Organocatalytic Chemoselective Asymmetric *N*-Allylic Alkylation of Enamides

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1. General Methods:

NMR spectra were recorded with tetramethylsilane as the internal standard. TLC was performed on glass-backed silica plates. Column chromatography was performed using silica gel (200-300 mesh) eluting with ethyl acetate and petroleum ether (EtOAc/PE). ^{1}H NMR spectra were recorded at 400 MHz (Varian) and ^{13}C NMR spectra were recorded at 100 or 50 MHz (Varian). Chemical shifts are reported in ppm downfield from $CDCl_3$ (δ = 7.27 ppm) for ^{1}H NMR and relative to the central $CDCl_3$ resonance (δ = 77.0 ppm) for ^{13}C NMR spectroscopy. Coupling constants are given in Hz. Optical rotations were measured at 589 nm at 20 ºC. Enantiomeric excess was determined by HPLC analysis on Chiralpak AS, AD and Chiralcel OD columns in comparison with authentic racemic samples. Toluene was distilled from CaH_2. All other chemicals were used without purification as commercially available. Cinchona alkaloid catalysts $(DHQD)_2PHAL$, $(DHQD)_2PYR$, $(DHQD)_2AQN$ and $(DHQ)_2AQN$ were purchased from Aldrich Chemical Company. Morita–Baylis–Hillman carbonates were prepared according to the literature.[1] Enamides **1a**[2]**, 1d–1k**[2] , **1b-1c**[3], **1h**[4] and **1i**[5] were prepared according to the reported methods.

(1) J. Feng, X. Lu, A. Kong and X. Han, *Tetrahedron*, 2007, **63**, 6035.

(2) C. Sun and S. M. Weinreb, *Synthesis*, 2006, **21**, 3585.

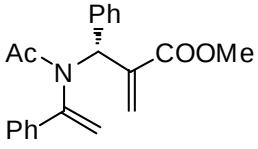
(3) H. Kiyohara, R. Matsubara and S. Kobayashi, *Org. Lett*., 2006, **8**, 5333.

(4) G. Zhu, Z. Chen and X. Zhang, *J. Org. Chem*., 1999, **64**, 6907.

(5) B. S. Jursic, S. Sagiraju, D. K. Ancalade, T. Clark, E. D. Stevens, *Synth. Commun*., 2007, **37**, 1709.

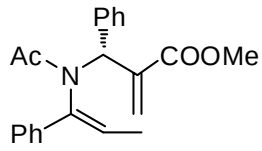
2. General procedure for asymmetric *N*-allylic alkylation reaction

To a solution of **1a** (32.2 mg, 0.2 mmol) and $(DHQD)_2AQN$ (8.6 mg, 5 mol%) in dry toluene (1.0 mL) was added **2a** (116.8 mg, 0.4 mmol). The reaction mixture was stirred at 50 ºC until consumption of **1a**, which was monitored by TLC. Flash chromatography on silica gel (EtOAc/petroleum ether = 1/5) give **3a** as a colourless oil (45.6 mg, 68%).

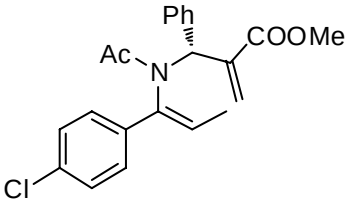


3a, 68% yield; $[\alpha]_D^{20}$ = -37.0 (*c* = 1.75 in $CHCl_3$); 85% ee, determined by HPLC analysis [Daicel Chiralpak AD, *n*-hexane/*i*-PrOH = 80/20, 1.0 mL/min, λ = 254 nm, t (major) = 11.154 min, t (minor) = 7.511 min]; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.26-7.11 (m, 10H), 6.40 (s, 1H), 6.39 (s, 1H), 5.72 (s, 1H), 5.66 (s, 1H), 5.30 (s, 1H), 3.62 (s, 3H), 2.08 (s, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 171.0, 166.6, 146.9, 139.6, 137.4, 136.7, 129.8, 128.6,

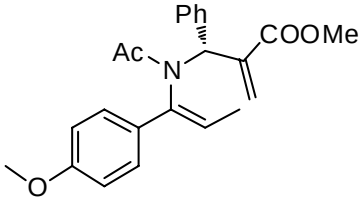
128.3, 127.7, 127.5, 126.6, 125.9, 115.8, 61.9, 51.9, 23.4 ppm; ESI-HRMS: calcd. for $C_{21}H_{21}NO_3$+Na 358.1419, found 358.1424.



3b, 71% yield; $[\alpha]_D^{20}$ = +13.6 (*c* = 0.22 in $CHCl_3$); 82% ee, determined by HPLC analysis [Daicel Chiralpak AD, *n*-hexane/*i*-PrOH = 80/20, 1.0 mL/min, λ = 254 nm, t (major) = 8.280 min, t (minor) = 6.804 min]; ^{1}H NMR (400 MHz, $CDCl_3$): major rotational isomer: δ = 7.30-7.06 (m, 5.67H), 6.39 (s, 0.57H), 6.28 (s, 0.57H), 6.23 (s, 0.57H), 5.90 (q, *J* = 6.8 Hz, 0.57H), 3.60 (s, 1.71H), 2.16 (s, 1.71H), 1.51 (d, *J* = 6.8 Hz, 1.71H) ppm; minor rotational isomer: δ = 7.30-7.06 (m, 4.33H), 5.83 (s, 0.43H), 5.82-5.77 (m, 0.43H), 5.76 (s, 0.43H), 5.67 (s, 0.43H), 3.66 (s, 1.29H), 2.14 (s, 1.29H), 1.74 (d, *J* = 7.2 Hz, 1.29H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): major rotational isomer: δ = 171.7, 166.6, 155.8, 141.3, 139.2, 136.1, 129.9, 128.3, 128.0, 127.8, 127.5, 126.6, 126.3, 125.0, 62.3, 51.9, 22.5, 14.6 ppm; minor rotational isomer: δ = 170.4, 162.5, 152.8, 140.1, 137.2, 130.2, 128.5, 128.2, 127.9, 127.6, 127.4, 126.4, 125.8, 111.3, 61.2, 51.8, 22.4, 14.6 ppm; ESI-HRMS: calcd. for $C_{22}H_{23}NO_3$+Na 372.1576, found 372.1571.

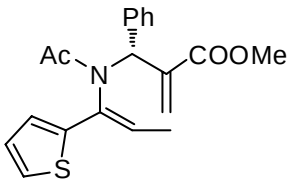


3c, 94% yield; $[\alpha]_D^{20}$ = -52.7 (*c* = 1.30 in $CHCl_3$); 87% ee, determined by HPLC analysis [Daicel Chiralpak AD, *n*-hexane/*i*-PrOH = 70/30, 1.0 mL/min, λ = 254 nm, t (major) = 5.930 min, t (minor) = 5.297 min]; ^{1}H NMR (400 MHz, $CDCl_3$): major rotational isomer: δ = 7.26-7.04 (m, 4.68H), 6.36 (s, 0.52H), 6.26 (s, 0.52H), 6.25 (s, 0.52H), 5.88 (q, *J* = 7.2 Hz, 0.52H), 3.60 (s, 1.56H), 2.13 (s, 1.56H), 1.52 (d, *J* = 6.8 Hz, 1.56H) ppm; minor rotational isomer: δ = 7.26-7.04 (m, 4.32H), 5.81-5.77 (m, 0.96H), 5.69 (s, 0.48H), 5.64 (s, 0.48H), 3.66 (s, 1.44H), 2.11 (s, 1.44H), 1.71 (d, *J* = 6.8 Hz, 1.44H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): major rotational isomer: δ = 171.6, 166.4, 140.3, 139.7, 137.8, 137.0, 133.9, 129.9, 128.5, 128.0, 127.7, 127.6, 127.0, 124.9, 62.3, 51.9, 22.5, 14.6 ppm; minor rotational isome: δ = 171.1, 166.4, 140.1, 139.2, 137.1, 135.9, 133.9, 130.1, 129.1, 128.4, 127.9, 127.7, 127.1, 126.3, 61.2, 51.9, 22.4, 14.5 ppm; ESI-HRMS: calcd. for $C_{22}H_{22}ClNO_3$+Na 406.1186, found 406.1188.

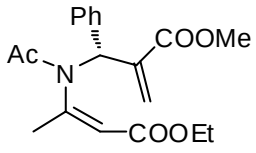


3d, 63% yield; $[\alpha]_D^{20}$ = -56.4 (*c* = 2.40 in $CHCl_3$); 86% ee, determined by HPLC analysis [Daicel Chiralpak AS, *n*-hexane/*i*-PrOH = 80/20, 1.0 mL/min, λ = 254 nm, t (major) = 8.638 min, t (minor) = 5.927 min]; ^{1}H NMR (400 MHz, $CDCl_3$): major rotational isomer: δ = 7.27-7.04 (m,

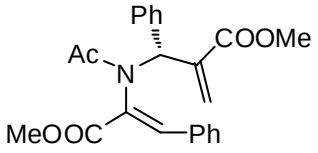
3.92H), 6.83-6.76 (m, 1.12H), 6.34 (s, 0.56H), 6.27 (s, 0.56H), 6.25 (s, 0.56H), 5.78 (q, *J* = 6.8 Hz, 0.56H), 3.82 (s, 1.68H), 3.60 (s, 1.68H), 2.15 (s, 1.68H), 1.50 (d, *J* = 7.2 Hz, 1.68H) ppm; minor rotational isomer: δ = 7.27-7.04 (m, 3.08H), 6.83-6.76 (m, 0.88H), 5.84 (s, 0.44H), 5.71 (s, 0.44H), 5.67 (q, *J* = 7.2 Hz, 0.44H), 5.64 (s, 0.44H), 3.80 (s, 1.32H), 3.66 (s, 1.32H), 2.12 (s, 1.32H), 1.68 (d, *J* = 7.2 Hz, 1.32H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): major rotational isomer: δ = 171.7, 166.5, 159.5, 140.8, 140.1, 137.4, 131.8, 130.2, 127.9, 127.6, 127.4, 126.8, 125.1, 113.7, 62.3, 55.3, 51.9, 22.5, 14.5 ppm; minor rotational isomer: δ = 171.2, 166.5, 159.5, 140.2, 139.4, 136.1, 131.1, 129.8, 127.8, 127.5, 127.2, 126.1, 124.7, 113.5, 61.3, 55.3, 51.8, 22.4, 14.3 ppm; ESI-HRMS: calcd. for $C_{23}H_{26}NO_4$+H 380.1862, found 380.1867.



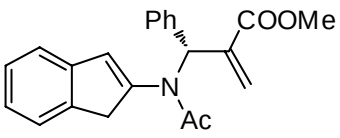
3e, 59% yield; $[\alpha]_D^{20}$ = -63.7 (*c* = 2.10 in $CHCl_3$); 89% ee, determined by HPLC analysis [Daicel Chiralpak AD, *n*-hexane/*i*-PrOH = 80/20, 1.0 mL/min, λ = 254 nm, t (major) = 8.607 min, t (minor) = 6.613 min]; ^{1}H NMR (400 MHz, $CDCl_3$): major rotational isomer: δ = 7.27-7.08 (m, 3.24H), 6.95-6.90 (m, 0.54H), 6.84-6.83 (m, 0.54H), 6.35 (s, 0.54H), 6.34 (s, 0.54H), 6.30 (s, 0.54H), 5.96-5.91 (m, 0.54H), 3.62 (s, 1.62H), 2.16 (s, 1.62H), 1.54 (d, *J* = 6.8 Hz, 1.62H) ppm; minor rotational isomer: δ = 7.27-7.08 (m, 2.76H), 6.95-6.90 (m, 0.92H), 5.98 (s, 0.46H), 5.88-5.82 (m, 0.92H), 5.77 (s, 0.46H), 3.65 (s, 1.38H), 2.10 (s, 1.38H), 1.69 (d, *J* = 7.2 Hz, 1.38H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): major rotational isomer: δ = 174.2, 166.5, 143.6, 139.7, 136.8, 135.9, 129.9, 128.8, 127.8, 127.6, 127.2, 125.8, 124.8, 124.5, 62.5, 51.8, 22.6, 14.2 ppm; minor rotational isomer: δ = 171.2, 166.5, 143.1, 139.2, 136.0, 135.2, 129.8, 128.3, 127.7, 127.5, 127.1, 125.0, 124.7, 124.2, 61.8, 51.8, 22.4, 14.0 ppm; ESI-HRMS: calcd. for $C_{20}H_{21}NO_3S$+Na 378.1140, found 378.1149.



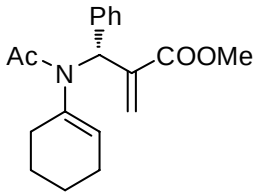
3f, 52% yield; $[\alpha]_D^{20}$ = -11.22 (*c* = 1.80 in CH_2Cl_2); 87% ee, determined by HPLC analysis [Daicel Chiralpak AD, *n*-hexane/*i*-PrOH = 80/20, 1.0 mL/min, λ = 254 nm, t (major) = 7.478 min, t (minor) = 6.691 min]; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.34-7.24 (m, 5H), 6.46 (s, 1H), 6.23 (s, 1H), 5.73 (s, 1H), 5.59 (s, 1H), 4.13 (q, *J* = 7.2 Hz, 2H), 3.71 (s, 3H), 2.14 (s, 3H), 2.12 (s, 3H), 1.26 (t, *J* = 7.2 Hz, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 169.0, 166.5, 165.4, 154.8, 139.2, 137.0, 128.8, 128.4, 127.9, 127.7, 122.1, 61.9, 60.3, 52.1, 23.2, 21.1, 14.1 ppm; ESI-HRMS: calcd. for $C_{19}H_{23}NO_5$+Na 368.1474, found 368.1469.



3g, 92% yield; $[\alpha]_D^{20}$ = -222.0 (*c* = 3.60 in $CHCl_3$); 84% ee, determined by HPLC analysis [Daicel Chiralpak AD, *n*-hexane/*i*-PrOH = 80/20, 1.0 mL/min, λ = 254 nm, t (major) = 14.599 min, t (minor) = 8.522 min]; ^{1}H NMR (400 MHz, $CDCl_3$): major rotational isomer: δ = 7.57-6.91 (m, 9.02H), 6.68 (s, 0.82H), 6.51 (s, 0.82H), 6.22 (s, 0.82H), 3.78 (s, 2.46H), 3.60 (s, 2.46H), 1.94 (s, 2.46H) ppm; minor rotational isomer: δ = 7.57-6.91 (m, 1.98H), 6.24 (s, 0.18H), 6.18 (s, 0.18H), 5.52 (s, 0.18H), 3.74 (s, 0.54H), 3.44 (s, 0.54H), 2.05 (s, 0.54H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): major rotational isomer: δ = 170.4, 166.5, 166.2, 140.5, 138.1, 134.8, 131.0, 130.3, 129.8, 128.8, 128.7, 128.3, 127.7, 125.0, 62.0, 52.4, 51.6, 22.7 ppm; minor rotational isomer: δ = 170.4, 166.5, 166.2, 139.8, 137.6, 132.3, 130.8, 130.1, 129.2, 128.8, 128.5, 127.8, 127.7, 127.3, 62.8, 52.4, 52.4, 22.7 ppm; ESI-HRMS: calcd. for $C_{23}H_{23}NO_5$+Na 416.1474, found 416.1474.

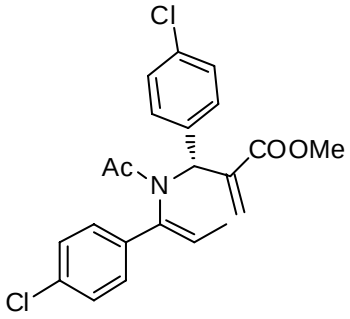


3h, 68% yield; $[\alpha]_D^{20}$ = -11.88 (*c* = 1.65 in CH_2Cl_2); 82% ee, determined by HPLC analysis [Daicel Chiralcel OD, *n*-hexane/*i*-PrOH = 80/20, 1.0 mL/min, λ = 254 nm, t (major) = 15.194 min, t (minor) = 13.537 min]; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.37-7.15 (m, 9H), 6.63 (s, 1H), 6.54 (s, 1H), 6.43 (s, 1H), 5.66 (s, 1H), 3.69 (s, 3H), 3.27 (d, *J* = 22.8 Hz, 1H), 2.97 (d, *J* = 22.4 Hz, 1H), 2.72 (s, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 170.2, 166.6, 145.8, 141.9, 141.0, 139.6, 137.5, 128.6, 128.5, 127.8, 127.5, 127.3, 126.7, 125.6, 123.6, 121.6, 61.1, 52.1, 41.2, 23.2 ppm; ESI-HRMS: calcd. for $C_{22}H_{21}NO_3$+Na 370.1419, found 370.1413.

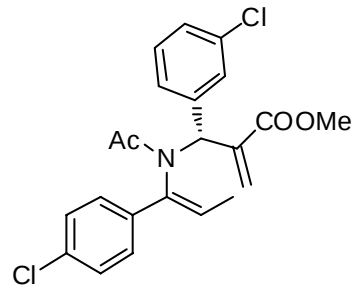


3i, 26% yield; $[\alpha]_D^{20}$ = -47.31 (*c* = 1.60 in $CHCl_3$); 69% ee, determined by HPLC analysis [Daicel Chiralpak AD, *n*-hexane/*i*-PrOH = 80/20, 1.0 mL/min, λ = 220 nm, t (major) = 9.197 min, t (minor) = 6.617 min]; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.30-7.21 (m, 5H), 6.40 (s, 1H), 6.31 (s, 1H), 5.60 (brs, 2H), 3.65 (s, 3H), 2.09 (s, 3H), 1.96-1.26 (m, 8H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 169.9, 166.8, 137.7, 130.7, 129.3, 128.0, 127.5, 126.2, 61.0, 51.9, 30.7, 24.9, 22.6, 22.5, 21.1 ppm; ESI-HRMS: calcd. for $C_{19}H_{23}NO_3$+Na 336.1576, found 336.1571.

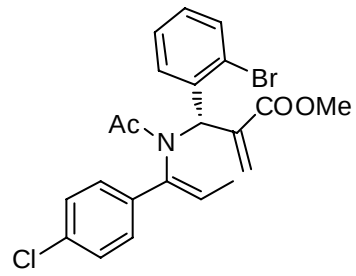
3j, 94% yield; $[\alpha]_D^{20}$ = -67.69 (*c* = 2.28 in $CHCl_3$); 90% ee, determined by HPLC analysis [Daicel Chiralpak AD, *n*-hexane/*i*-PrOH = 80/20, 1.0 mL/min, λ = 254 nm, t (major) = 8.017 min, t (minor) = 7.156 min]; ^{1}H NMR (400 MHz, $CDCl_3$): major rotational isomer: δ = 7.30-7.01 (m, 4.16H), 6.30 (s, 0.52H), 6.29 (s, 0.52H), 6.27 (s, 0.52H), 5.93 (q, *J* = 7.2 Hz, 0.52H), 3.62 (s, 1.56H), 2.14 (s, 1.56H), 1.58



(d, *J* = 7.2 Hz, 1.56H) ppm; minor rotational isomer: δ = 7.30-7.01 (m, 3.84H), 5.85-5.82 (m, 0.96H), 5.69 (s, 0.48H), 5.66 (s, 0.48H), 3.68 (s, 1.44H), 2.12 (s, 1.44H), 1.74 (d, *J* = 7.2 Hz, 1.44H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): major rotational isomer: δ = 171.6, 166.3, 140.2, 139.6, 137.5, 135.7, 134.1, 133.8, 131.3, 128.9, 128.5, 127.8, 127.2, 126.6, 61.7, 52.0, 22.4, 14.7 ppm; minor rotational isomer: δ = 171.1, 166.1, 139.7, 138.8, 136.9, 134.7, 133.9, 133.5, 131.1, 128.6, 127.9, 127.8, 127.1, 125.3, 60.8, 51.9, 22.3, 14.5 ppm; ESI-HRMS: calcd. for $C_{22}H_{21}Cl_2NO_3$+Na 440.0796, found 440.0789.

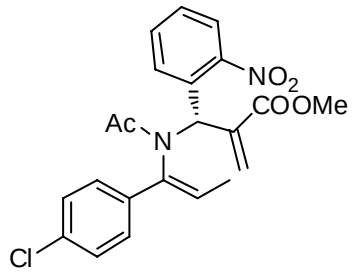


3k, 78% yield; $[\alpha]_D^{20}$ = -51.29 (*c* = 1.70 in $CHCl_3$); 82% ee, determined by HPLC analysis [Daicel Chiralcel OD, *n*-hexane/*i*-PrOH = 80/20, 1.0 mL/min, λ = 254 nm, t (major) = 6.253 min, t (minor) = 14.444 min]; ^{1}H NMR (400 MHz, $CDCl_3$): major rotational isomer: δ = 7.28-6.95 (m, 4.0H), 6.30 (s, 0.5H), 6.29 (s, 0.5H), 6.27 (s, 0.5H), 5.93-5.90 (m, 0.5H), 3.67 (s, 1.5H), 2.14 (s, 1.5H), 1.73 (d, *J* = 6.8 Hz, 1.5H) ppm; minor rotational isomer: δ = 7.28-6.95 (m, 4.0H), 5.83-5.80 (m, 1.0H), 5.73 (s, 0.5H), 5.63 (s, 0.5H), 3.61 (s, 1.5H), 2.11 (s, 1.5H), 1.56 (d, *J* = 6.8 Hz, 1.5H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): major rotational isomer: δ = 171.6, 166.3, 140.1, 139.3, 138.2, 136.9, 133.5, 130.0, 129.1, 128.9, 128.6, 128.2, 127.8, 127.4, 126.9, 125.9, 61.6, 52.1, 22.4, 14.7 ppm; minor rotational isomer: δ = 171.1, 166.1, 139.7, 138.5, 137.6, 134.1, 134.0, 133.5, 129.8, 129.1, 128.6, 128.2, 127.9, 127.8, 127.2, 125.9, 60.8, 52.0, 22.3, 14.6 ppm; ESI-HRMS: calcd. for $C_{22}H_{21}Cl_2NO_3$+Na 440.0796, found 440.0791.

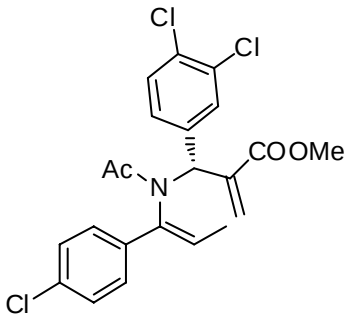


3l, 99% yield; $[\alpha]_D^{20}$ = +146.1 (*c* = 3.30 in $CHCl_3$); 87% ee, determined by HPLC analysis [Daicel Chiralpak AD, *n*-hexane/*i*-PrOH = 80/20, 1.0 mL/min, λ = 254 nm, t (major) = 7.483 min, t (minor) = 5.630 min]; ^{1}H NMR (400 MHz, $CDCl_3$): major rotational isomer: δ = 7.54-7.00 (m, 5.28H), 6.18 (s, 0.66H), 6.13 (s, 0.66H), 5.81 (q, *J* = 6.8 Hz, 0.66H), 5.24 (s, 0.66H), 3.67 (s, 1.98H), 2.11 (s, 1.98H), 1.60 (d, *J* = 6.8 Hz, 1.98H) ppm; minor rotational isomer: δ = 7.54-7.00 (m, 2.72H), 6.69 (s, 0.34H), 6.25 (s, 0.34H), 5.84-5.79 (m, 0.34H), 5.61 (s, 0.34H), 3.54 (s, 1.02H), 2.14 (s, 1.02H), 1.74 (d, *J* = 6.8 Hz, 1.02H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): major rotational

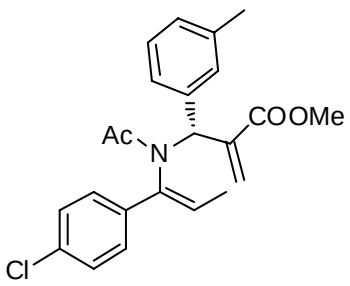
isomer: δ = 171.2, 166.1, 140.0, 138.8, 137.5, 136.8, 133.7, 132.5, 130.7, 129.7, 128.9, 128.2, 127.7, 126.6, 126.3, 124.9, 60.9, 51.8, 22.1, 14.4 ppm; minor rotational isomer: δ = 171.3, 165.9, 139.6, 138.6, 137.8, 136.6, 133.5, 132.7, 131.3, 129.1, 128.9, 128.2, 128.1, 126.9, 126.5, 125.5, 61.2, 51.7, 22.3, 15.3 ppm; ESI-HRMS: calcd. for $C_{22}H_{21}BrClNO_3$+Na 484.0291, found 484.0296.



