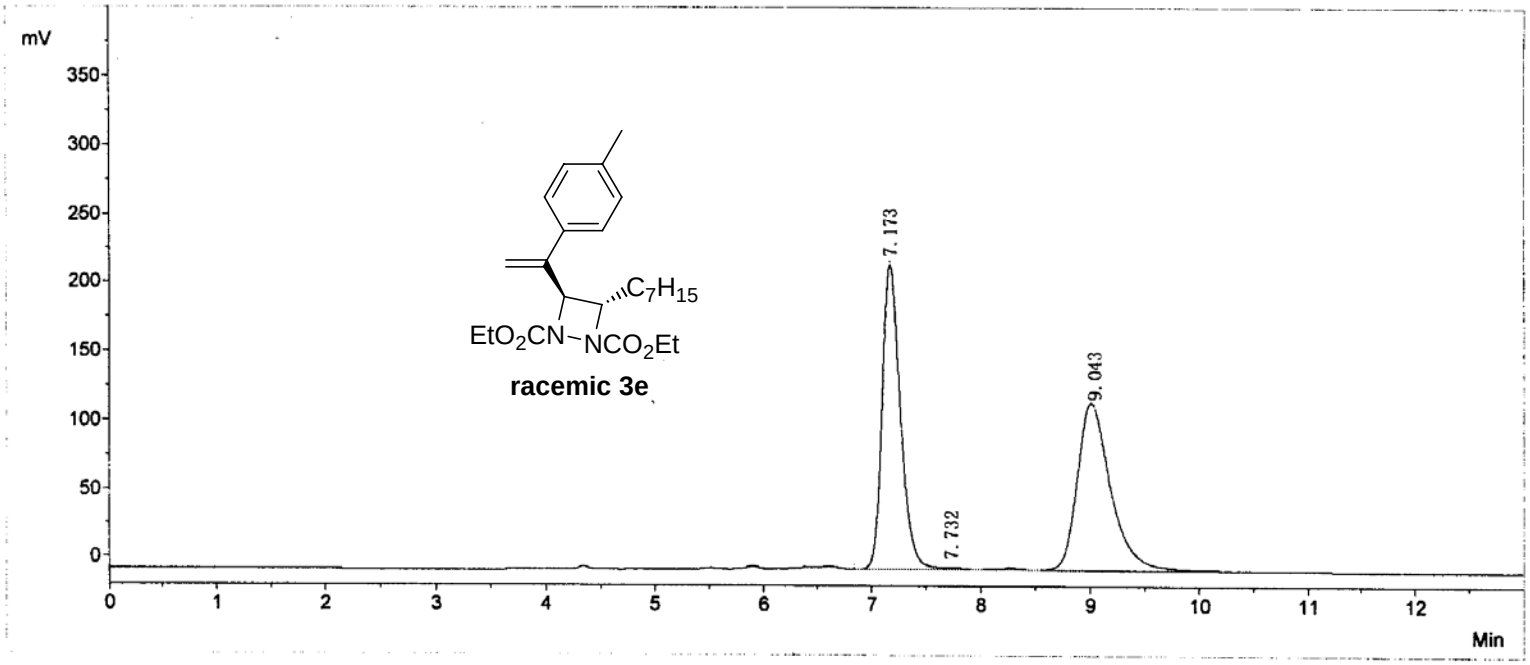
Comment: CHCl3
c=1.05

Integration Time: 5.0 s

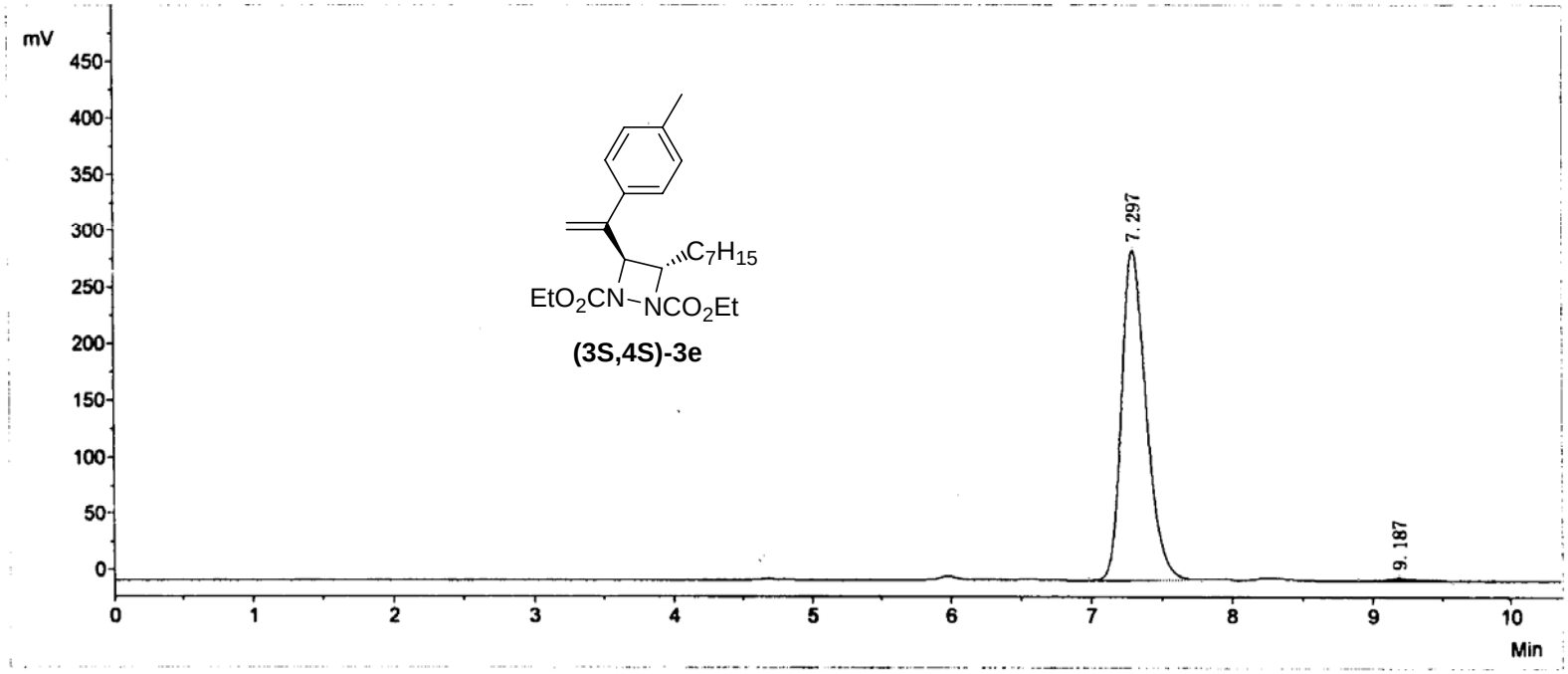
Cell Path: 100.00 mm

Sample	Wavelength	Temperature	Concentration	Rotation	Spec.Rotation	
0002	589 nm	20.0°C	1.0500	+0.420°	+40.0	°
0003	589 nm	20.0°C	1.0500	+0.421°	+40.1	°
0004	589 nm	20.0°C	1.0500	+0.421°	+40.1	°
0005	589 nm	20.0°C	1.0500	+0.420°	+40.0	°
0006	589 nm	20.0°C	1.0500	+0.421°	+40.1	°
0007	589 nm	20.0°C	1.0500	+0.421°	+40.1	°
0008	589 nm	20.0°C	1.0500	+0.421°	+40.1	°
0009	589 nm	20.0°C	1.0500	+0.421°	+40.1	°

+40.1°



No.	R.Time	PeakHeight	PeakArea	PerCent
1	7.173	220878.7	2585819.3	49.6723
2	7.732	1577.2	41938.9	0.8056
3	9.043	117723.6	2578003.6	49.5221
Total		340179.5	5205761.8	100.0000



No.	R.Time	PeakHeight	PeakArea	PerCent
1	7.297	291034.9	3511543.5	99.2354
2	9.187	1872.1	27055.7	0.7646
Total		292907.0	3538599.2	100.0000