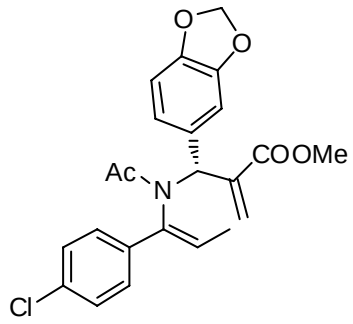
3m, 70% yield; $[\alpha]_D^{20}$ = +128.2 (*c* = 1.48 in $CHCl_3$); 92% ee, determined by HPLC analysis [Daicel Chiralpak AD, *n*-hexane/*i*-PrOH = 80/20, 1.0 mL/min, λ = 254 nm, t (major) = 8.190 min, t (minor) = 7.248 min]; ^{1}H NMR (400 MHz, $CDCl_3$): major rotational isomer: δ = 7.80-7.06 (m, 5.52H), 6.45 (s, 0.69H), 6.21 (s, 0.69H), 5.84 (q, *J* = 7.2 Hz, 0.69H), 5.16 (s, 0.69H), 3.72 (s, 2.07H), 2.13 (s, 2.07H), 1.55 (d, *J* = 6.8 Hz, 2.07H) ppm; minor rotational isomer: δ = 7.80-7.06 (m, 2.48H), 6.77 (s, 0.31H), 6.31 (s, 0.31H), 5.90 (q, *J* = 7.2 Hz, 0.31H), 5.64 (s, 0.31H), 3.59 (s, 0.93H), 2.15 (s, 0.93H), 1.70 (d, *J* = 7.2 Hz, 0.93H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): major rotational isomer: δ = 171.4, 165.8, 148.8, 137.8, 137.2, 134.1, 133.7, 132.3, 131.0, 129.1, 128.5, 128.3, 128.2, 127.8, 127.5, 124.4, 56.6, 52.0, 22.0, 14.3 ppm; minor rotational isomer: δ = 171.5, 165.8, 148.8, 139.8, 137.4, 136.8, 133.8, 132.4, 131.6, 130.7, 128.9, 128.4, 128.3, 128.0, 127.8, 124.5, 56.3, 51.9, 22.2, 14.6 ppm; ESI-HRMS: calcd. for $C_{22}H_{21}ClN_2O_5$+Na 451.1037, found 451.1033.



3n, 99% yield; $[\alpha]_D^{20}$ = -56.57 (*c* = 1.75 in $CHCl_3$); 91% ee, determined by HPLC analysis [Daicel Chiralcel OD, *n*-hexane/*i*-PrOH = 80/20, 1.0 mL/min, λ = 254 nm, t (major) = 6.452 min, t (minor) = 13.795 min]; ^{1}H NMR (400 MHz, $CDCl_3$): major rotational isomer: δ = 7.30-6.92 (m, 3.5H), 6.32 (s, 0.5H), 6.17 (s, 0.5H), 5.94 (q, *J* = 7.2 Hz, 0.5H), 5.73 (s, 0.5H), 3.67 (s, 1.5H), 2.13 (s, 1.5H), 1.74 (d, *J* = 7.2 Hz, 1.5H) ppm; minor rotational isomer: δ = 7.30-6.92 (m, 3.5H), 6.32 (s, 0.5H), 5.86 (q, *J* = 7.2 Hz, 0.5H), 5.84 (s, 0.5H), 5.64 (s, 0.5H), 3.61 (s, 1.5H), 2.12 (s, 1.5H), 1.60 (d, *J* = 6.8 Hz, 1.5H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): major rotational isomer: δ = 171.6, 166.1, 140.1, 139.1, 138.8, 137.6, 136.8, 136.4, 134.3, 131.7, 129.6, 129.2, 128.7, 127.8, 127.2, 126.4, 61.2, 52.1, 22.4, 14.7 ppm; minor rotational isomer: δ = 171.1, 165.9, 140.0, 139.8, 138.5, 138.2, 137.3, 136.9, 136.7, 134.2, 131.5, 129.6, 128.9, 128.6, 127.5, 127.1, 60.5, 52.0, 22.2, 14.6 ppm; ESI-HRMS: calcd. for $C_{22}H_{20}Cl_3NO_3$+Na 474.0406, found 474.0412.

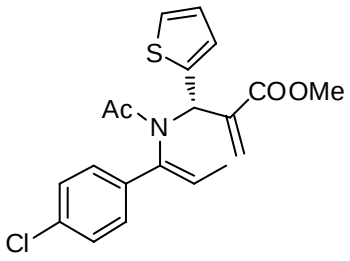


3o, 88% yield; $[\alpha]_D^{20}$ = -44.71 (*c* = 1.72 in CH_2Cl_2); 87% ee, determined by HPLC analysis [Daicel Chiralcel OD, *n*-hexane/*i*-PrOH = 80/20, 1.0 mL/min, λ = 254 nm, t (major) = 5.916 min, t (minor) = 12.014 min]; ^{1}H NMR (400 MHz, $CDCl_3$): major rotational isomer: δ = 7.26-7.19 (m, 1.12H), 7.08-6.76 (m, 3.36H), 6.30 (s, 0.56H), 6.27 (s, 0.56H), 6.25 (s, 0.56H), 5.87 (q, *J* = 7.2 Hz, 0.56H), 3.60 (s, 1.68H), 2.18 (s, 1.68H), 2.14 (s, 1.68H), 1.53 (d, *J* = 7.2 Hz, 1.68H) ppm; minor rotational isomer: δ = 7.26-7.19 (m, 0.88H), 7.08-6.76 (m, 2.64H), 5.79 (s, 0.44H), 5.75 (q, *J* = 6.8 Hz, 0.44H), 5.70 (s, 0.44H), 5.65 (s, 0.44H), 3.67 (s, 1.32H), 2.15 (s, 1.32H), 2.11 (s, 1.32H), 1.73 (d, *J* = 7.2 Hz, 1.32H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): major rotational isomer: δ = 171.5, 166.6, 140.3, 139.7, 138.0, 137.1, 135.7, 133.6, 130.9, 128.8, 128.3, 127.6, 127.3, 126.9, 125.8, 62.1, 51.9, 22.5, 21.3, 14.7 ppm; minor rotational isomer: δ = 171.1, 166.4, 140.1, 139.2, 137.2, 136.7, 133.8, 131.0, 129.0, 128.4, 127.9, 127.5, 127.2, 126.7, 124.8, 61.4, 51.9, 22.4, 21.2, 14.5 ppm; ESI-HRMS: calcd. for $C_{23}H_{24}ClNO_3$+Na 420.1342, found 420.1349.

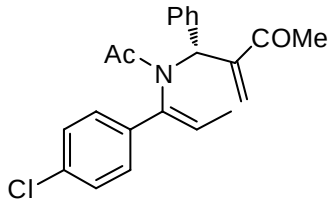


3p, 99% yield; $[\alpha]_D^{20}$ = -66.25 (*c* = 2.85 in $CHCl_3$); 90% ee, determined by HPLC analysis [Daicel Chiralcel OD, *n*-hexane/*i*-PrOH = 80/20, 1.0 mL/min, λ = 254 nm, t (major) = 8.842 min, t (minor) = 19.214 min]; ^{1}H NMR (400 MHz, $CDCl_3$): major rotational isomer: δ = 7.28-7.22 (m, 1.0H), 7.11-7.08 (m, 1.0H), 6.62-6.46 (m, 1.5H), 6.26 (s, 0.5H), 6.25 (s, 0.5H), 6.24 (s, 0.5H), 5.93-5.82 (m, 1.5H), 3.68 (s, 1.5H), 2.14 (s, 1.5H), 1.74 (d, *J* = 7.2 Hz, 1.5H) ppm; minor rotational isomer: δ = 7.28-7.22 (m, 1.0H), 7.11-7.08 (m, 1.0H), 6.62-6.46 (m, 1.5H), 5.93-5.82 (m, 1.5H), 5.79 (s, 0.5H), 5.67 (s, 0.5H), 5.63 (s, 0.5H), 3.62 (s, 1.5H), 2.11 (s, 1.5H), 1.57 (d, *J* = 7.2 Hz, 1.5H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): major rotational isomer: δ = 171.5, 166.5, 147.0, 146.9, 140.2, 139.7, 137.8, 133.8, 130.6, 129.0, 128.4, 127.1, 125.7, 123.9, 110.5, 107.5, 101.1, 61.9, 51.9, 22.5, 14.7 ppm; minor rotational isomer: δ = 171.1, 166.3, 147.2, 147.0, 140.2, 139.3, 137.1, 133.7, 129.6, 128.4, 127.8, 127.0, 124.4, 123.8, 110.2, 107.3, 101.0, 61.0, 51.9, 22.3, 14.5 ppm; ESI-HRMS: calcd. for $C_{23}H_{22}ClNO_5$+Na 450.1084, found 450.1089.

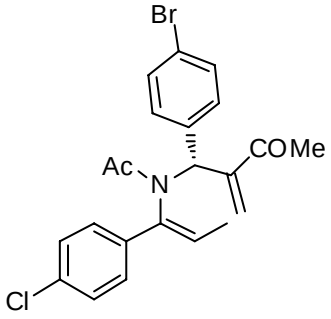
3q, 75% yield; $[\alpha]_D^{20}$ = -60.16 (*c* = 1.27 in $CHCl_3$); 71% ee, determined by HPLC analysis [Daicel Chiralcel OD, *n*-hexane/*i*-PrOH = 80/20, 1.0 mL/min, λ = 254 nm, t (major) = 7.290 min, t (minor) = 9.252 min]; ^{1}H NMR (400 MHz, $CDCl_3$): major rotational isome: δ = 7.27-7.11 (m, 2.9H), 6.88-6.81 (m,



1.16H), 6.63 (s, 0.58H), 6.27 (s, 0.58H), 6.01 (s, 0.58H), 5.96 (q, *J* = 6.8Hz, 0.58H), 3.68 (s, 1.74H), 2.11 (s, 1.74H), 1.52 (d, *J* = 6.8 Hz, 1.74H) ppm; minor rotational isomer: δ = 7.27-7.11 (m, 2.1H), 6.74-6.68 (m, 0.84H), 6.38 (s, 0.42H), 6.37 (s, 0.42H), 6.03 (q, *J* = 7.2 Hz, 0.42H), 5.92 (s, 0.42H), 3.70 (s, 1.26H), 2.09 (s, 1.26H), 1.72 (d, *J* = 6.8 Hz, 1.26H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): major rotational isomer: δ = 171.7, 170.8, 166.4, 150.6, 139.5, 138.8, 136.9, 133.9, 128.9, 128.6, 128.4, 127.6, 127.0, 125.9, 55.9, 52.2, 22.4, 14.5 ppm; minor rotational isomer: δ = 171.3, 170.5, 166.4, 150.1, 140.3, 139.2, 137.4, 133.9, 128.7, 128.5, 128.3, 127.2, 126.2, 125.9, 54.9, 52.1, 22.2, 14.5 ppm; ESI-HRMS: calcd. for $C_{20}H_{20}ClNO_3S$+K 428.0489, found 428.0464.

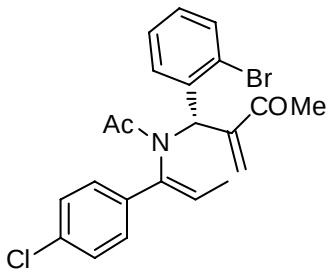


3r, 94% yield; $[\alpha]_D^{20}$ = -31.79 (*c* = 1.40 in CH_2Cl_2); 80% ee, determined by HPLC analysis [Daicel Chiralpak AD, *n*-hexane/*i*-PrOH = 80/20, 1.0 mL/min, λ = 254 nm, t (major) = 8.718 min, t (minor) = 6.386 min]; ^{1}H NMR (400 MHz, $CDCl_3$): major rotational isomer: δ = 7.28-7.04 (m, 4.86H), 5.96 (s, 0.54H), 5.82-5.76 (m, 0.54H), 5.56 (s, 0.54H), 5.40 (s, 0.54H), 2.36 (s, 1.62H), 2.07 (s, 1.62H), 1.56-1.53 (m, 1.62H) ppm; minor rotational isomer: δ = 7.28-7.04 (m, 4.14H), 6.31 (s, 0.46H), 6.10 (s, 0.46H), 5.99 (s, 0.46H), 5.82-5.76 (m, 0.46H), 2.15 (s, 1.38H), 2.11 (s, 1.38H), 1.56-1.53 (m, 1.38H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): major rotational isomer: δ = 198.8, 171.4, 149.0, 140.3, 137.8, 136.5, 134.0, 130.0, 128.8, 128.4, 127.8, 127.7, 126.8, 123.0, 62.0, 26.5, 22.5, 14.7 ppm; minor rotational isomer: δ = 198.2, 171.1, 147.5, 139.9, 137.5, 136.4, 133.8, 129.9, 128.5, 127.9, 127.8, 127.7, 126.0, 123.0, 60.3, 26.1, 22.4, 14.3 ppm; ESI-HRMS: calcd. for $C_{22}H_{23}ClNO_2$+H 368.1417, found 368.1424.

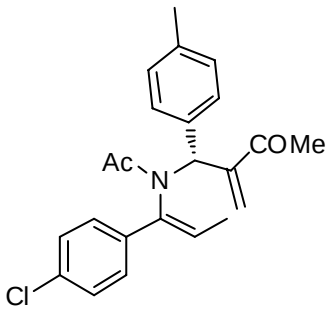


3s, 78% yield; $[\alpha]_D^{20}$ = -28.52 (*c* = 1.35 in $CHCl_3$); 86% ee, determined by HPLC analysis [Daicel Chiralpak AD, *n*-hexane/*i*-PrOH = 80/20, 1.0 mL/min, λ = 254 nm, t (major) = 9.257 min, t (minor) = 7.174 min]; ^{1}H NMR (400 MHz, $CDCl_3$): major rotational isomer: δ = 7.29-7.26 (m, 2.12H), 7.21-7.18 (m, 0.53H), 7.08-7.06 (m, 0.53H), 7.00-6.94 (m, 1.06H), 6.19 (s, 0.53H), 5.99 (s, 0.53H), 5.85-5.81 (m, 0.53H), 5.59 (s, 0.53H), 2.34 (s, 1.59H), 2.07 (s, 1.59H), 1.60-1.57 (m, 1.59H) ppm; minor rotational isomer: δ = 7.29-7.26 (m, 1.88H), 7.21-7.18 (m, 0.47H), 7.08-7.06 (m, 0.47H), 7.00-6.94 (m, 0.94H), 6.13 (s, 0.47H), 6.03 (s, 0.47H), 5.85-5.81 (m, 0.47H), 5.41 (s, 0.47H), 2.16 (s, 1.41H), 2.11 (s, 1.41H), 1.60-1.57 (m, 1.41H) ppm; ^{13}C NMR (100 MHz,

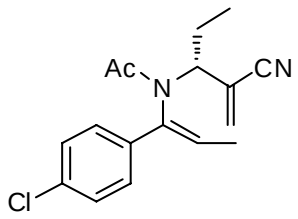
$CDCl_3$): major rotational isomer: δ = 198.5, 171.6, 148.4, 140.2, 137.4, 136.3, 134.2, 131.5, 131.0, 128.7, 127.9, 126.9, 123.5, 112.1, 61.3, 26.4, 22.5, 14.7 ppm; minor rotational isomer: δ = 198.5, 171.2, 147.1, 139.9, 136.7, 135.8, 134.1, 131.5, 130.8, 128.6, 127.5, 126.5, 121.7, 112.1, 59.9, 26.0, 22.4, 14.4 ppm; ESI-HRMS: calcd. for $C_{22}H_{21}BrClNO_2$+Na 468.0342, found 468.0349.



3t, 90% yield; $[\alpha]_D^{20}$ = +129.7 (*c* = 2.05 in $CHCl_3$); 87% ee, determined by HPLC analysis [Daicel Chiralpak AD, *n*-hexane/*i*-PrOH = 80/20, 1.0 mL/min, λ = 254 nm, t (major) = 7.990 min, t (minor) = 6.059 min]; ^{1}H NMR (400 MHz, $CDCl_3$): major rotational isomer: δ = 7.70-7.68 (m, 0.64H), 7.45-6.99 (m, 4.48H), 5.87-5.85 (m, 1.28H), 5.76 (q, *J* = 6.8 Hz, 0.64H), 5.12 (s, 0.64H), 2.38 (s, 1.92H), 2.08 (s, 1.92H), 1.43 (d, *J* = 7.2 Hz, 1.92H) ppm; minor rotational isomer: δ = 7.45-6.99 (m, 2.88H), 6.46 (s, 0.36H), 6.06 (s, 0.36H), 5.87-5.81 (m, 0.36H), 5.67 (s, 0.36H), 2.22 (s, 1.08H), 2.14 (s, 1.08H), 1.79 (d, *J* = 7.2 Hz, 1.08H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): major rotational isomer: δ = 198.5, 171.6, 148.1, 140.2, 138.3, 136.1, 134.1, 132.6, 131.3, 129.0, 128.3, 128.0, 127.3, 126.1, 124.8, 124.1, 61.0, 26.6, 22.3, 14.3 ppm; minor rotational isomer: δ = 198.5, 171.6, 147.2, 140.2, 137.6, 136.1, 134.1, 132.8, 131.3, 129.2, 128.5, 128.2, 127.8, 126.6, 124.8, 124.1, 61.1, 26.3, 22.6, 15.4 ppm; ESI-HRMS: calcd. for $C_{22}H_{22}BrClNO_2$+H 446.0522, found 446.0524.



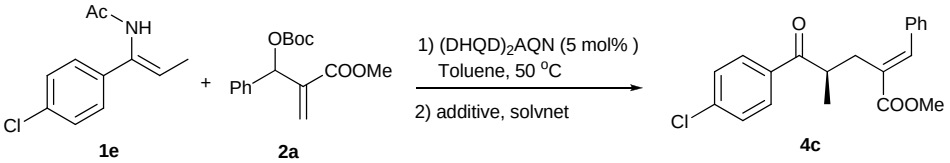
3u, 72% yield; $[\alpha]_D^{20}$ = -15.9 (*c* = 1.35 in $CHCl_3$); 86% ee, determined by HPLC analysis [Daicel Chiralcel AD, *n*-hexane/*i*-PrOH = 80/20, 1.0 mL/min, λ = 254 nm, t (major) = 6.888 min, t (minor) = 5.823 min]; ^{1}H NMR (400 MHz, $CDCl_3$): major rotational isomer: δ = 7.28-7.20 (m, 2.08H), 7.07-6.94 (m, 2.08H), 6.07 (s, 0.52H), 5.94 (s, 0.52H), 5.80-5.78 (m, 0.52H), 5.56 (s, 0.52H), 2.35 (s, 1.56H), 2.27 (s, 1.56H), 2.06 (s, 1.56H), 1.56-1.53 (m, 1.56H) ppm; minor rotational isomer: δ = 7.28-7.20 (m, 1.92H), 7.07-6.94 (m, 1.92H), 6.26 (s, 0.48H), 5.99 (s, 0.48H), 5.80-5.78 (m, 0.48H), 5.37 (s, 0.48H), 2.28 (s, 1.44H), 2.14 (s, 1.44H), 2.11 (s, 1.44H), 1.56-1.53 (m, 1.44H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): major rotational isomer: δ = 198.8, 171.4, 149.2, 140.4, 137.8, 137.4, 134.5, 133.4, 129.8, 128.5, 128.4, 128.0, 126.7, 122.9, 61.8, 26.5, 22.6, 21.1, 14.7 ppm; minor rotational isomer: δ = 198.2, 171.1, 147.7, 140.0, 137.5, 136.5, 134.0, 130.0, 128.7, 128.4, 128.4, 127.7, 125.7, 122.9, 60.2, 26.1, 22.4, 21.1, 14.3 ppm; ESI-HRMS: calcd. for $C_{23}H_{25}ClNO_2$+H 382.1574, found 382.1579.



3v, 45% yield; $[\alpha]_D^{20}$ = -37.9 (*c* = 0.80 in $CHCl_3$); 39% ee, determined by HPLC analysis [Daicel Chiralpak AD, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t (major) = 6.606 min, t (minor) = 7.474 min]; ^{1}H NMR (400 MHz, $CDCl_3$): major rotational isomer: δ = 7.34-7.23 (m, 2.6H), 6.24 (q, *J* = 7.2Hz, 0.65H), 6.13 (s, 0.65H), 6.09 (s, 0.65H), 5.21 (dd, *J* = 10.8 Hz, 5.6 Hz, 0.65H), 2.06 (s, 1.95H), 1.78 (d, *J* = 7.2 Hz, 1.95H), 1.42-1.21 (m, 1.3H), 0.73 (t, *J* = 7.2 Hz, 1.95H) ppm; minor rotational isomer: δ = 7.34-7.23 (m, 1.4H), 6.29 (q, *J* = 7.2Hz, 0.35H), 5.95 (s, 0.35H), 5.83 (s, 0.35H), 4.93 (dd, *J* = 10.8 Hz, 5.6 Hz, 0.35H), 2.05 (s, 1.05H), 1.84 (d, *J* = 6.8 Hz, 1.05H), 1.42-1.21 (m, 0.7H), 0.85 (t, *J* = 7.2 Hz, 1.05H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): major rotational isomer: δ = 170.8, 137.5, 137.1, 136.5, 134.3, 129.8, 128.8, 127.2, 122.1, 117.1, 58.6, 24.7, 21.5, 15.1, 10.9 ppm; minor rotational isomer: δ = 175.1, 137.4, 136.7, 136.3, 134.2, 128.9, 128.8, 127.0, 122.0, 117.1, 59.1, 23.9, 21.7, 14.8, 10.7 ppm; ESI-HRMS: calcd. for $C_{17}H_{19}ClN_2O$+K 341.0823, found 341.0826.

3. General procedure for the one-pot *N*-alkylation–Aza-Cope rearrangement.

Optimisation of the one-pot *N*-alkylation–aza-Cope rearrangement[a]

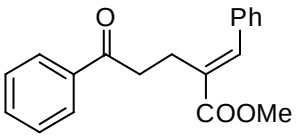


Entry	additive	Solvent	T (°C)	*t* (h)	Yield[b] (%)	ee[c] (%)
1[d]	/	Toluene/H_2O	110	49	ND	/
2	/	DMF/H_2O	110	49	59	77
3	$LiClO_4$	MeOH/H_2O	80	48	< 10	
4[d]	$TiCl_4$	Toluene	50	24	ND	/
5[d]	TMSOTf	Toluene	100	24	ND	/
6	PTSA	DMF/H_2O	110	21	64	82

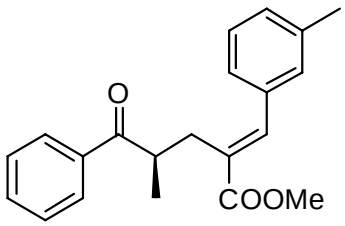
[a] Unless otherwise noted, reactions were performed with 0.2 mmol of **1e**, 0.4 mmol of **2a**, 0.01 mmol of catalyst in 1 mL toluene at 50 °C. After the consumption of **1e**, the solvent was removed under reduced pressure, and new solvent was added to the residue, followed by the additive. [b] Isolated yield of two steps. [c] Determined by chiral HPLC analysis. [d] Not determined.

To a solution of **1e** (42.0 mg, 0.2 mmol) and $(DHQD)_2AQN$ (8.6 mg, 5 mol%) in dry toluene (1.0 mL) was added **2a** (116.8 mg, 0.4 mmol). The reaction mixture was stirred at 50 ºC until consumption of **1e**, which was monitored by TLC analysis. Then the solvent was removed under reduced pressure, DMF (400 μL) and H_2O (100 μL) were added to the residue, followed by PTSA (10.3 mg, 0.06 mmol). The mixture was stirred at 110 °C until consumption of **3c**. The reaction mixture was cooled to room temperature,

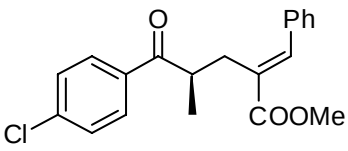
extracted with ethyl acetate, washed with H_2O and brine, dried over anhydrous Na_2SO_4 and concentrated under vacuum after filtration. Purification by flash column chromatography on silica gel (EtOAc/PE = 1/100) afforded the product **4c** as a yellowish oil (44.0 mg, 64%).



4a, 84% yield; *E*/*Z* > 95/5 (determined by crude ^{1}H NMR); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.99-7.96 (m, 2H), 7.77 (s, 1H), 7.58-7.31 (m, 8H), 3.84 (s, 1H), 3.25-3.21 (m, 2H), 3.00-2.96 (m, 2H) ppm; ^{13}C NMR (50 MHz, $CDCl_3$): δ = 199.1, 168.5, 140.3, 136.7, 135.3, 133.0, 131.7, 129.2, 128.7, 128.6, 128.1, 52.1, 38.0, 22.7 ppm; ESI-HRMS: calcd. for $C_{19}H_{18}O_3$+Na 317.1154, found 317.1154.

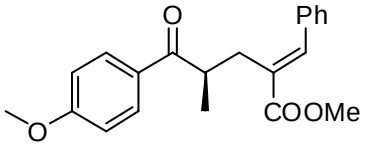


4b, 62% yield; *E*/*Z* > 95/5 (determined by crude ^{1}H NMR); $[\alpha]_D^{20}$ = +63.93 (*c* = 1.50 in $CHCl_3$); 80% ee, determined by HPLC analysis [Daicel Chiralpak AD, *n*-hexane/*i*-PrOH = 95/5, 1.0 mL/min, λ = 254 nm, t (major) = 7.500 min, t (minor) = 7.069 min]; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.98-7.96 (m, 2H), 7.77 (s, 1H), 7.56-7.13 (m, 7H), 3.87-3.81 (m, 4H), 2.95 (dd, *J* = 9.6 Hz, 4.8 Hz, 1H), 2.81 (dd, *J* = 13.6 Hz, 9.2 Hz, 1H), 2.35 (s, 3H), 1.08 (d, *J* = 6.8 Hz, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 203.7, 168.6, 141.7, 138.1, 136.2, 135.4, 132.9, 130.5, 129.7, 129.2, 128.5, 128.4, 128.4, 126.0, 52.0, 39.6, 30.8, 21.4, 16.2 ppm; ESI-HRMS: calcd. for $C_{21}H_{22}O_3$+Na 345.1467, found 345.1469.

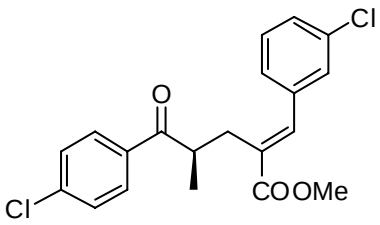


4c, 64% yield; *E*/*Z* = 89/11 (determined by crude ^{1}H NMR); $[\alpha]_D^{20}$ = +61.63 (*c* = 3.00 in $CHCl_3$); 82% ee, determined by HPLC analysis [Daicel Chiralpak AD, *n*-hexane/*i*-PrOH = 95/5, 1.0 mL/min, λ = 254 nm, t (major) = 11.424 min, t (minor) = 10.065 min]; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.95-7.90 (m, 2H), 7.80 (s, 1H), 7.45-7.31 (m, 7H), 3.82-3.76 (m, 4H), 2.92 (dd, *J* = 13.6 Hz, 5.2 Hz, 1H), 2.78 (dd, *J* = 14.0 Hz, 5.2 Hz, 1H), 1.06 (d, *J* = 6.8 Hz, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 202.4, 168.5, 141.7, 139.3, 136.7, 135.3, 134.5, 130.4, 129.9, 129.0, 128.6, 128.0, 52.1, 39.6, 30.8, 16.0 ppm; ESI-HRMS: calcd. for $C_{20}H_{19}ClO_3$+Na 365.0920, found 365.0924.

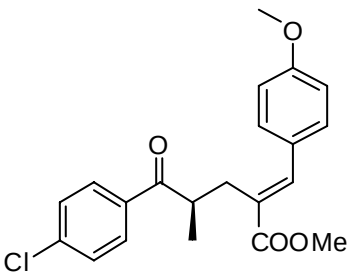
4d, 58% yield; *E*/*Z* > 95/5 (determined by crude ^{1}H NMR); $[\alpha]_D^{20}$ = +23.5 (*c* = 2.75 in CH_2Cl_2); 80% ee, determined by HPLC analysis [Daicel Chiralcel AD, *n*-hexane/*i*-PrOH = 95/5, 1.0 mL/min, λ = 254 nm, t



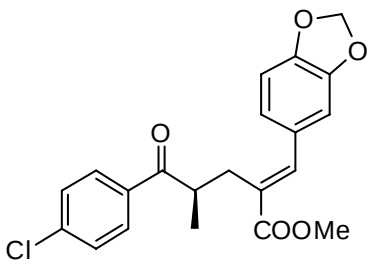
(major) = 20.158 min, t (minor) = 16.242 min]; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.98-7.96 (m, 2H), 7.79 (s, 1H), 7.41-7.26 (m, 5H), 6.94-6.91 (m, 2H), 3.88-3.77 (m, 7H), 2.92 (dd, *J* = 13.6 Hz, 5.2 Hz, 1H), 2.80 (dd, *J* = 13.6 Hz, 5.2 Hz, 1H), 1.06 (d, *J* = 6.8 Hz, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 202.2, 168.7, 163.4, 141.5, 135.5, 130.8, 130.8, 129.2, 129.1, 128.6, 128.5, 113.7, 55.4, 52.1, 39.2, 31.0, 16.2 ppm; ESI-HRMS: calcd. for $C_{21}H_{22}O_4$+Na 361.1416, found 361.1417.



4e, 55% yield; *E*/*Z* > 95/5 (determined by crude ^{1}H NMR); $[\alpha]_D^{20}$ = +63.36 (*c* = 0.72 in $CHCl_3$); 59% ee, determined by HPLC analysis [Daicel Chiralpak AD, *n*-hexane/*i*-PrOH = 95/5, 1.0 mL/min, λ = 254 nm, t (major) = 9.193 min, t (minor) = 7.859 min]; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.92-7.90 (m, 2H), 7.71 (s, 1H), 7.44-7.42 (m, 2H), 7.33-7.22 (m, 4H), 3.83-3.76 (m, 4H), 2.91 (dd, *J* = 13.6 Hz, 5.2 Hz, 1H), 2.70 (dd, *J* = 14.0 Hz, 5.2 Hz, 1H), 1.06 (d, *J* = 6.8 Hz, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 202.1, 168.1, 140.0, 139.4, 137.1, 134.5, 134.4, 131.8, 129.9, 128.9, 128.9, 128.5, 126.9, 52.2, 39.5, 30.9, 16.2 ppm; ESI-HRMS: calcd. for $C_{20}H_{18}Cl_2O_3$+Na 399.0531, found 399.0529.

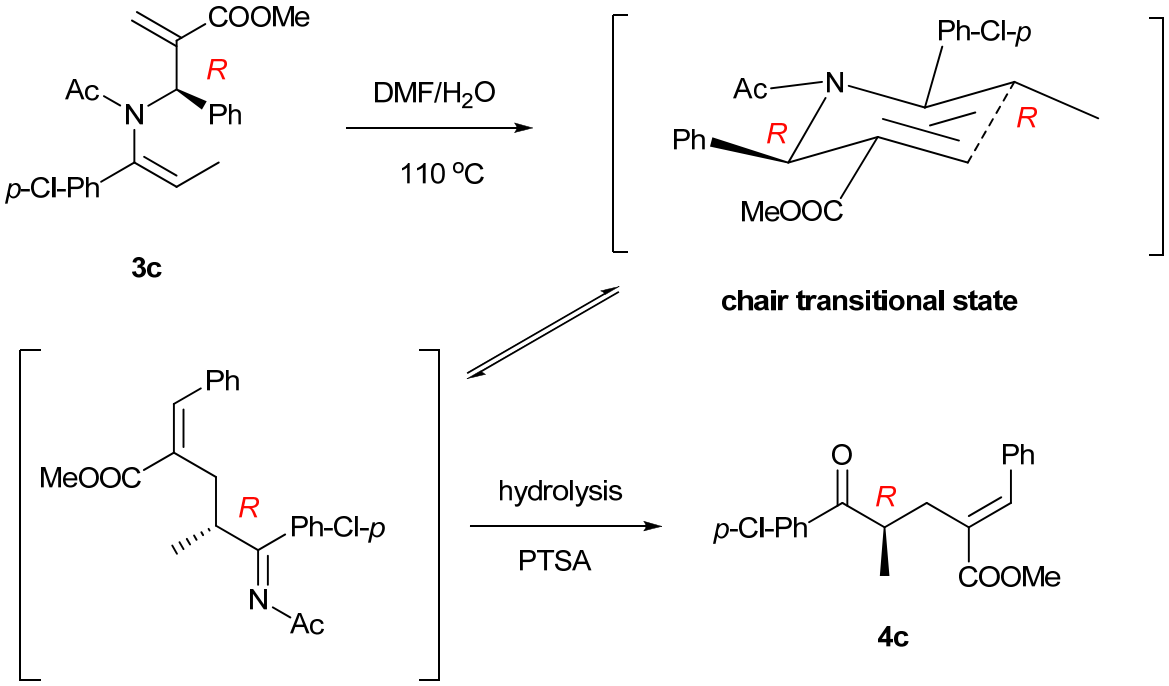


4f, 72% yield; *E*/*Z* > 95/5 (determined by crude ^{1}H NMR); $[\alpha]_D^{20}$ = +79.81 (*c* = 2.70 in $CHCl_3$); 88% ee, determined by HPLC analysis [Daicel Chiralpak AD, *n*-hexane/*i*-PrOH = 95/5, 1.0 mL/min, λ = 254 nm, t (major) = 15.641 min, t (minor) = 14.417 min]; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.94-7.92 (m, 2H), 7.73 (s, 1H), 7.43-7.36 (m, 4H), 6.92-6.90 (m, 2H), 3.89-3.81 (m, 7H), 2.94 (dd, *J* = 13.6 Hz, 5.2 Hz, 1H), 2.84 (dd, *J* = 13.6 Hz, 5.2 Hz, 1H), 1.09 (d, *J* = 6.8 Hz, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 202.6, 168.7, 159.9, 141.4, 139.3, 134.6, 130.9, 129.9, 128.8, 128.2, 127.6, 114.0, 55.3, 52.0, 39.6, 30.9, 16.1 ppm; ESI-HRMS: calcd. for $C_{21}H_{21}ClO_4$+Na 395.1026, found 395.1029.



4g, 66% yield; *E*/*Z* > 95/5 (determined by crude ^{1}H NMR); $[\alpha]_D^{20}$ = +89.12 (*c* = 2.50 in $CHCl_3$); 85% ee, determined by HPLC analysis [Daicel Chiralpak AD, *n*-hexane/*i*-PrOH = 95/5, 1.0 mL/min, λ = 254 nm, t (major) = 21.284 min, t (minor) = 16.159 min]; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.95-7.92 (m, 2H), 7.69 (s, 1H), 7.44-7.41 (m, 2H), 6.90-6.81 (m, 3H), 6.00 (s, 2H), 3.84-3.80 (m, 4H), 2.92 (dd, *J* = 14.0 Hz, 5.2 Hz, 1H), 2.79 (dd, *J* = 13.6 Hz, 5.2 Hz, 1H), 1.09 (d, *J* = 6.8 Hz, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 202.5, 168.6, 147.9, 147.8, 141.4, 139.3, 134.5, 129.9, 129.1, 128.9, 128.9, 124.0, 109.1, 108.5, 101.4, 52.0, 39.6, 30.9, 16.1 ppm; ESI-HRMS: calcd. for $C_{21}H_{19}ClO_5$+Na 409.0819, found 409.0822.

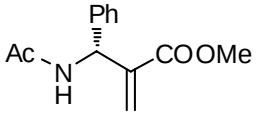
Speculation of the absolute configuration of aza-Cope rearrangement products.



Currently we are not successful to obtain the crystals suitable for the assignment of the absolute configuration of the chiral ketone products **4** by X-ray analysis. Nevertheless, based on the concerted mechanism of aza-Cope rearrangement, we proposed the possible configuration. The *N*-alkylation product **3c** prefers to undergo the [3,3]-sigmatropic rearrangement process through the chair transition state, in which the aryl group orients pseudoequatorially, as shown in the above scheme. Thus the newly generated chiral centre would be assigned to *R*.

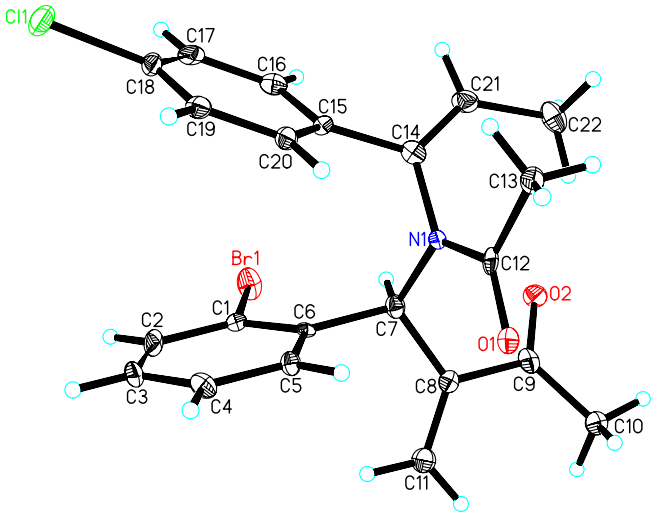
4. Transformation of *N*-allylic alkylation product

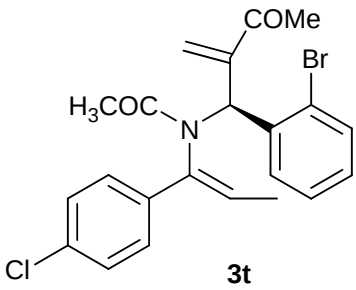
To a solution of **3a** (33.5 mg, 0.1 mmol) in dry DCM (0.5 mL) was added trifluoromethanesulfonic acid (17.6 μL, 0.2 mmol). The mixture was stirred at ambient temperature for 2 days. Then the solution was extracted with DCM, washed with aqueous $NaHCO_3$, concentrated and purified by column chromatography on silica gel (EtOAc/PE = 1/2). **5** was obtained as a colourless oil (12.4 mg, 53%).



5, 53% yield; $[\alpha]_D^{20}$ = -14.35 (*c* = 1.15 in CH_2Cl_2); 85% ee, determined by HPLC analysis [Daicel Chiralpak AD, *n*-hexane/*i*-PrOH = 80/20, 1.0 mL/min, λ = 254 nm, t (major) = 5.932 min, t (minor) = 5.289 min]; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.33-7.23 (m, 5H), 6.67-6.65 (m, 1H), 6.37 (s, 1H), 6.01 (d, *J* = 4.8 Hz, 1H), 5.93 (s, 1H), 3.70 (s, 3H), 2.05 (s, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 169.1, 166.2, 139.5, 139.0, 128.5, 127.5, 127.4, 126.4, 54.7, 51.9, 23.3 ppm; ESI-HRMS: calcd. for $C_{13}H_{15}NO_3$+Na 256.0950, found 256.0955.

5. Crystal data of enantiopure 3t



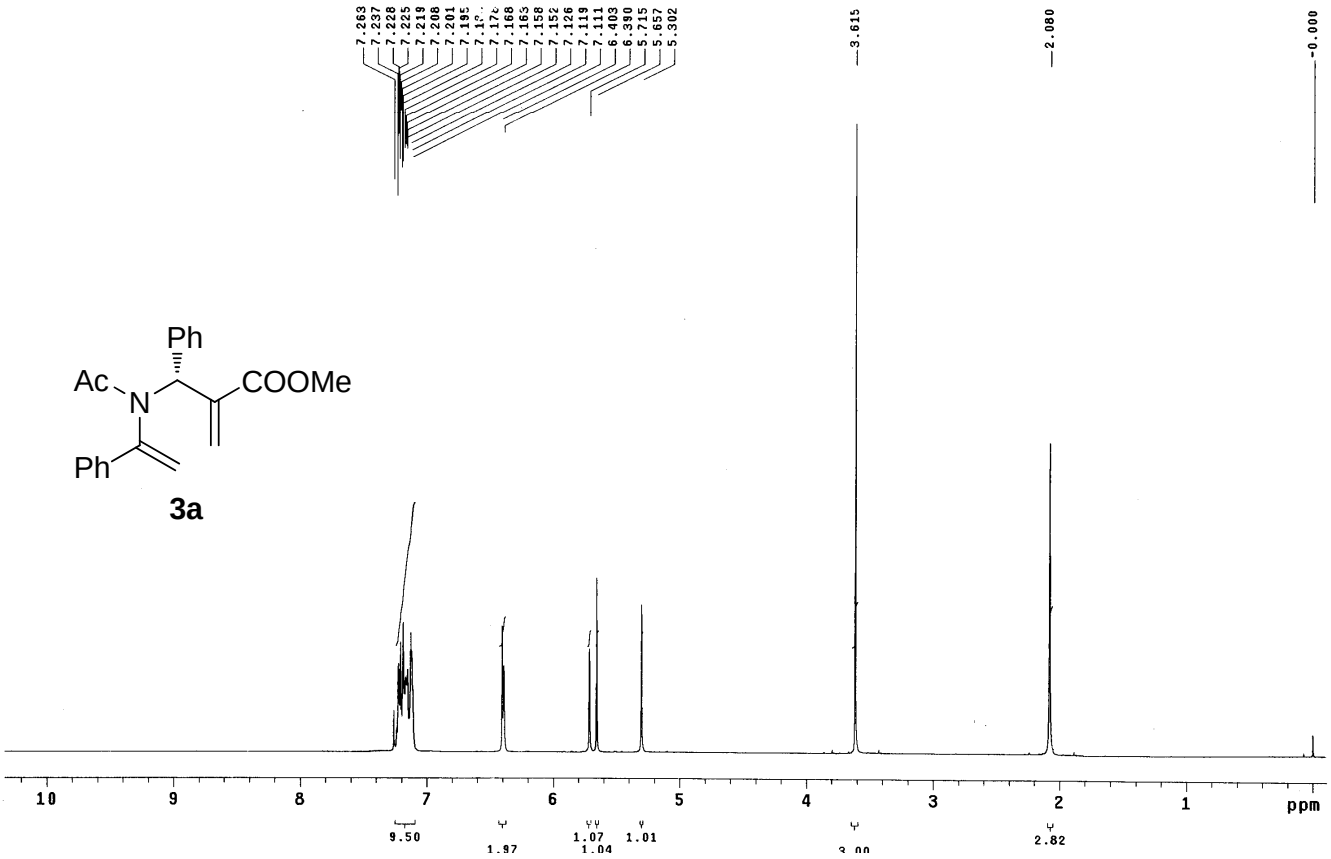


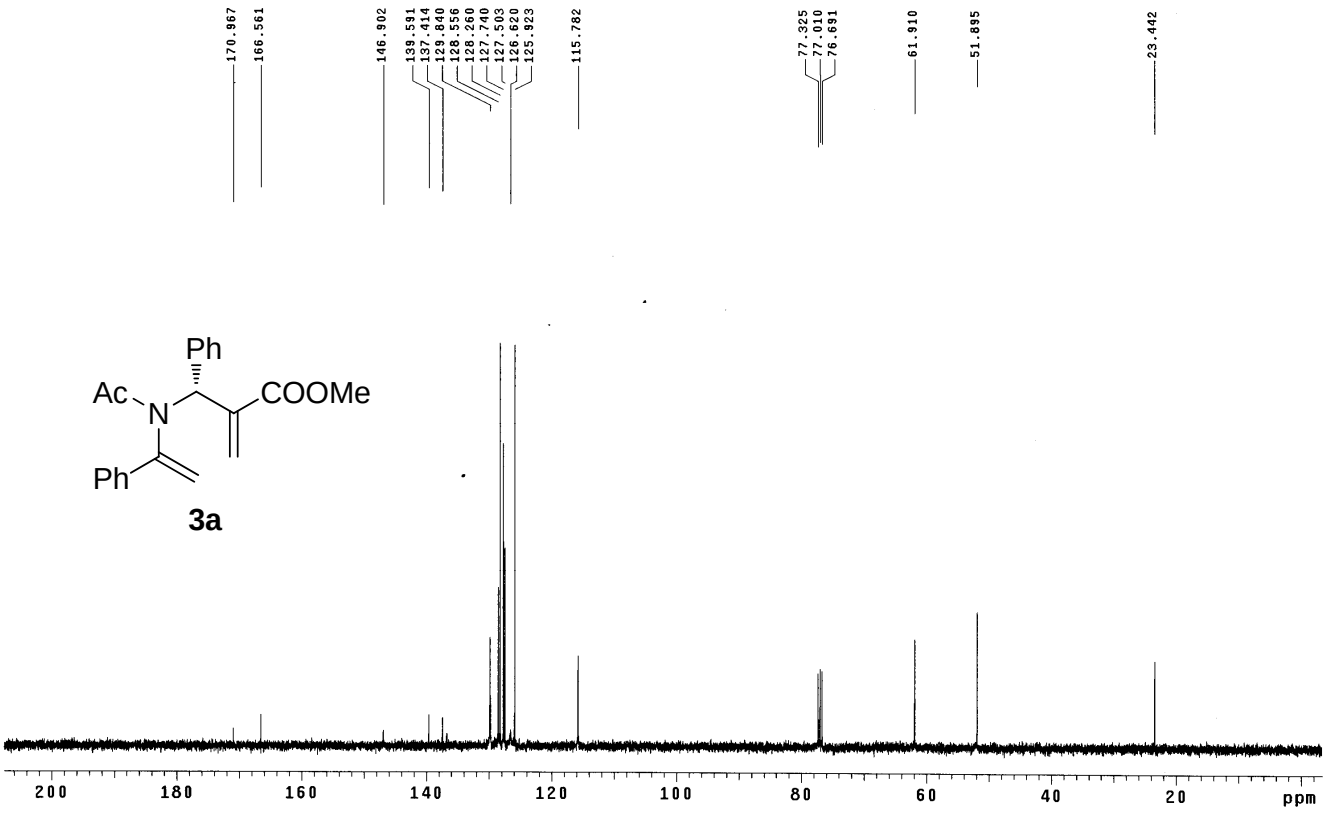
3t

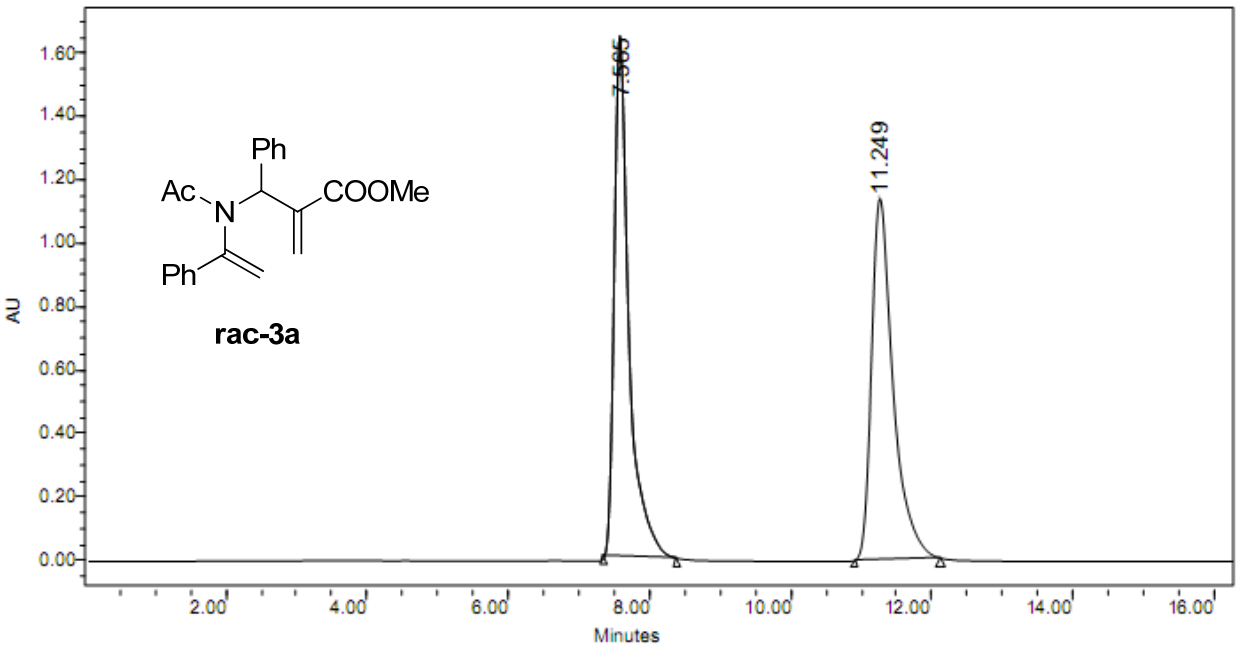
Identification code	**3t**
Empirical formula	C22 H21 Br Cl N O2
Formula weight	446.76
Temperature	103(2) K
Wavelength	0.71073 A

Crystal system, space group	Monoclinic, P2(1)
Unit cell dimensions	a = 7.0532(14) A alpha = 90 deg. b = 15.697(3) A beta = 99.348(3) deg. c = 8.9730(19) A gamma = 90 deg.
Volume	980.3(3) A^3
Z, Calculated density	2, 1.514 Mg/m^3
Absorption coefficient	2.250 mm^-1
F(000)	456
Crystal size	0.43 x 0.40 x 0.30 mm
Theta range for data collection	3.20 to 27.49 deg.
Limiting indices	-9<=h<=9, -20<=k<=20, -9<=l<=11
Reflections collected / unique	9373 / 4423 [R(int) = 0.0321]
Completeness to theta = 27.49	99.7 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.5517 and 0.4425
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4423 / 1 / 247
Goodness-of-fit on F^2	0.795
Final R indices [I>2sigma(I)]	R1 = 0.0284, wR2 = 0.0336
R indices (all data)	R1 = 0.0353, wR2 = 0.0344
Absolute structure parameter	0.029(5)
Largest diff. peak and hole	0.912 and -0.374 e.A^-3