98.5% ee

Polarimeter 341

RESULTS TABLE

Operator: _____

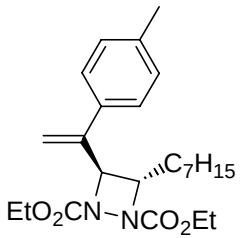
Sample No.: 0001

Sample ID: _____

ex-2-119

Comment: CH$_2$Cl$_2$

c = 1.05

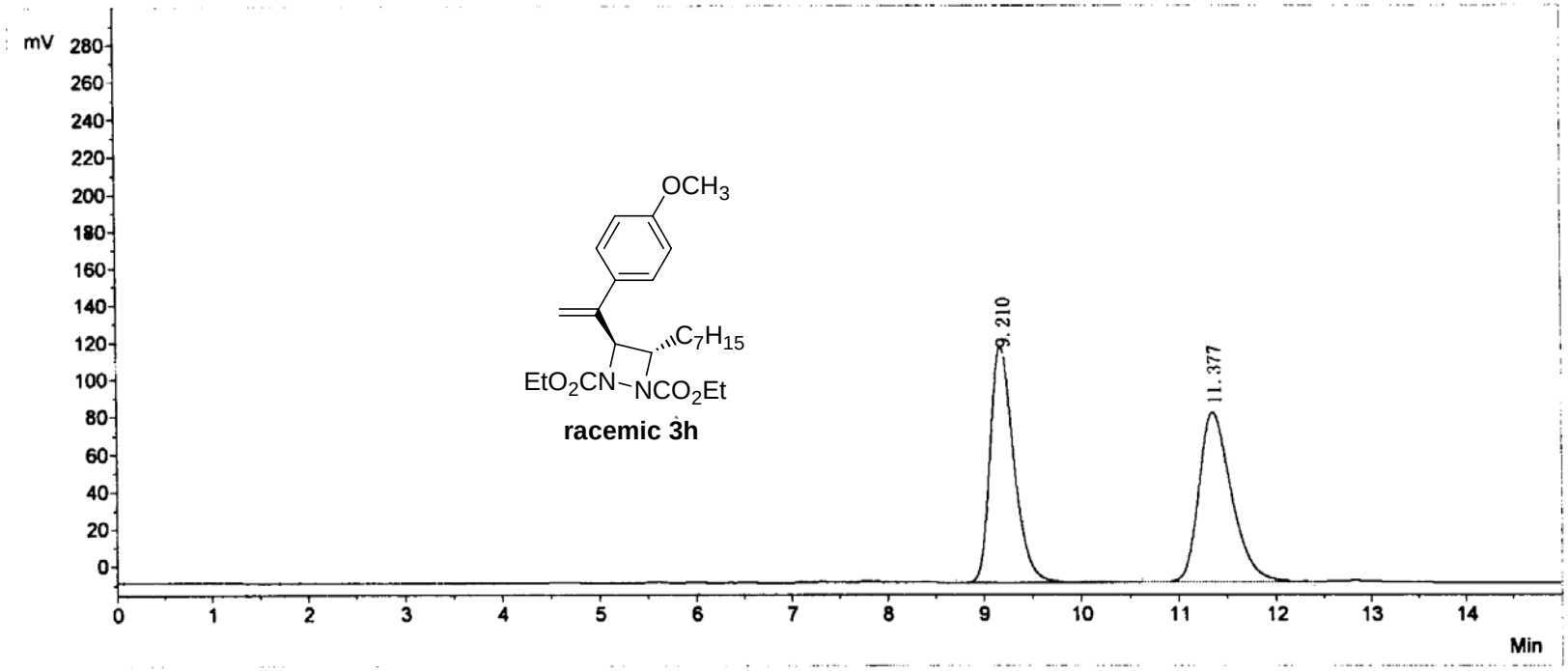


(3S,4S)-3e

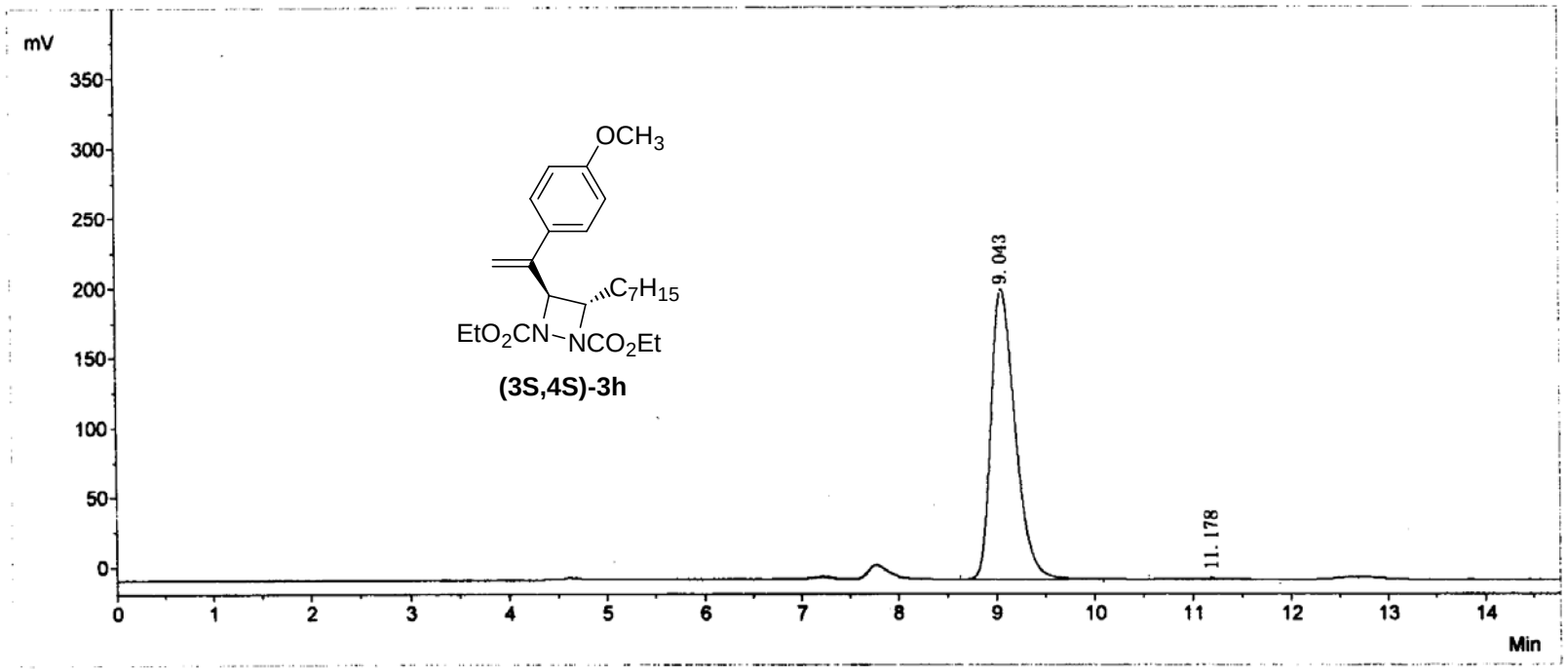
Integration Time: 5.0 s

Cell Path: 100.00 mm

Sample	Wavelength	Temperature	Concentration	Rotation	Spec.Rotation	
0001	589 nm	20.0°C	1.0500	+0.367°	+34.9	°
0002	589 nm	20.0°C	1.0500	+0.372°	+35.4	°
0003	589 nm	20.0°C	1.0500	+0.372°	+35.4	°
0004	589 nm	20.0°C	1.0500	+0.374°	+35.6	° +35.5°
0005	589 nm	20.0°C	1.0500	+0.374°	+35.6	°
0006	589 nm	20.0°C	1.0500	+0.374°	+35.6	°
0007	589 nm	20.0°C	1.0500	+0.374°	+35.6	°
0008	589 nm	20.0°C	1.0500	+0.374°	+35.6	°



No.	R.Time	PeakHeight	PeakArea	PerCent
1	9.210	120796.7	2142405.4	50.2710
2	11.377	90023.4	2119305.8	49.7290