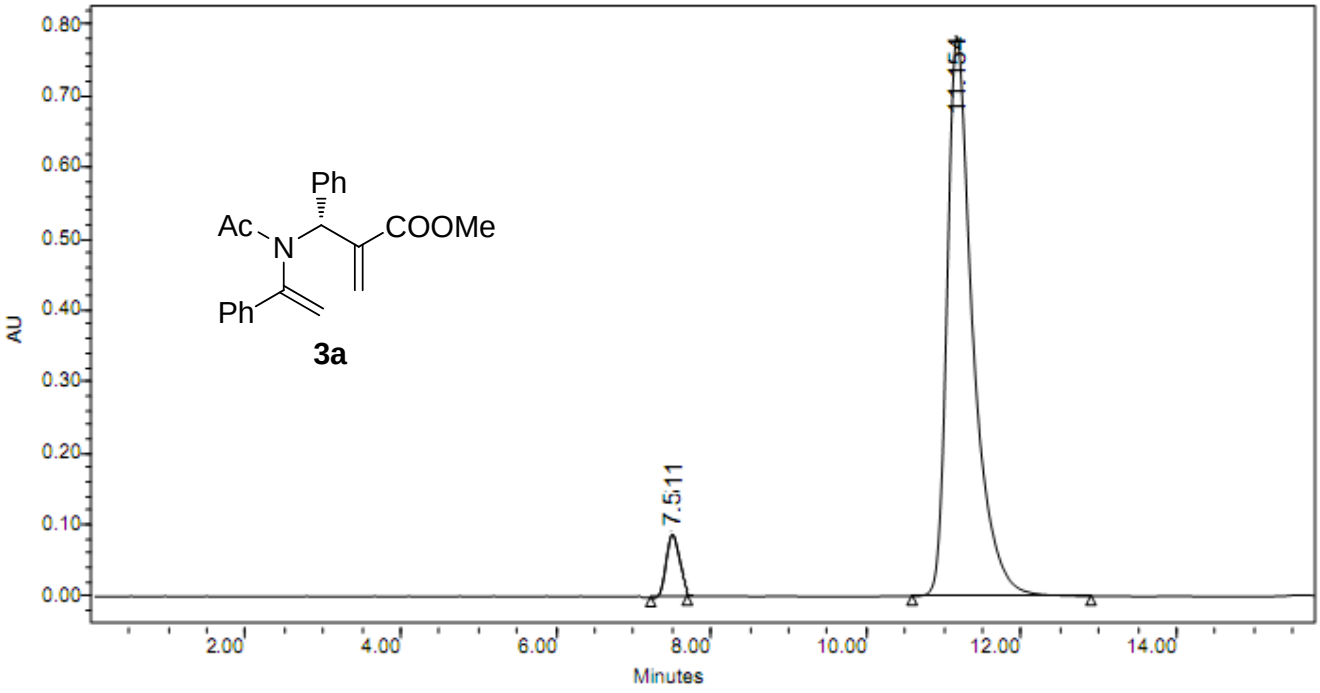
6. NMR spectra and HPLC chromatograms:



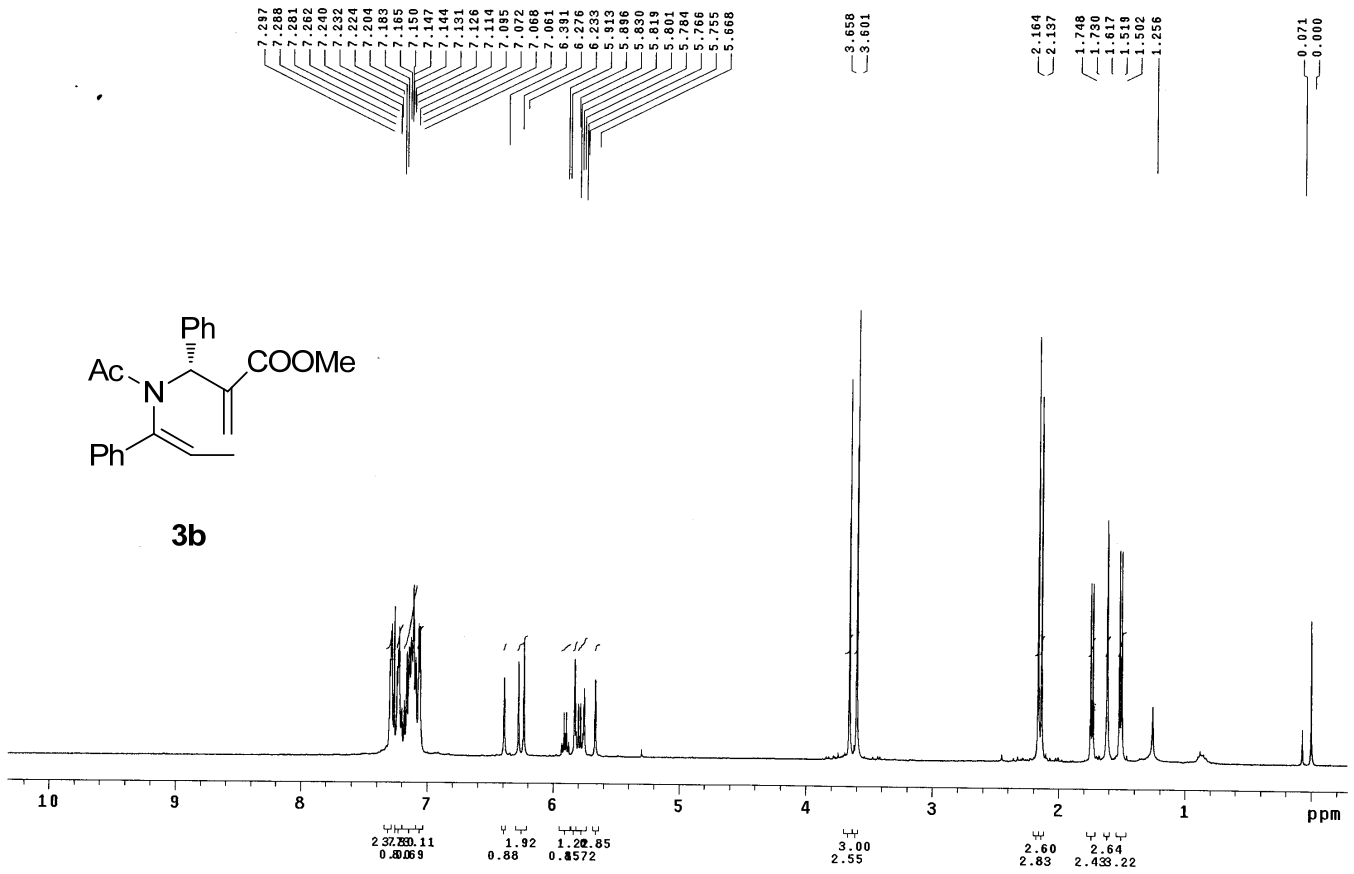




	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	7.565	23838826	49.60	1642577	59.04
2	11.249	24224415	50.40	1139406	40.96



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	7.511	1420390	7.31	89089	10.20
2	11.154	18009716	92.69	784656	89.80


7.297
7.288
7.281
7.262
7.240
7.232
7.224
7.204
7.183
7.165
7.150
7.147
7.144
7.131
7.126
7.114
7.095
7.072
7.066
7.061
6.391
6.276
6.233
5.913
5.896
5.830
5.819
5.801
5.784
5.766
5.755
5.668
3.658
3.601
2.164
2.137
1.748
1.730
1.617
1.519
1.502
1.256
0.071
0.000
Ph
Ac
N
COOMe
Ph
3b
10
9
8
7
6
5
4
3
2
1
ppm
1.92
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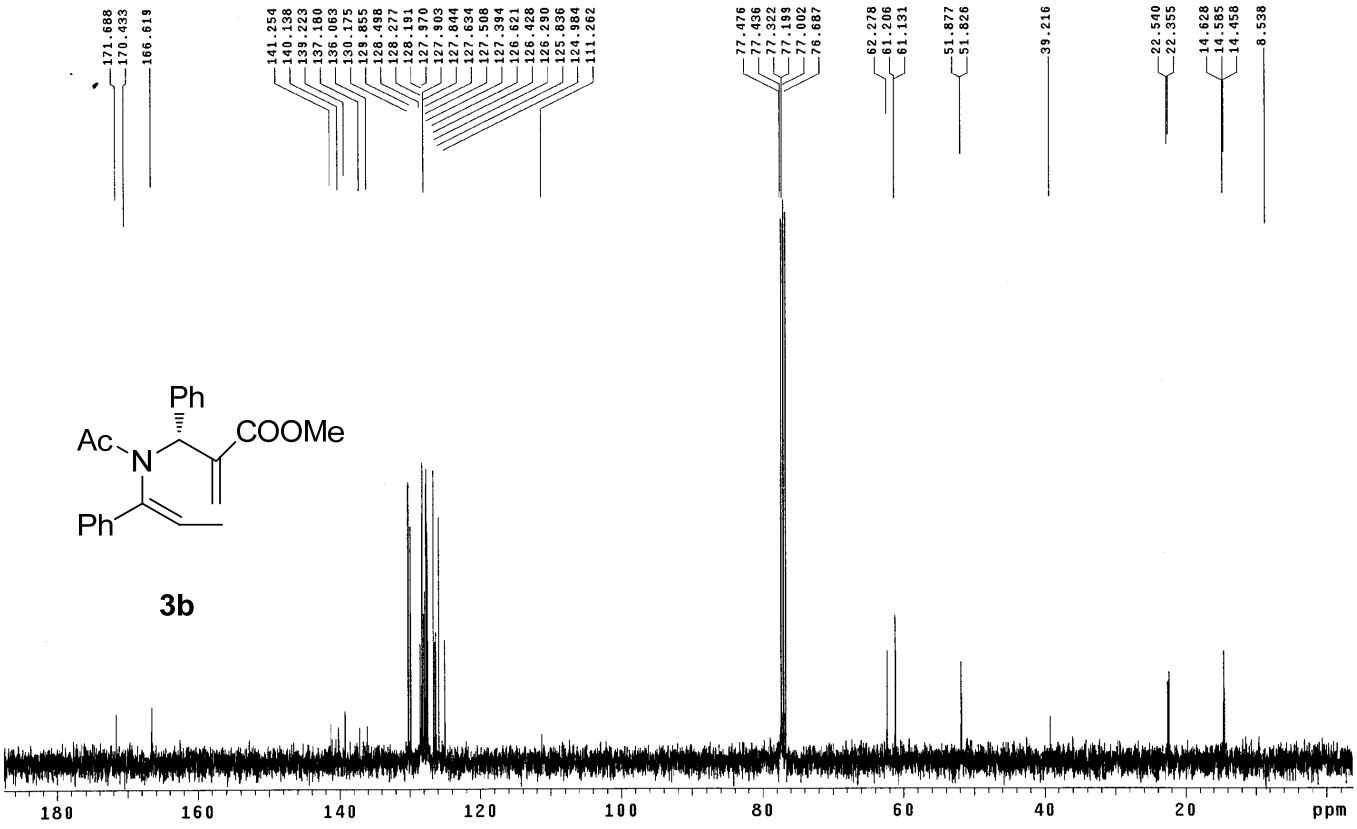
3.00
2.55
2.60
2.83
2.64


171.688
170.433
166.619
141.254
140.138
139.223
137.180
136.063
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129.855
128.498
128.277
128.191
127.970
127.903
127.844
127.634
127.508
127.394
126.621
126.428
126.290
125.836
124.984
111.262
77.476
77.436
77.322
77.199
77.002
76.687
62.278
61.206
61.131
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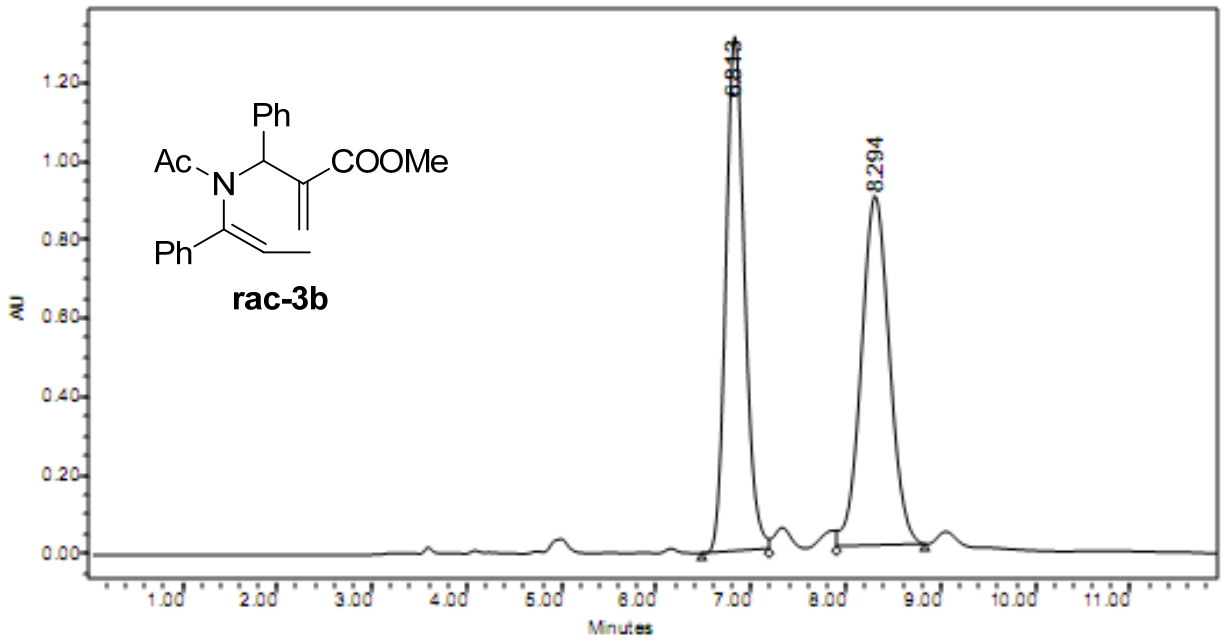
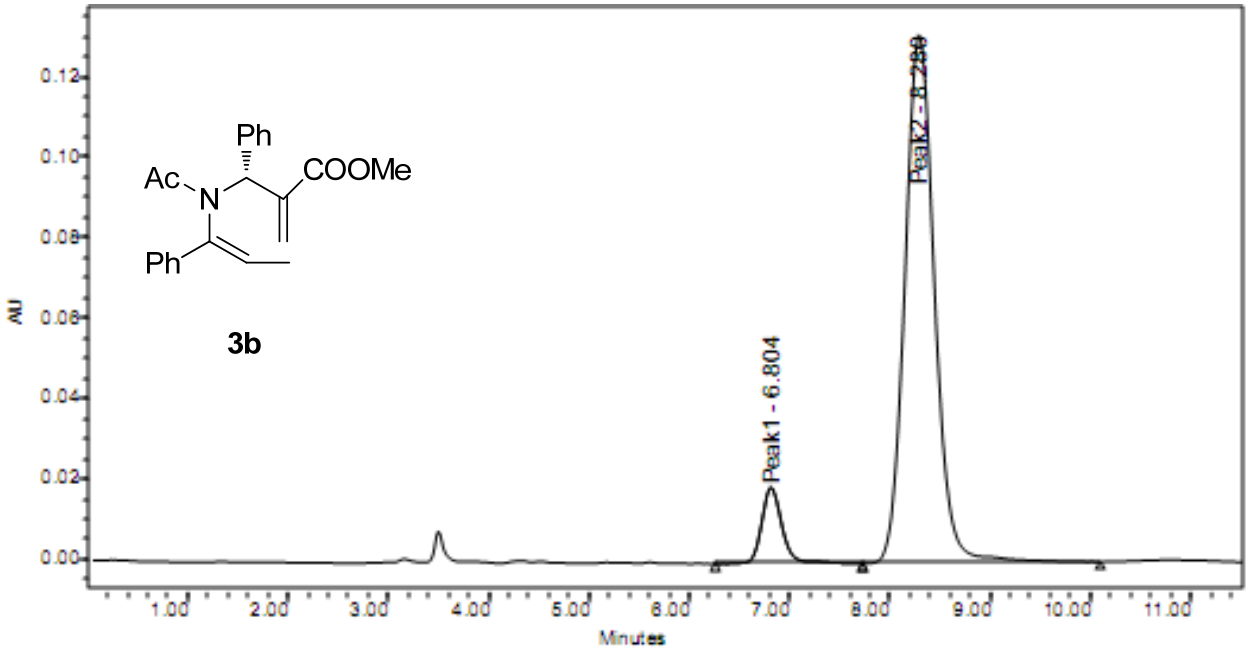
51.826
39.216
22.540
22.355
14.628
14.585
14.458
8.538
Ph
Ac
N
COOMe
Ph
3b
180
160
140
120
100
80
60
40
20
ppm



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	6.813	18138364	49.51	1313763	59.55
2	8.294	18497730	50.49	892512	40.45



	Peak Name	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
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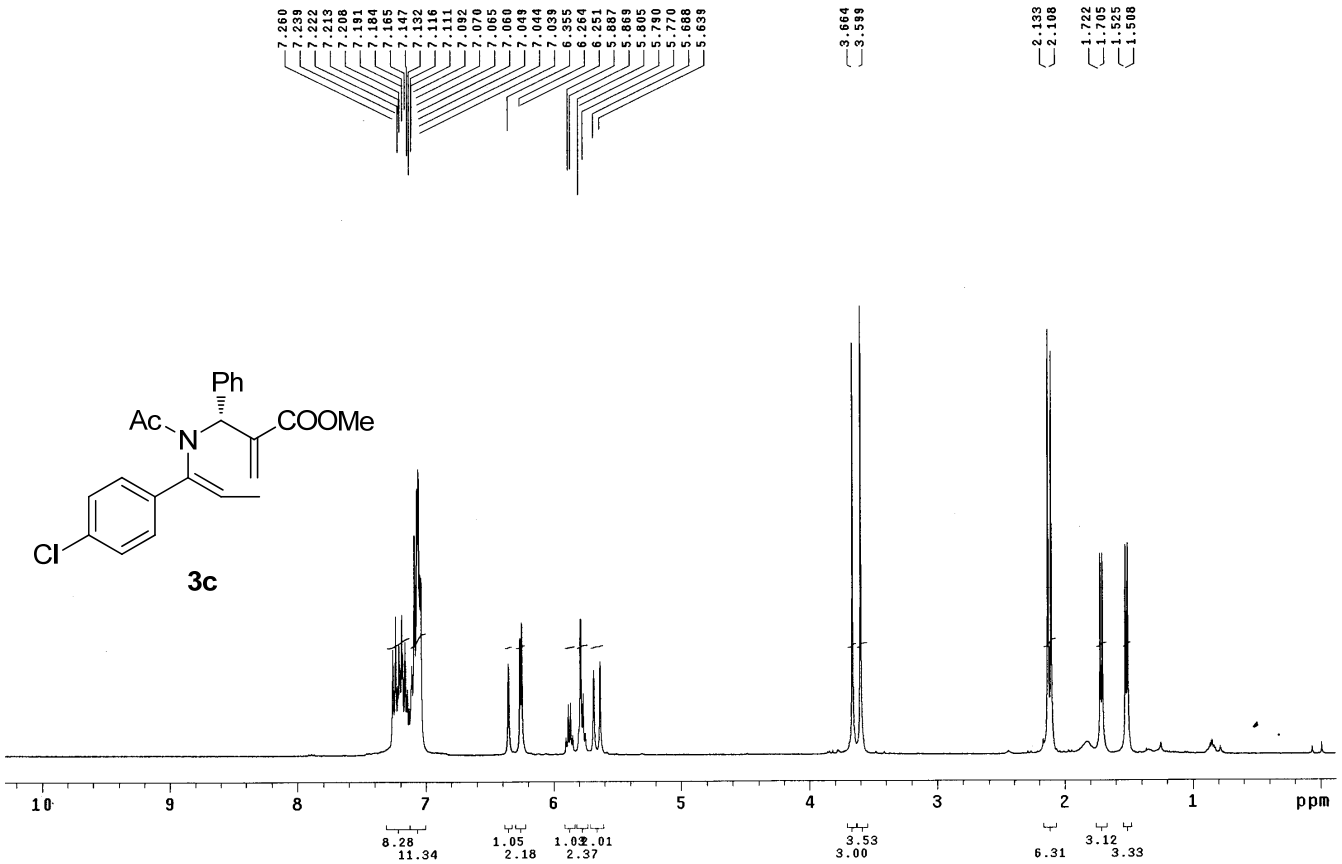
2	Peak2	8.280	2696962	91.00	131278	87.41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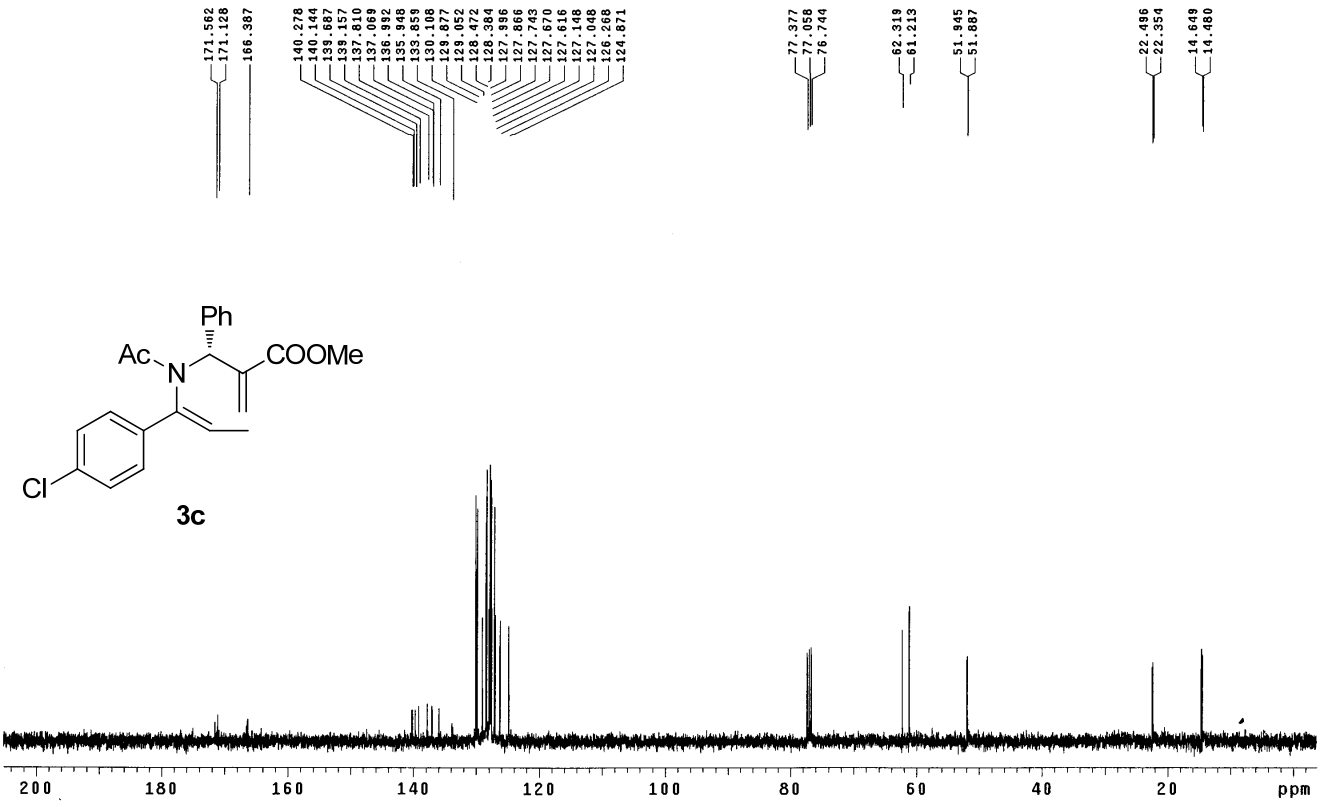
7.213
7.208
7.191
7.184
7.165
7.147
7.132
7.116
7.111
7.092
7.070
7.065
7.060
7.049
7.044
7.039
6.355
6.264
6.251
5.887
5.869
5.805
5.790
5.770
5.688
5.639
3.664
3.599
2.133
2.108
1.722
1.705
1.525
1.508
Ph
Ac
N
COOMe
Cl
3c
10
9
8
7
6
5
4
3
2
1
ppm
8.28
11.34
1.05
2.18
1.02
2.01
2.37
3.53
3.00
6.31
3.12
3.33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166.387
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140.144
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139.157
137.810
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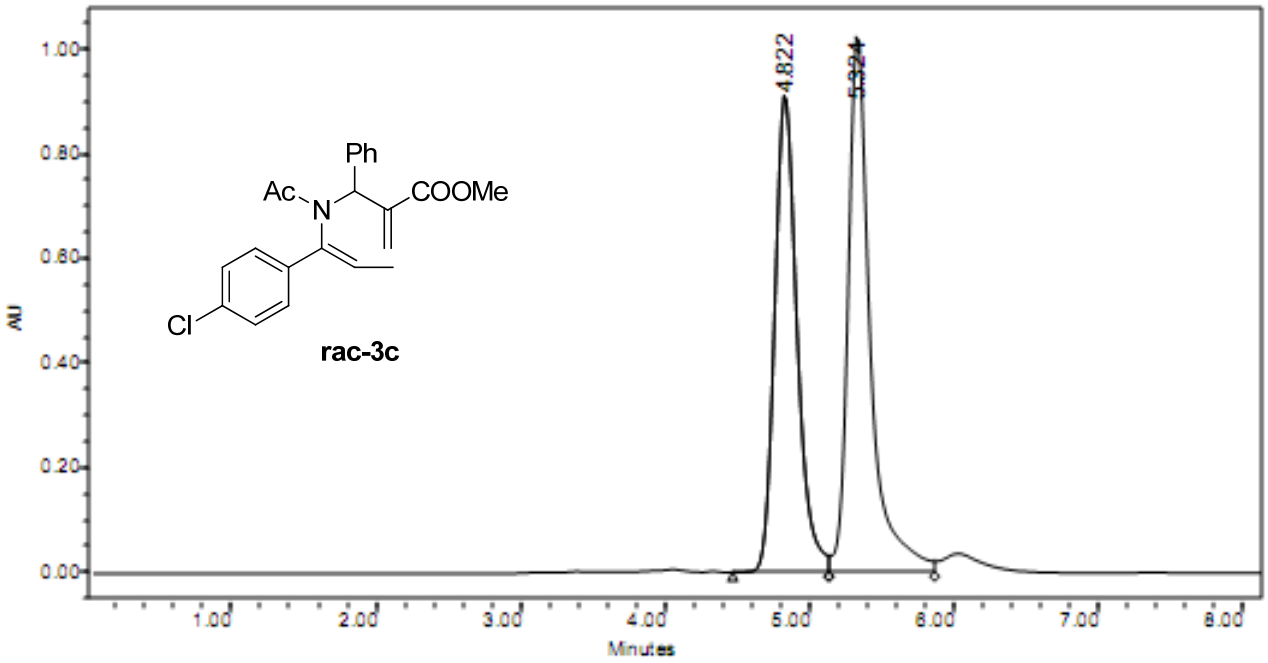
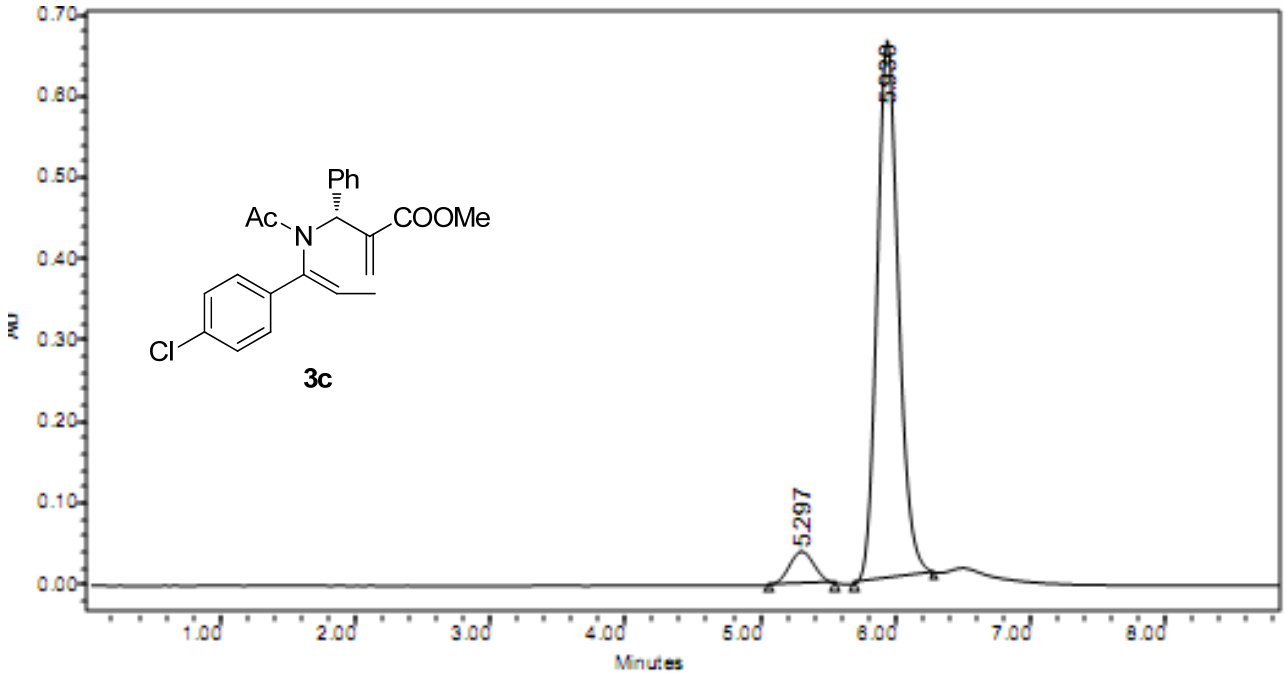
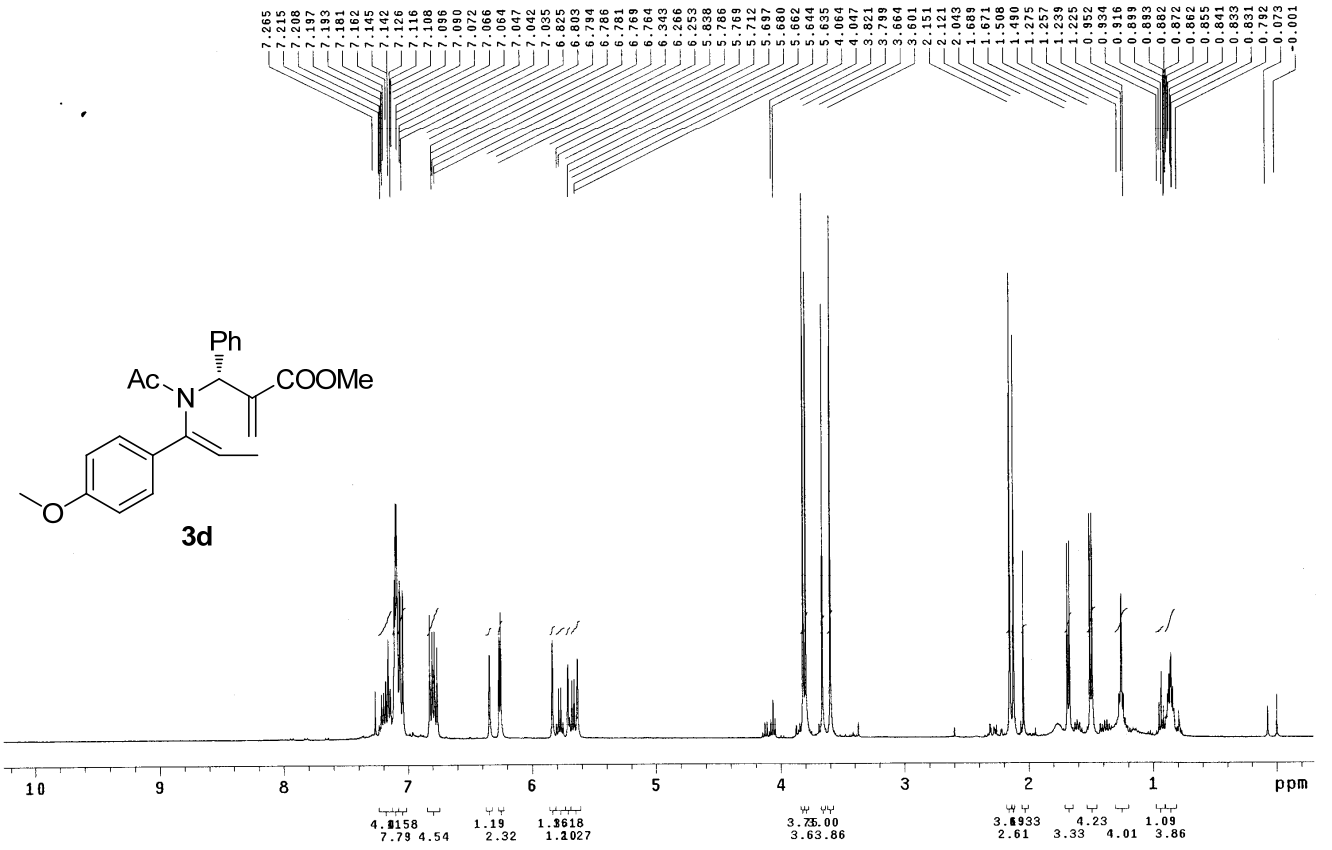
136.992
135.946
133.859
130.108
129.877
129.052
128.472
128.384
127.996
127.866
127.743
127.670
127.616
127.148
127.048
126.268
124.871
77.377
77.058
76.744
62.319
61.213
51.945
51.887
22.496
22.354
14.649
14.480
Ph
Ac
N
COOMe
Cl
3c
200
180
160
140
120
100
80
60
40
20
ppm

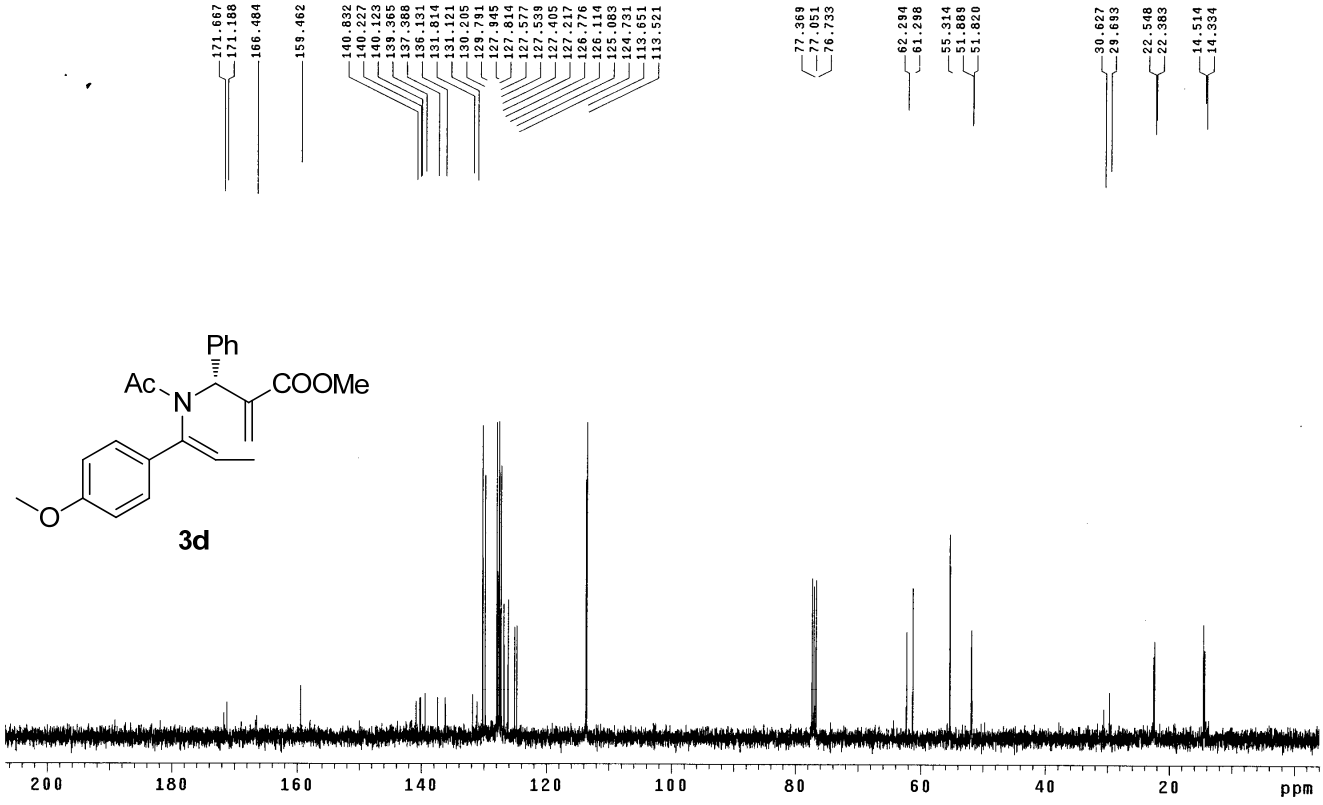


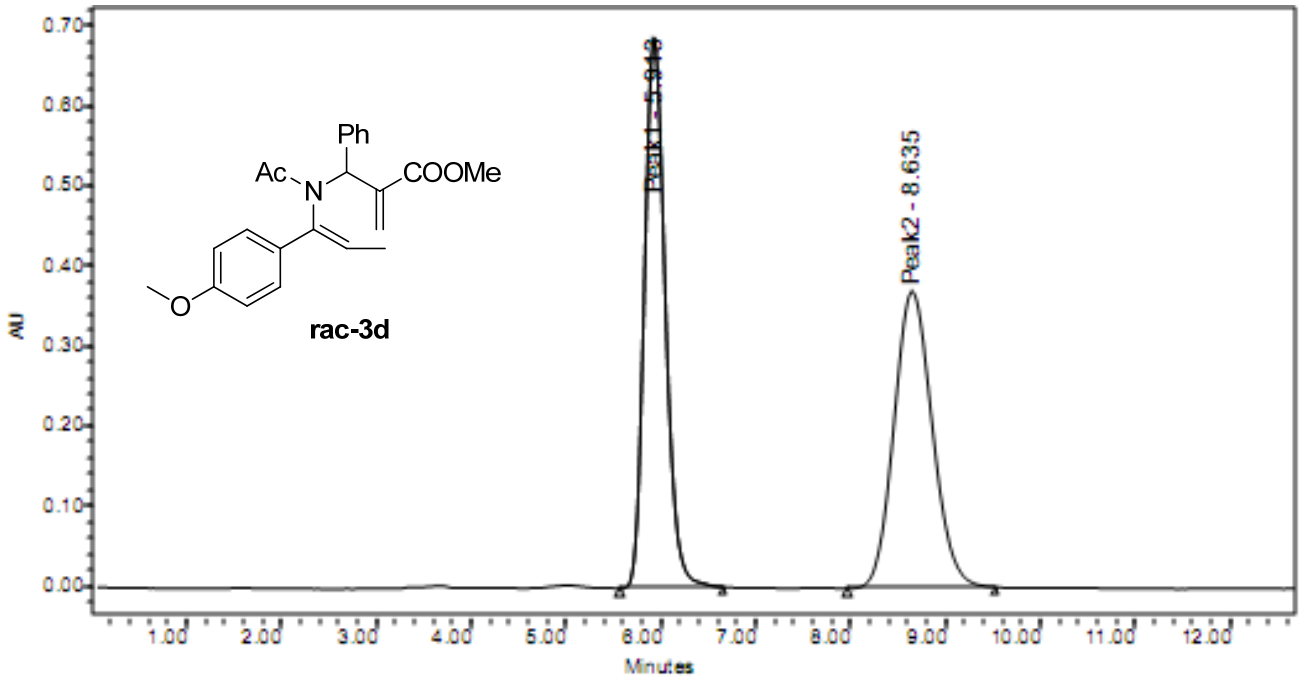
	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	4.822	9894774	47.17	914855	47.02
2	5.324	11082667	52.83	1030638	52.98



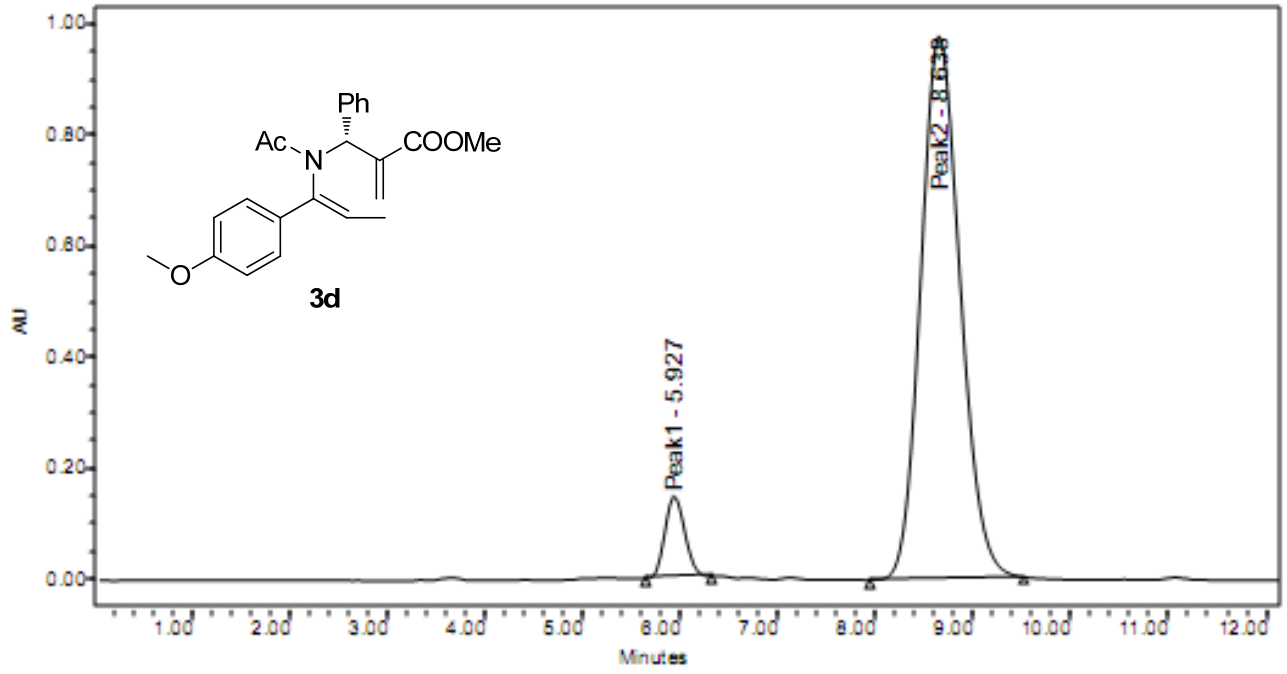
	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	5.297	515117	6.59	39907	5.69
2	5.930	7301502	93.41	661558	94.31


Ph
Ac
N
COOMe
O
3d
ppm

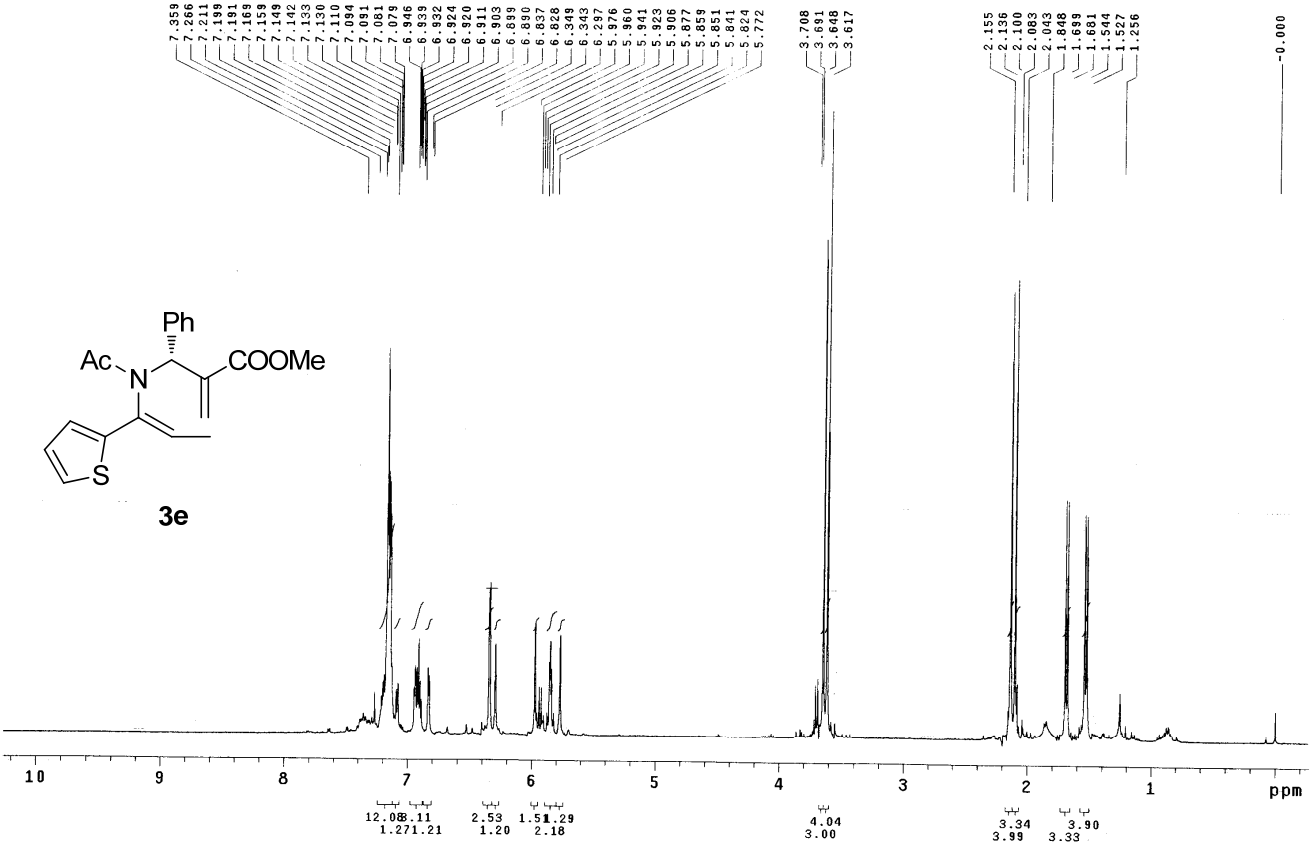

Ph
Ac
N
COOMe
O
3d
ppm



	Peak Name	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	Peak1	5.913	10483837	50.20	687974	64.99
2	Peak2	8.635	10401412	49.80	370646	35.01