Total		210820.0	4261711.2	100.0000



No.	R.Time	PeakHeight	PeakArea	PerCent
1	9.043	204445.2	3545900.2	99.2547
2	11.178	703.8	26625.2	0.7453
Total		205149.0	3572525.4	100.0000

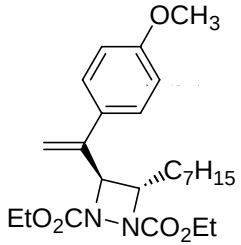
98.5% ee

Polarimeter 341

RESULTS TABLE

Date: 01/11/2007 12:14:53

Operator: _____



(3S,4S)-3h

Sample Name: CX-3-120

Comment: CHCl3
C=1.05

Integration Time: 5.0 s

Cell Path: 100.00 mm

Sample	Wavelength	Temperature	Concentration	Rotation	Spec.Rotation	
0002	589 nm	20.0°C	1.0500	+0.367°	+34.9	°
0003	589 nm	20.0°C	1.0500	+0.368°	+35.0	°
0004	589 nm	20.0°C	1.0500	+0.367°	+35.0	°
0005	589 nm	20.0°C	1.0500	+0.367°	+35.0	° +34.9°
0006	589 nm	20.0°C	1.0500	+0.365°	+34.8	°
0007	589 nm	20.0°C	1.0500	+0.365°	+34.8	°
0008	589 nm	20.0°C	1.0500	+0.366°	+34.8	°
0009	589 nm	20.0°C	1.0500	+0.366°	+34.8	°