	Peak Name	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	Peak1	5.927	2080576	7.06	145694	12.98
2	Peak2	8.638	27392832	92.94	976331	87.02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7.211
7.199
7.191
7.169
7.159
7.149
7.142
7.133
7.130
7.110
7.094
7.091
7.081
7.079
6.946
6.933
6.932
6.924
6.920
6.911
6.903
6.899
6.890
6.837
6.828
6.349
6.343
6.297
5.976
5.960
5.941
5.923
5.906
5.877
5.859
5.851
5.841
5.824
5.772
3.708
3.691
3.648
3.617
2.155
2.136
2.100
2.083
2.043
1.848
1.699
1.681
1.544
1.527
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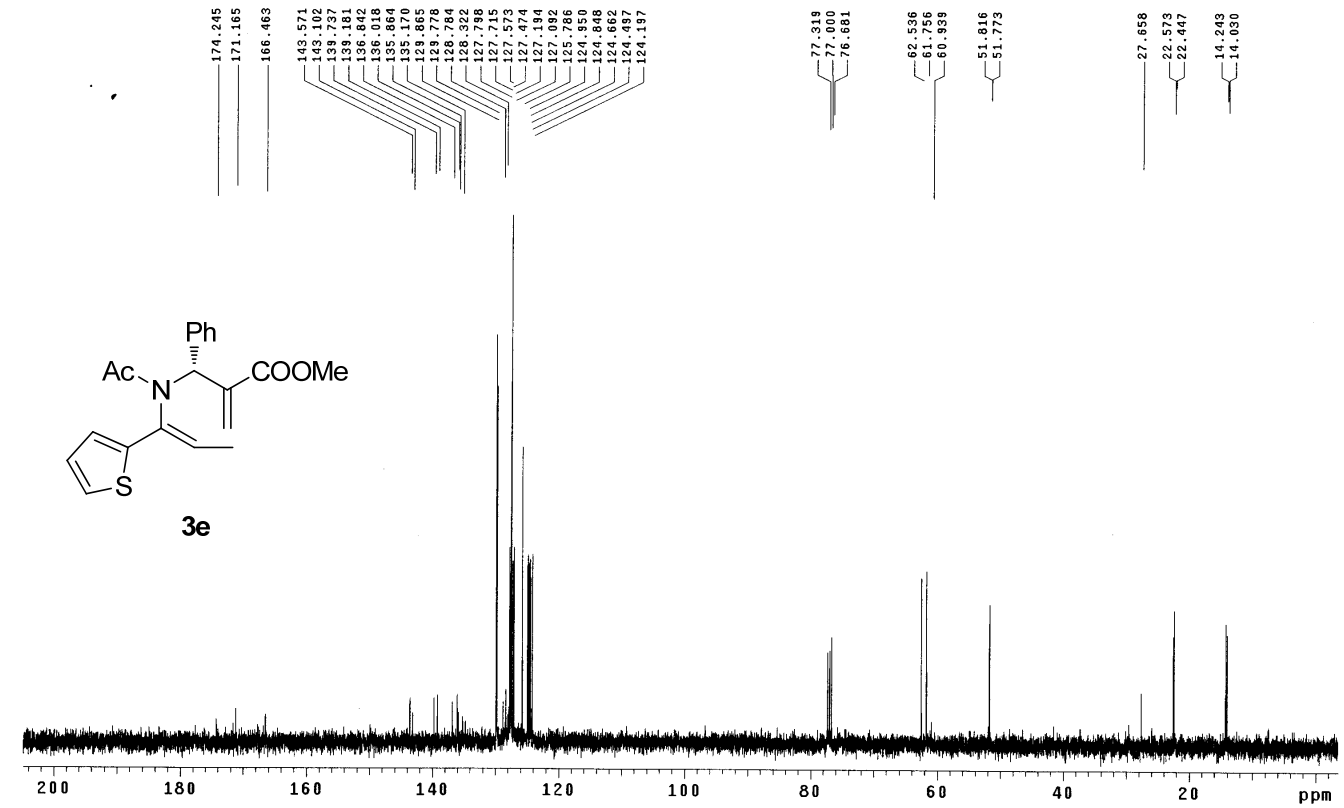
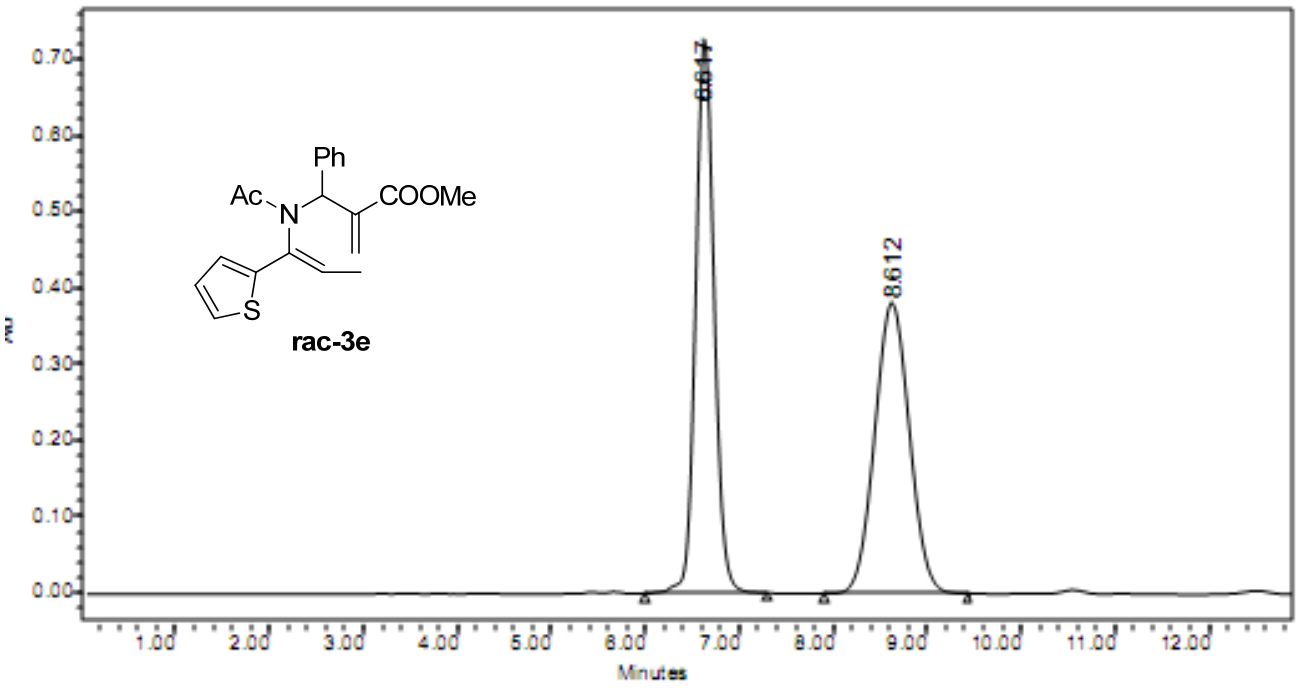
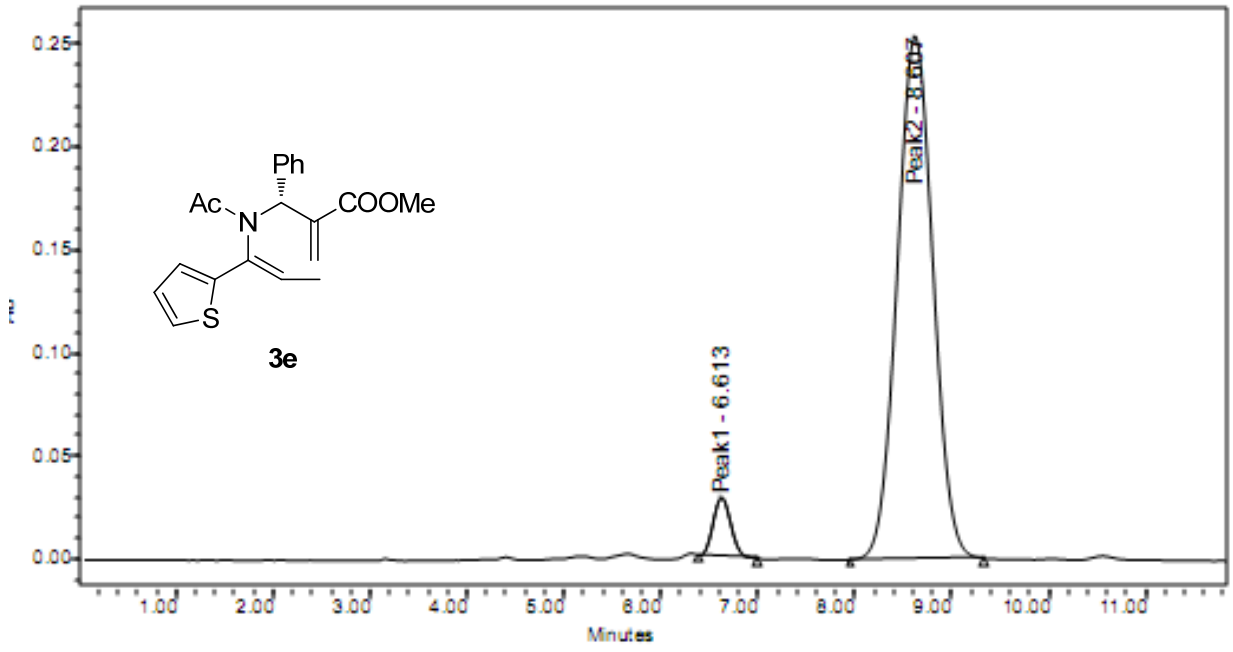
Ph
Ac
N
COOMe
S
3e
10
9
8
7
6
5
4
3
2
1
ppm
12.08
8.11
1.27
1.21
2.53
1.20
1.51
1.29
2.18
4.04
3.00
3.34
3.99
3.90
3.33

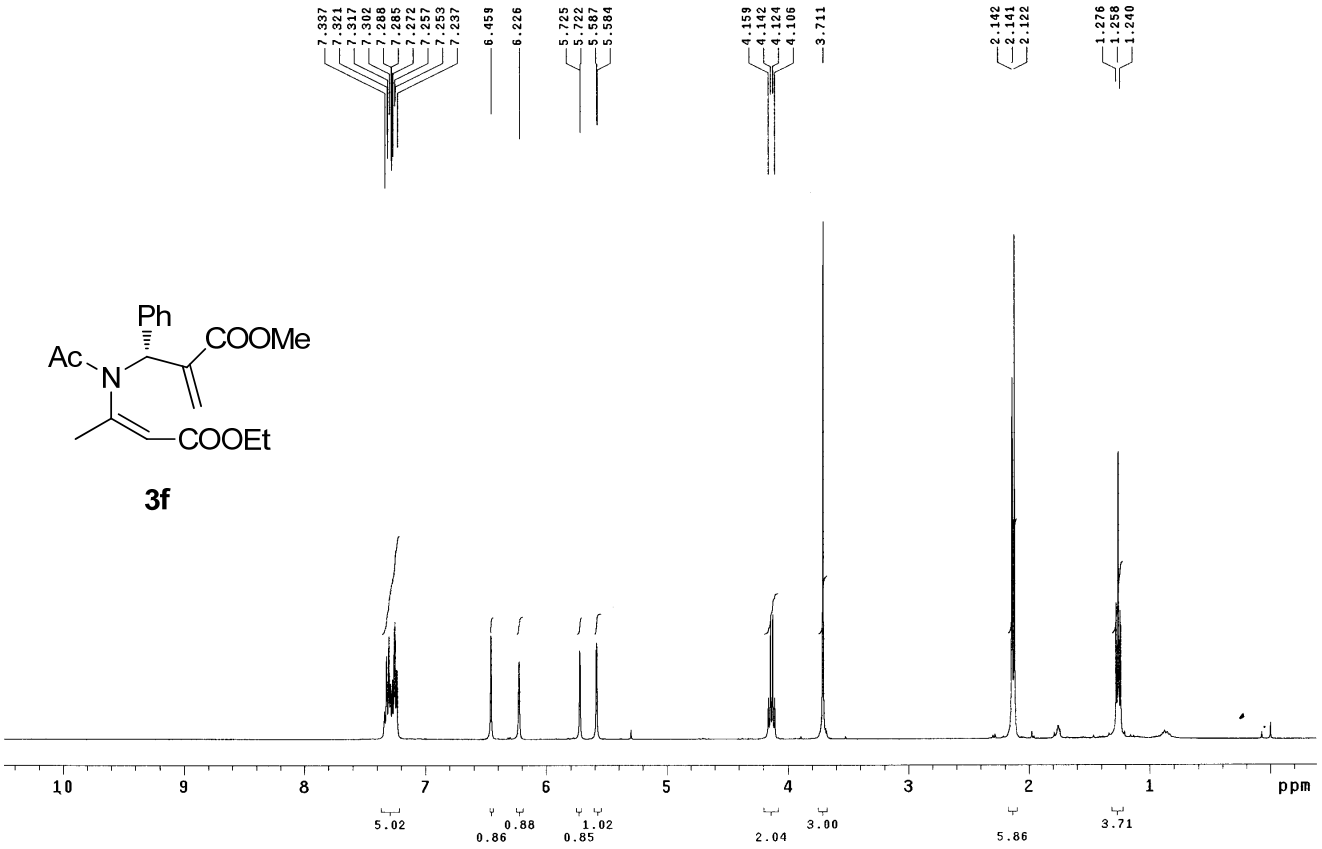

174.245
171.165
166.463
143.571
143.102
139.737
139.181
136.842
136.018
135.864
135.170
129.865
129.778
128.784
128.322
127.798
127.715
127.573
127.474
127.194
127.092
125.786
124.950
124.848
124.662
124.497
124.197
77.319
77.000
76.681
62.536
61.756
60.939
51.816
51.773
27.658
22.573
22.447
14.243
14.030
Ph
Ac
N
COOMe
S
3e
200
180
160
140
120
100
80
60
40
20
ppm



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	6.617	9979054	50.45	726168	65.58
2	8.612	9801535	49.55	381142	34.42



	Peak Name	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	Peak1	6.613	365375	5.32	28710	10.16
2	Peak2	8.607	6507272	94.68	254004	89.84


7.337
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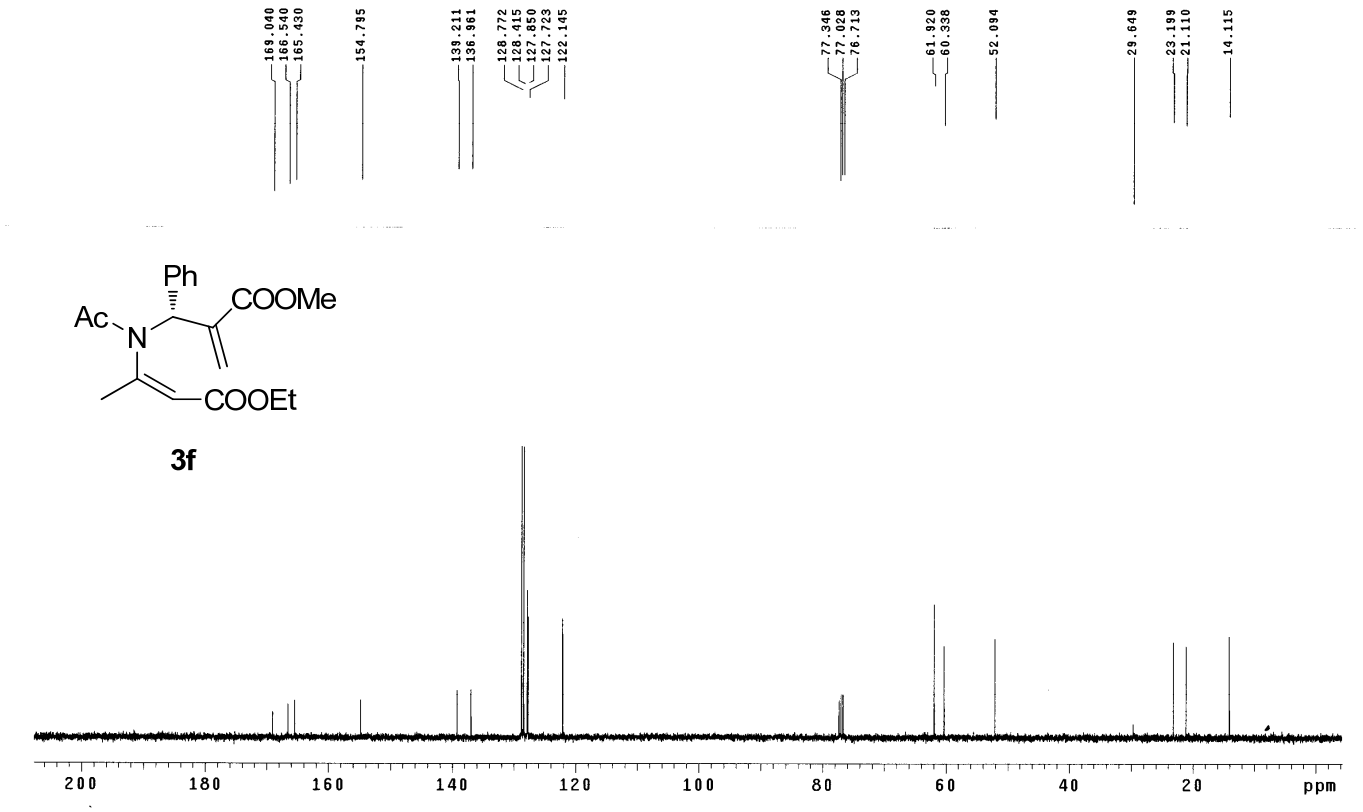
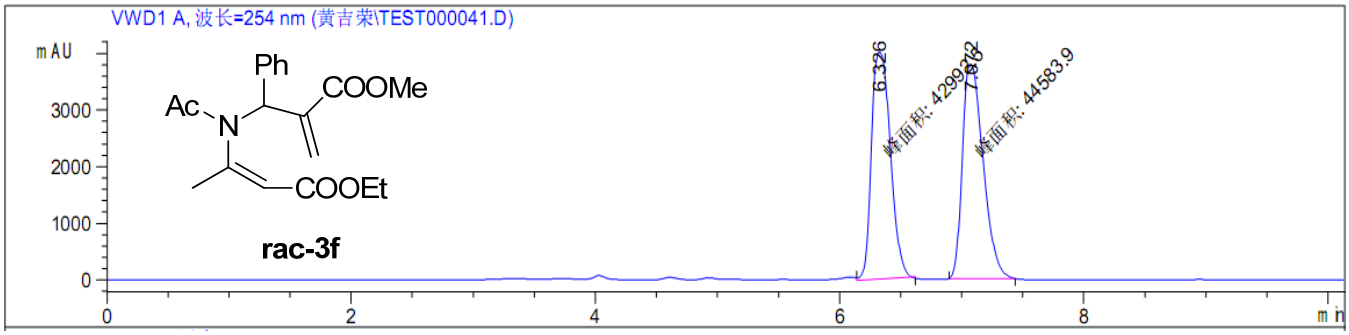
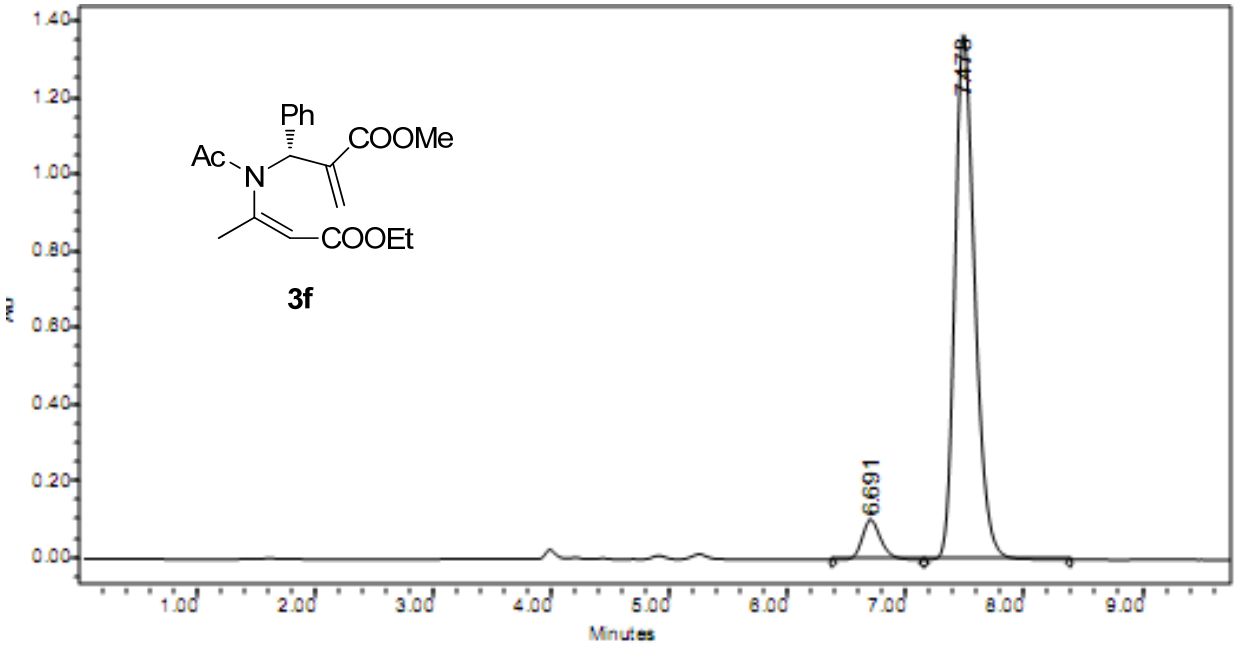
7.317
7.302
7.288
7.285
7.272
7.257
7.253
7.237
6.459
6.226
5.725
5.722
5.587
5.584
4.159
4.142
4.124
4.106
3.711
2.142
2.141
2.122
1.276
1.258
1.240
Ph
COOMe
Ac
N
COOEt
3f
10
9
8
7
6
5
4
3
2
1
ppm
5.02
0.86
0.88
0.85
1.02
2.04
3.00
5.86
3.71


169.040
166.540
165.430
154.795
139.211
136.961
128.772
128.415
127.850
127.723
122.145
77.346
77.028
76.713
61.920
60.338
52.094
29.649
23.199
21.110
14.115
Ph
COOMe
Ac
N
COOEt
3f
200
180
160
140
120
100
80
60
40
20
ppm

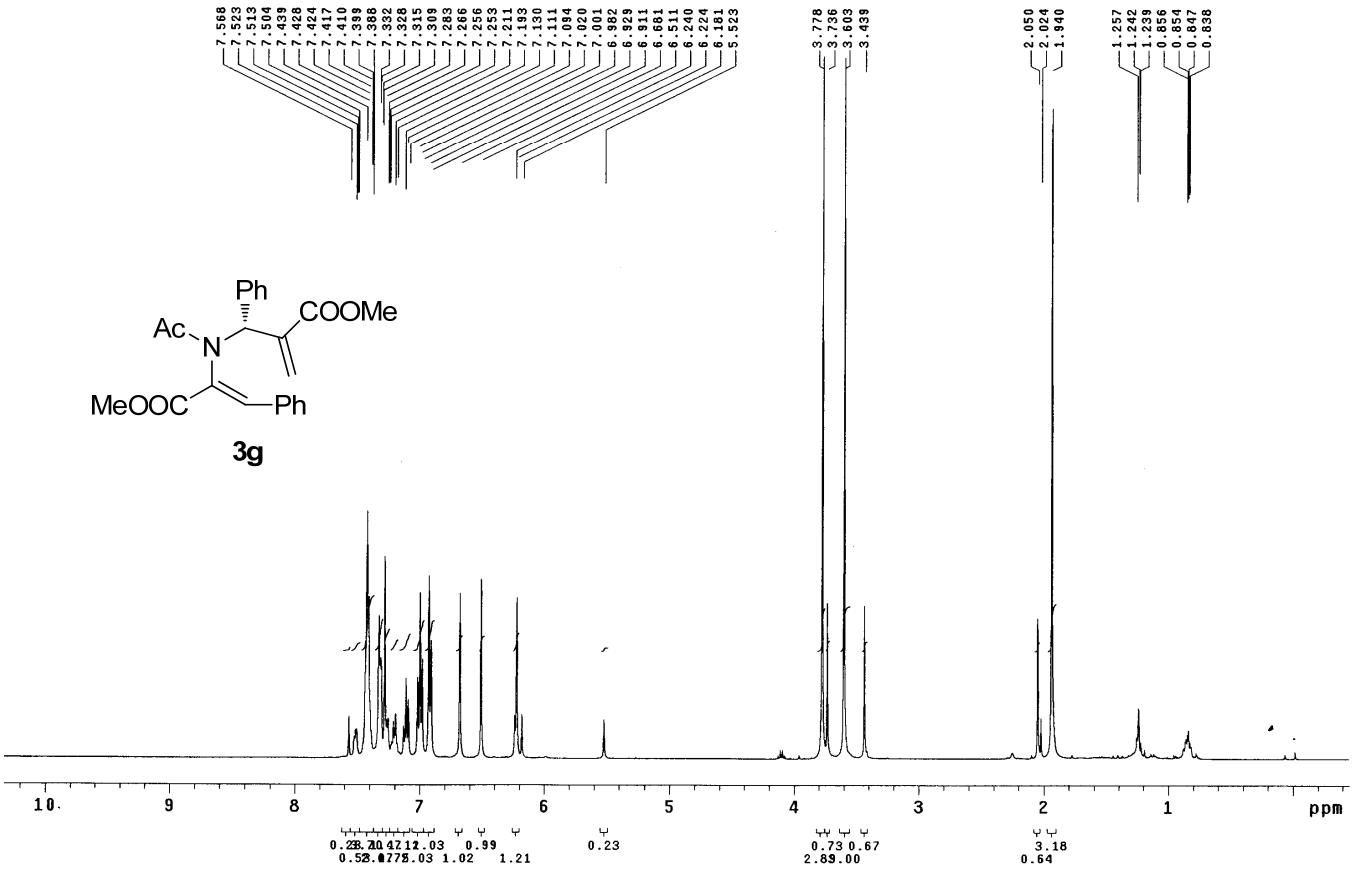


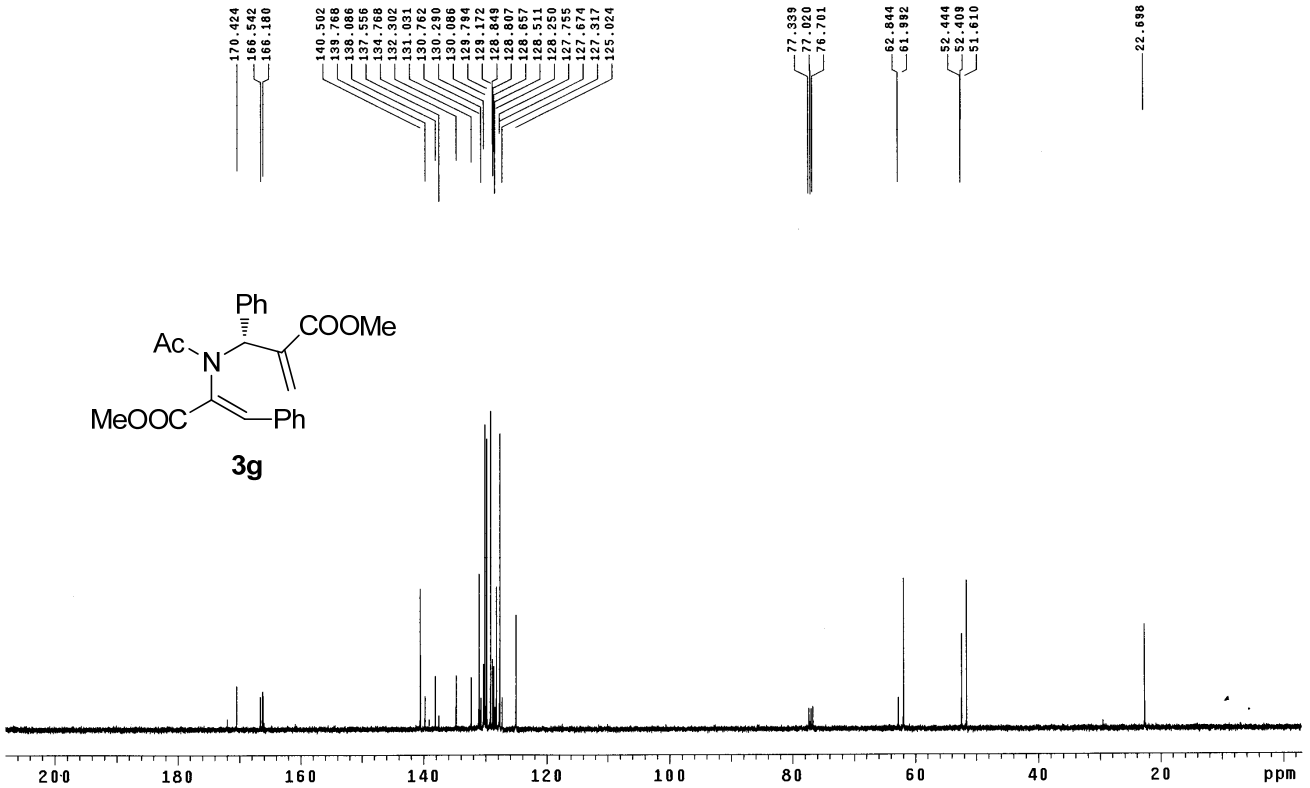
信号 1: VWD1 A, 波长=254 nm

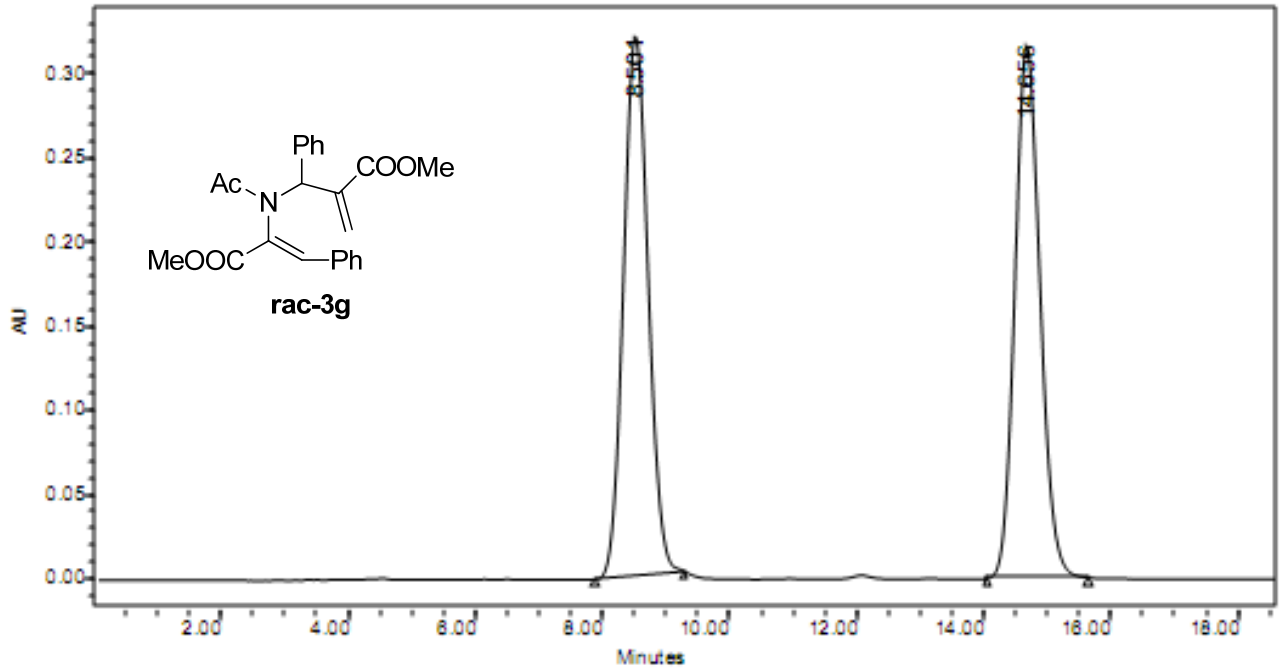
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	6.326	MM	0.1790	4.29925e4	4003.25488	49.0914
2	7.072	MM	0.1959	4.45839e4	3792.66479	50.9086



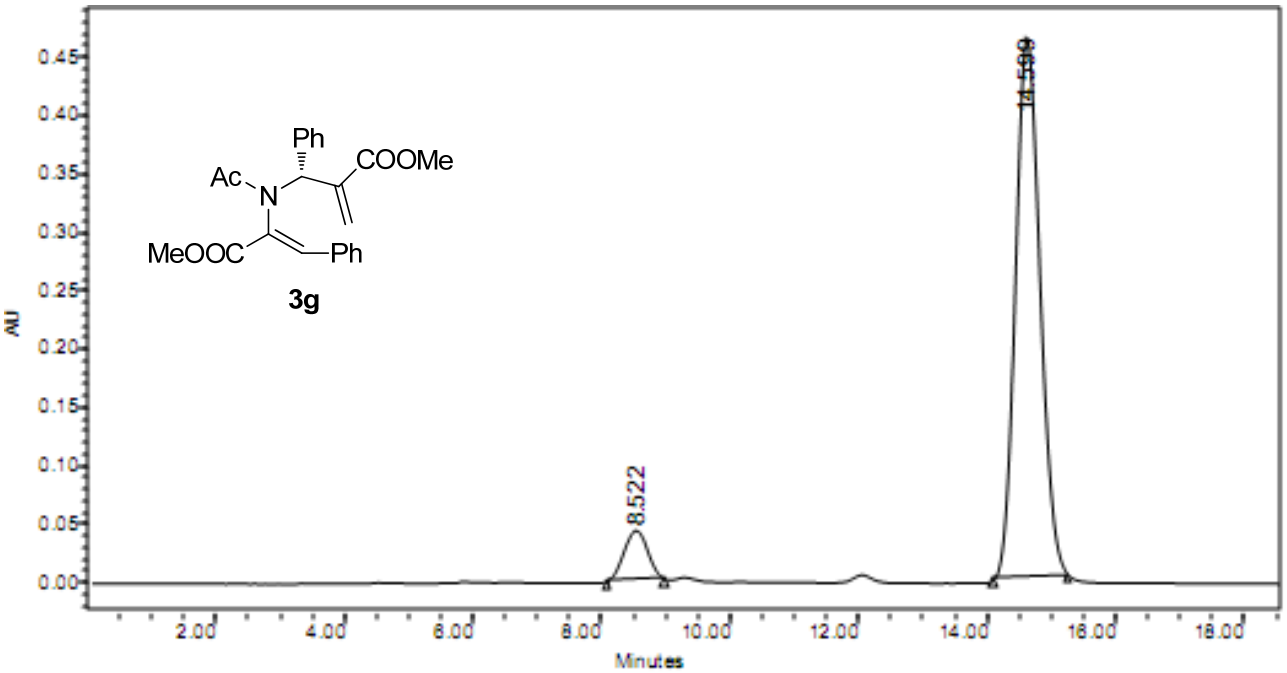
	RT (min)	Area (V*sec)	% Area	Height (V)	% Height
1	6.691	1142151	6.66	103523	7.02
2	7.478	16014926	93.34	1370909	92.98


Ph
Ac
N
COOMe
MeOOC
Ph
3g
ppm

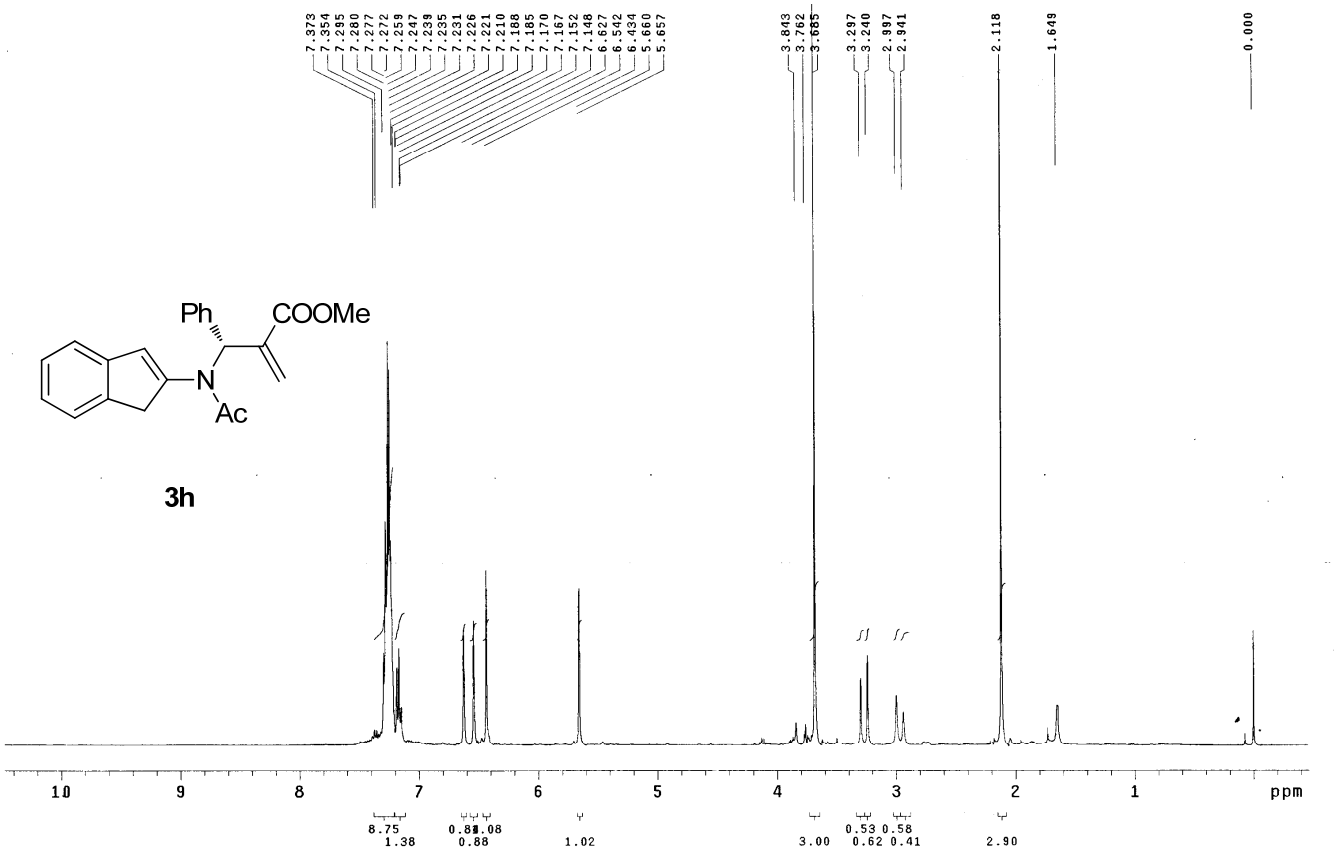

Ph
Ac
N
COOMe
MeOOC
Ph
3g
ppm



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	8.501	8633119	49.79	320316	50.30
2	14.656	8704258	50.21	316467	49.70



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	8.522	1075953	8.07	42074	8.34
2	14.599	12249129	91.93	462649	91.66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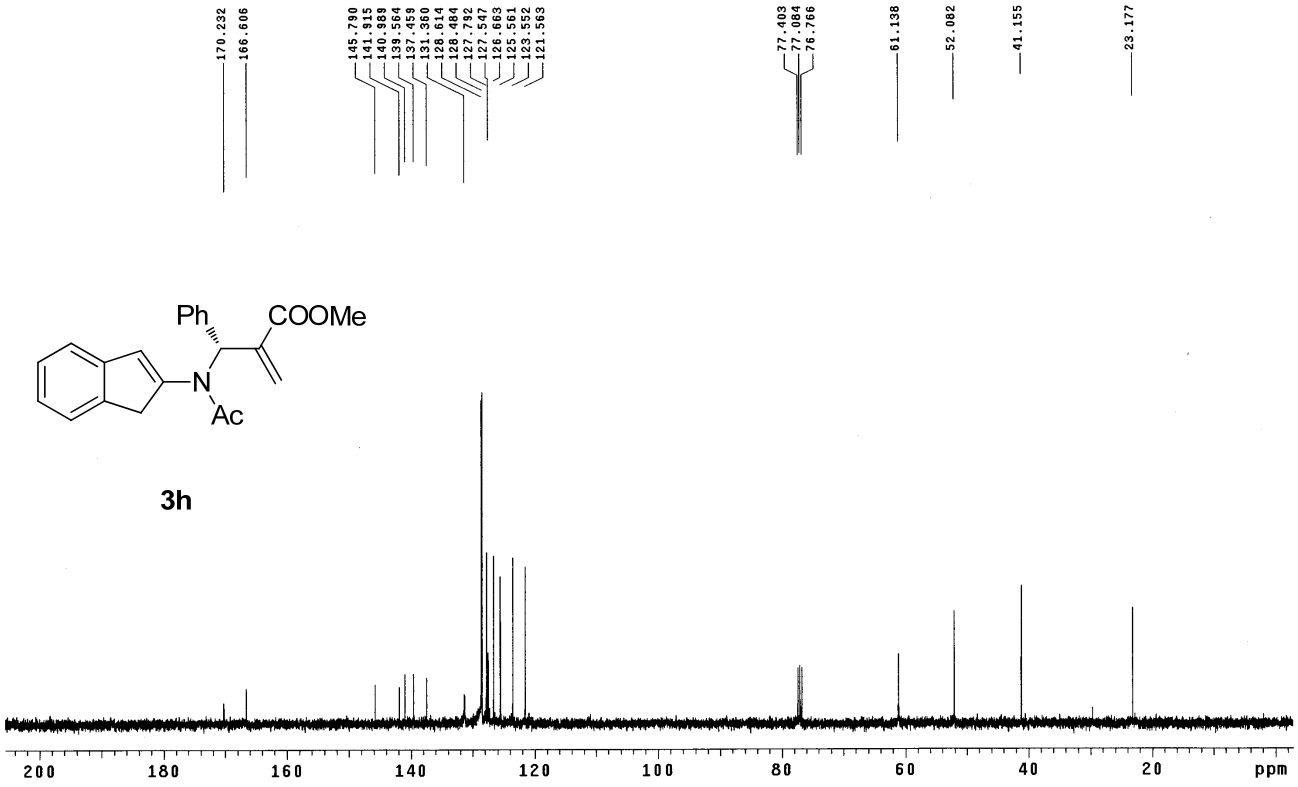
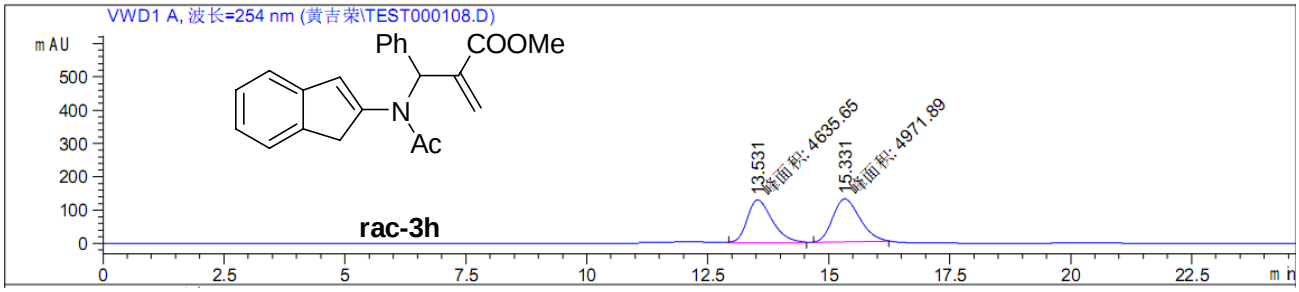
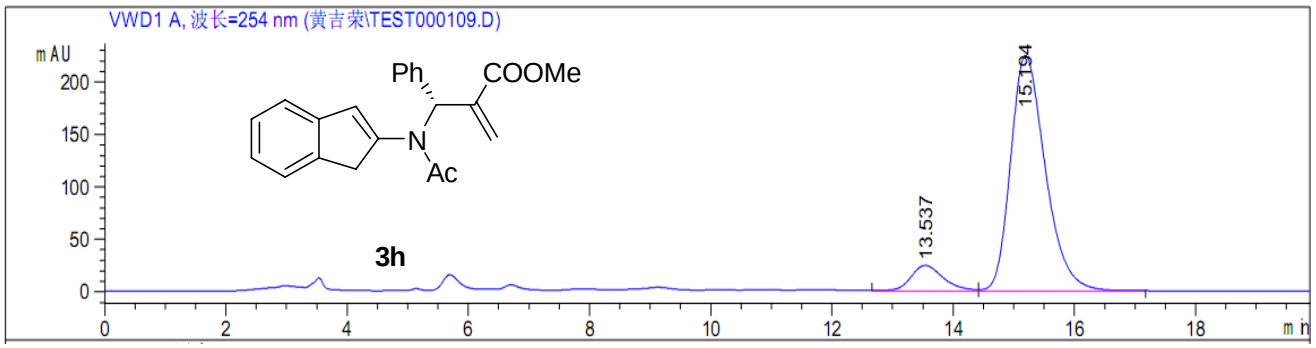
7.280
7.277
7.272
7.259
7.247
7.239
7.235
7.231
7.226
7.221
7.210
7.188
7.185
7.170
7.167
7.152
7.148
6.627
6.542
6.434
5.660
5.657
3.843
3.762
3.685
3.297
3.240
2.997
2.941
2.118
1.649
0.000
Ph
COOMe
N
Ac
3h
10
9
8
7
6
5
4
3
2
1
ppm
8.75
1.38
0.88
1.08
1.02
3.00
0.53
0.62
0.58
0.41
2.90


170.232
166.606
145.790
141.815
140.989
139.564
137.459
131.360
128.614
128.484
127.792
127.547
126.663
125.561
123.552
121.563
77.403
77.084
76.766
61.138
52.082
41.155
23.177
Ph
COOMe
N
Ac
3h
200
180
160
140
120
100
80
60
40
20
ppm



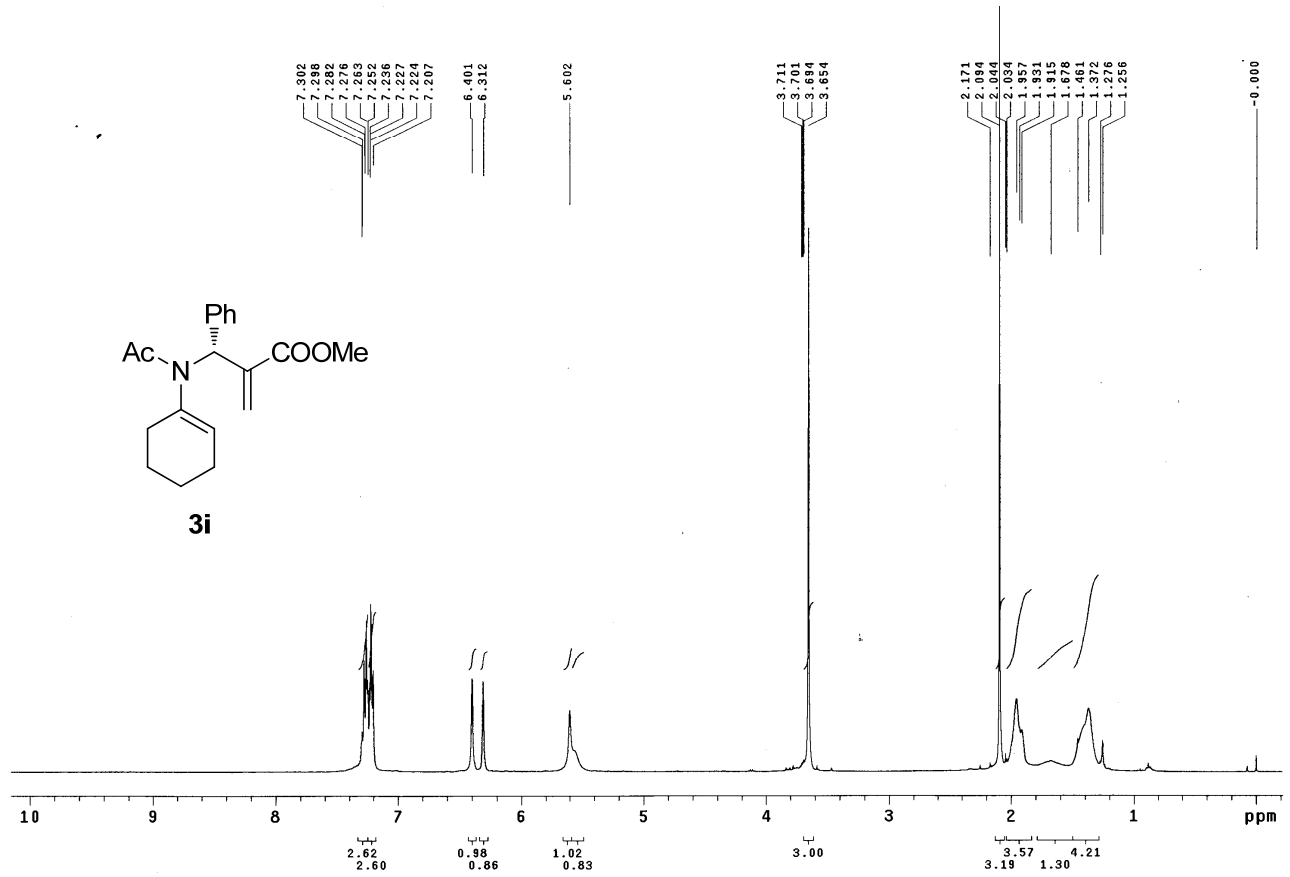
信号 1: VWD1 A, 波长=254 nm

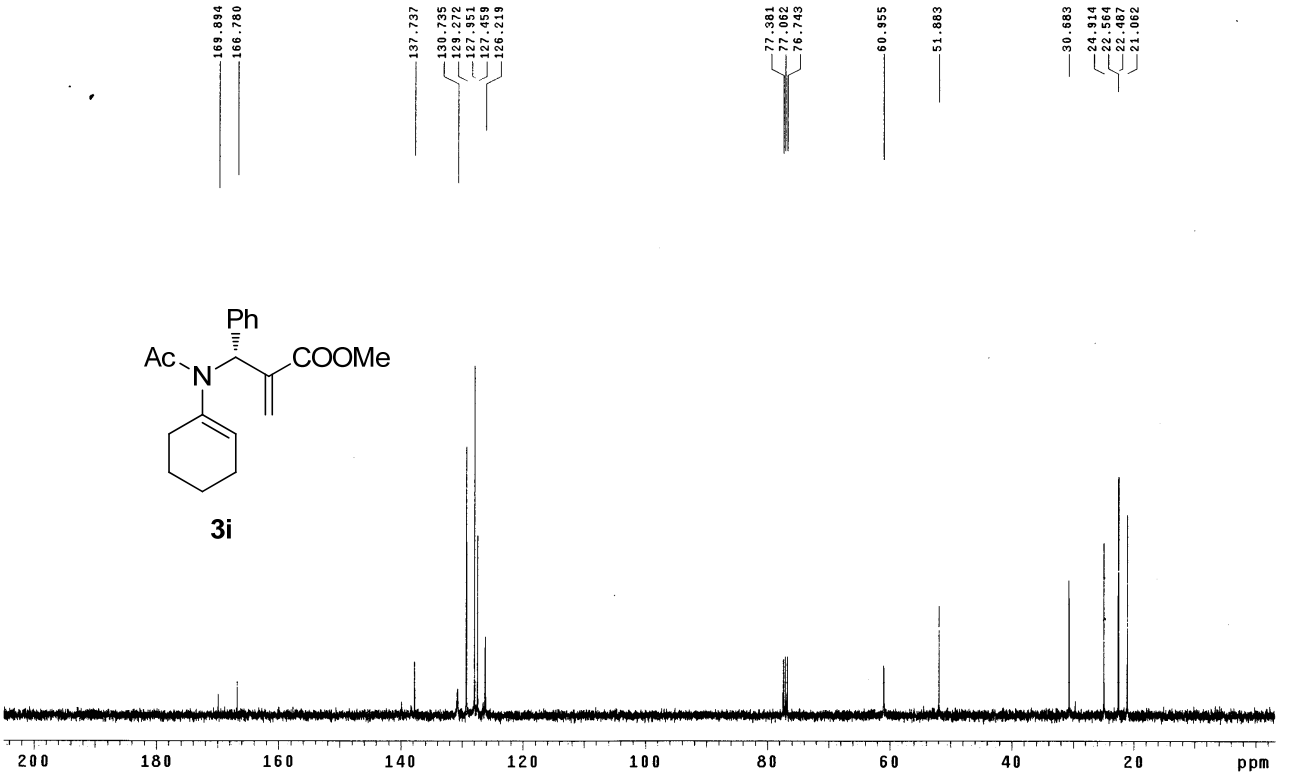
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	13.531	MM	0.6010	4635.65332	128.55125	48.2501
2	15.331	MM	0.6472	4971.89307	128.04141	51.7499

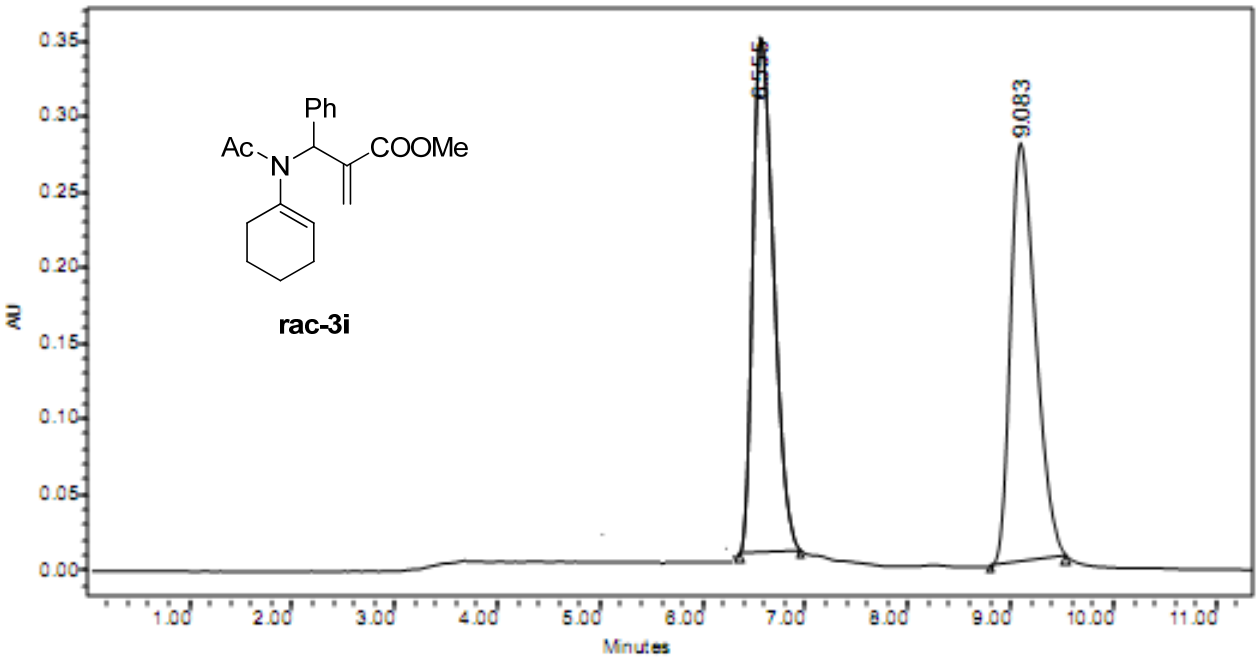


信号 1: VWD1 A, 波长=254 nm

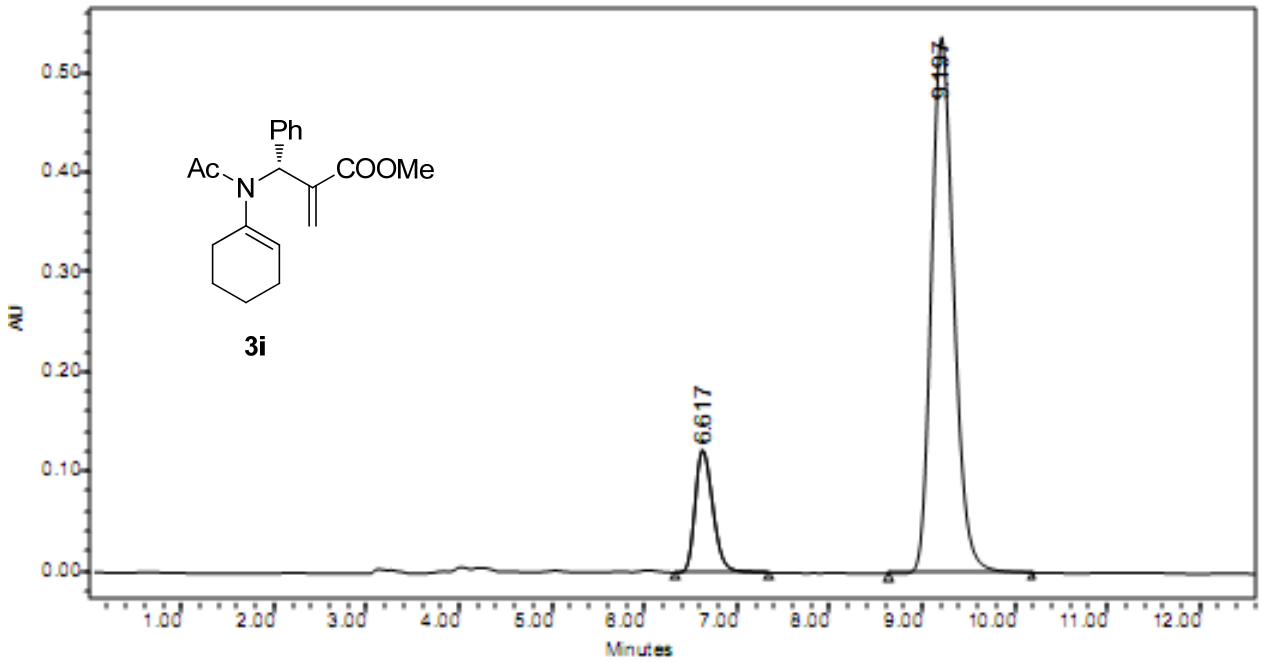
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	13.537	VV	0.5669	900.13379	24.25486	9.1715
2	15.194	VB	0.6117	8914.34375	224.51759	90.8285


7.302
7.298
7.282
7.276
7.263
7.252
7.236
7.227
7.224
7.207
6.401
6.312
5.602
3.711
3.701
3.694
3.654
2.171
2.094
2.044
2.034
1.957
1.931
1.915
1.678
1.461
1.372
1.276
1.256
-0.000
Ph
Ac
N
COOMe
3i
10
9
8
7
6
5
4
3
2
1
ppm
2.62
2.60
0.98
0.86
1.02
0.83
3.00
3.57
3.19
4.21
1.30

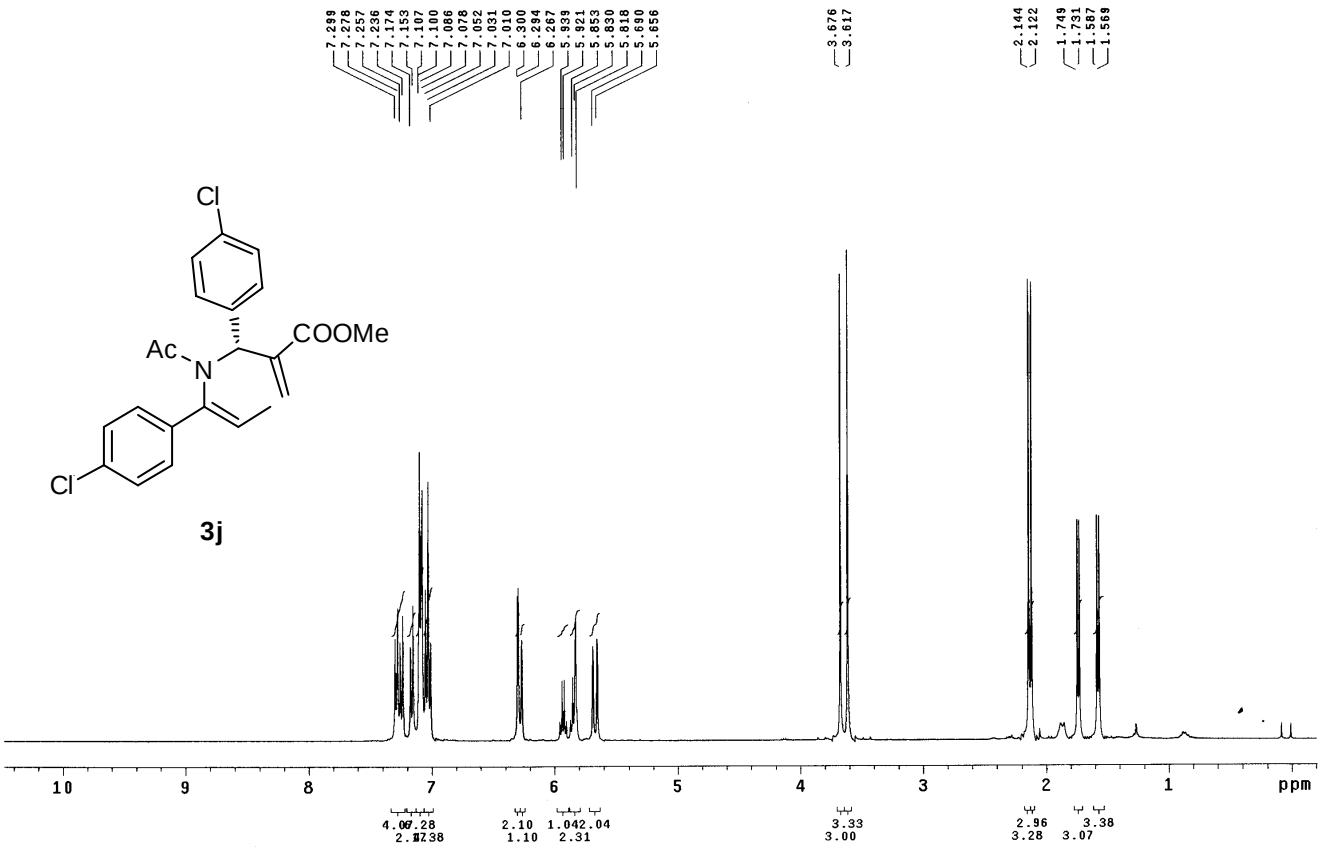

169.894
166.780
137.737
130.735
129.272
127.951
127.459
126.219
77.381
77.062
76.743
60.955
51.883
30.683
24.914
22.564
22.487
21.062
Ph
Ac
N
COOMe
3i
200
180
160
140
120
100
80
60
40
20
ppm

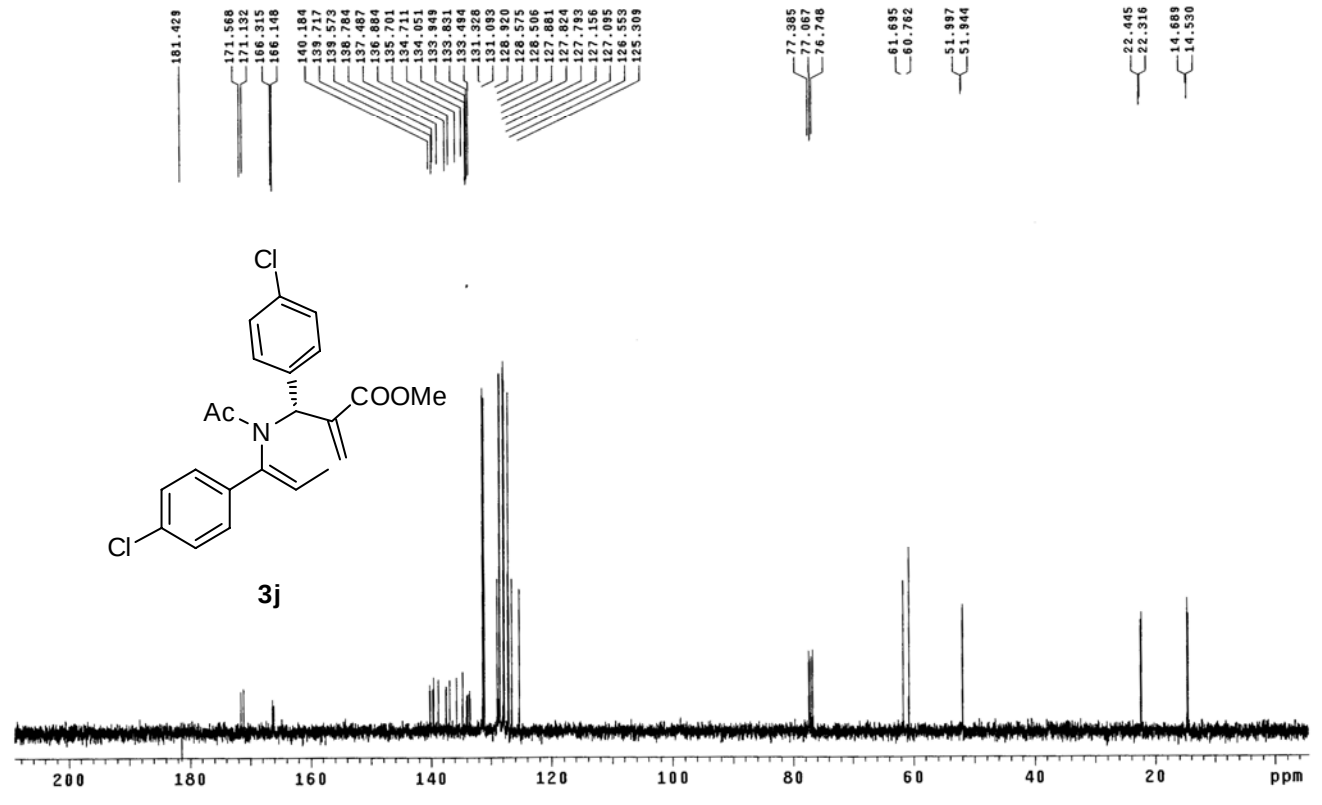


	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	6.555	4802605	50.39	341544	55.22
2	9.083	4727620	49.61	276922	44.78



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	6.617	1655113	15.48	122617	18.57
2	9.197	9034446	84.52	537663	81.43


7.299
7.278
7.257
7.236
7.174
7.153
7.107
7.100
7.086
7.078
7.052
7.031
7.010
6.300
6.294
6.267
5.939
5.921
5.853
5.830
5.818
5.690
5.656
3.676
3.617
2.144
2.122
1.749
1.731
1.587
1.569
Cl
Ac
N
COOMe
Cl
3j
10
9
8
7
6
5
4
3
2
1
ppm
4.07
7.28
2.10
1.10
1.04
2.31
2.04
3.33
3.00
2.96
3.28
3.38
3.07


181.429
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171.132
166.315
166.148
140.184
139.717
139.573
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137.487
136.884
135.701
134.711
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127.881
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127.793
127.156
127.095
126.553
125.309
77.385
77.067
76.748
61.695
60.762
51.997
51.944
22.445
22.316
14.689
14.530
Cl
Ac
N
COOMe
Cl
3j
200
180
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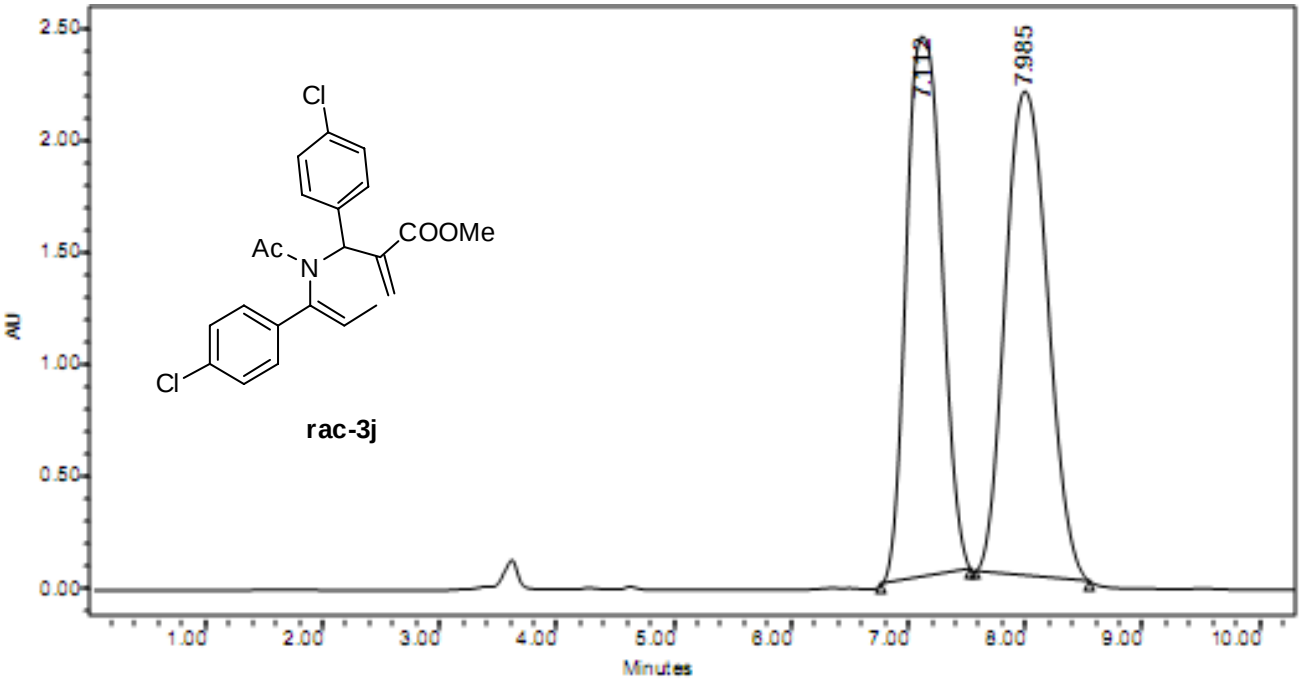
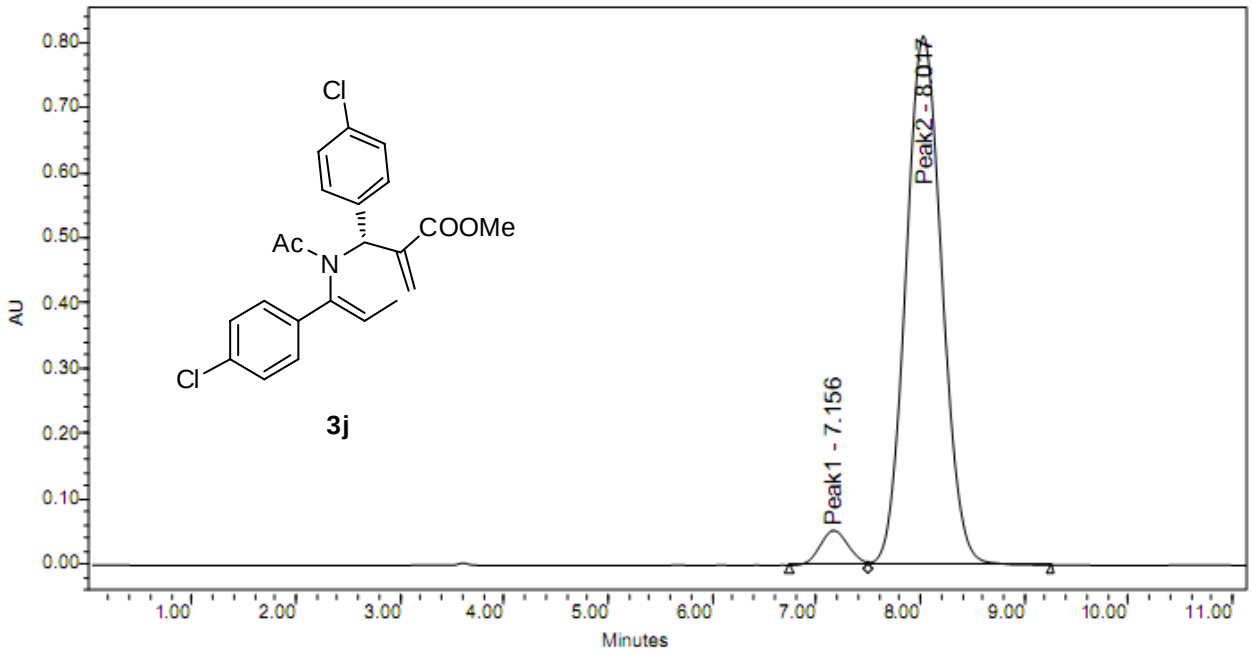
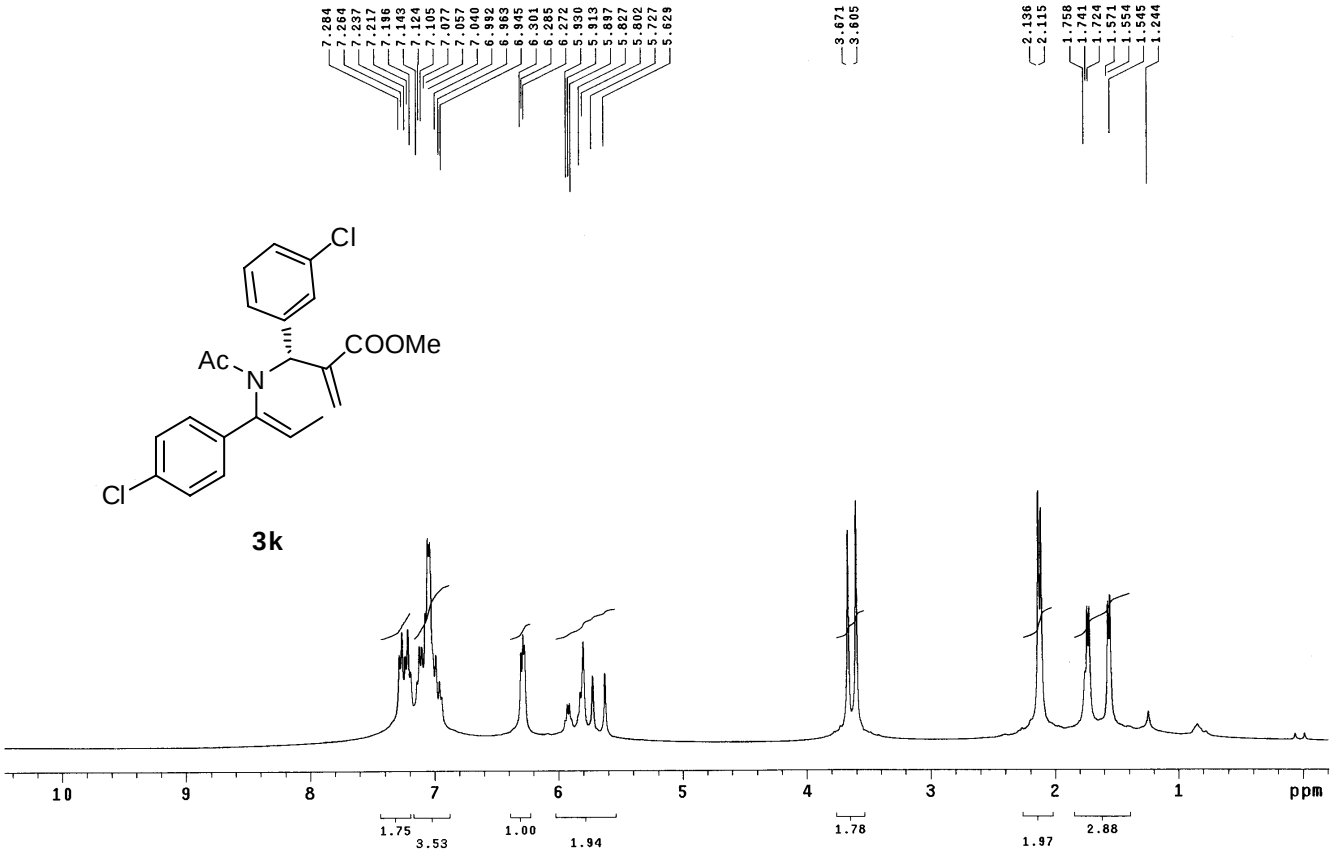
140
120
100
80
60
40
20
ppm



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	7.112	51050880	48.37	2414677	52.77
2	7.985	54500552	51.63	2160927	47.23



	Peak Name	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	Peak1	7.156	1023754	4.97	53230	6.15
2	Peak2	8.017	19582068	95.03	811648	93.85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7.237
7.217
7.196
7.143
7.124
7.105
7.077
7.057
7.040
6.992
6.963
6.945
6.301
6.285
6.272
5.930
5.913
5.897
5.827
5.802
5.727
5.629
3.671
3.605
2.136
2.115
1.758
1.741
1.724
1.571
1.554
1.545
1.244
Cl
Ac
N
COOMe
Cl
3k
10
9
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6
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4
3
2
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ppm
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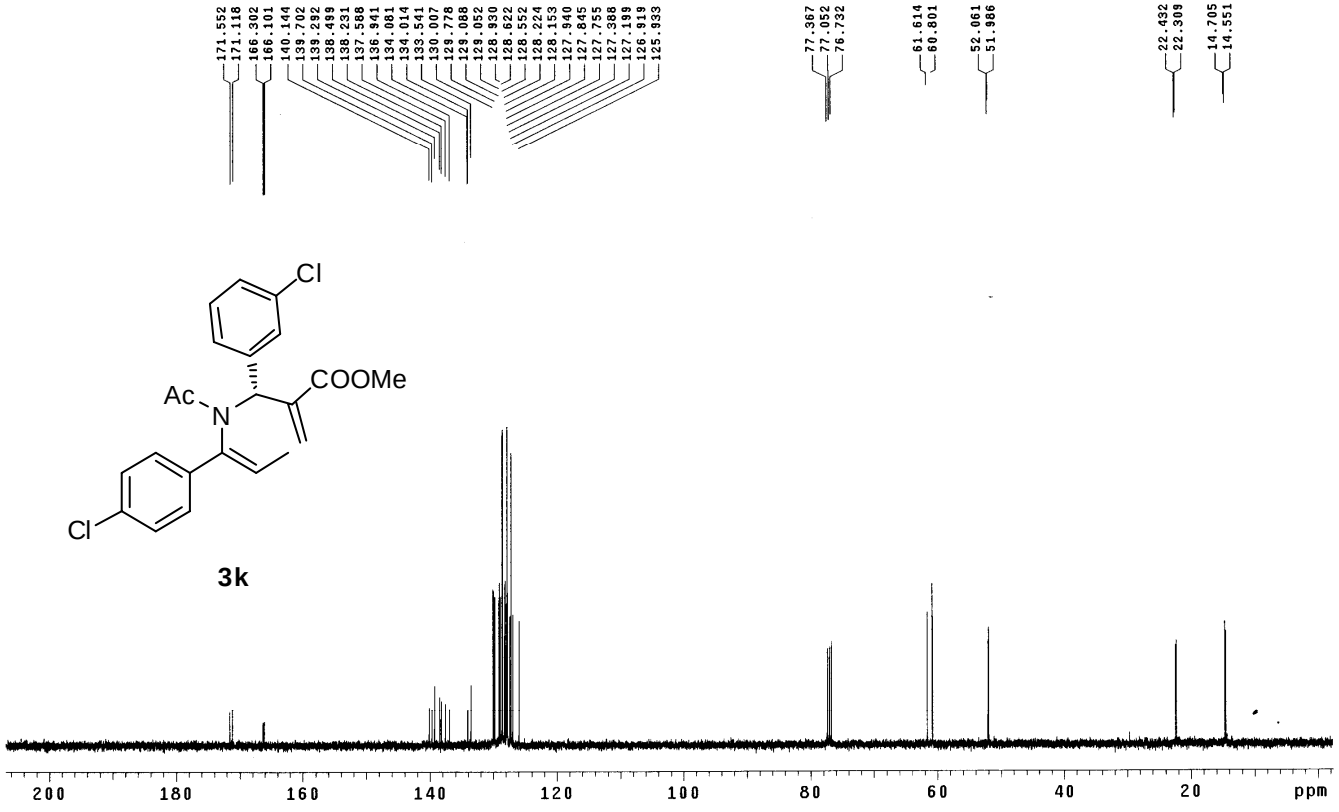
1.97
2.88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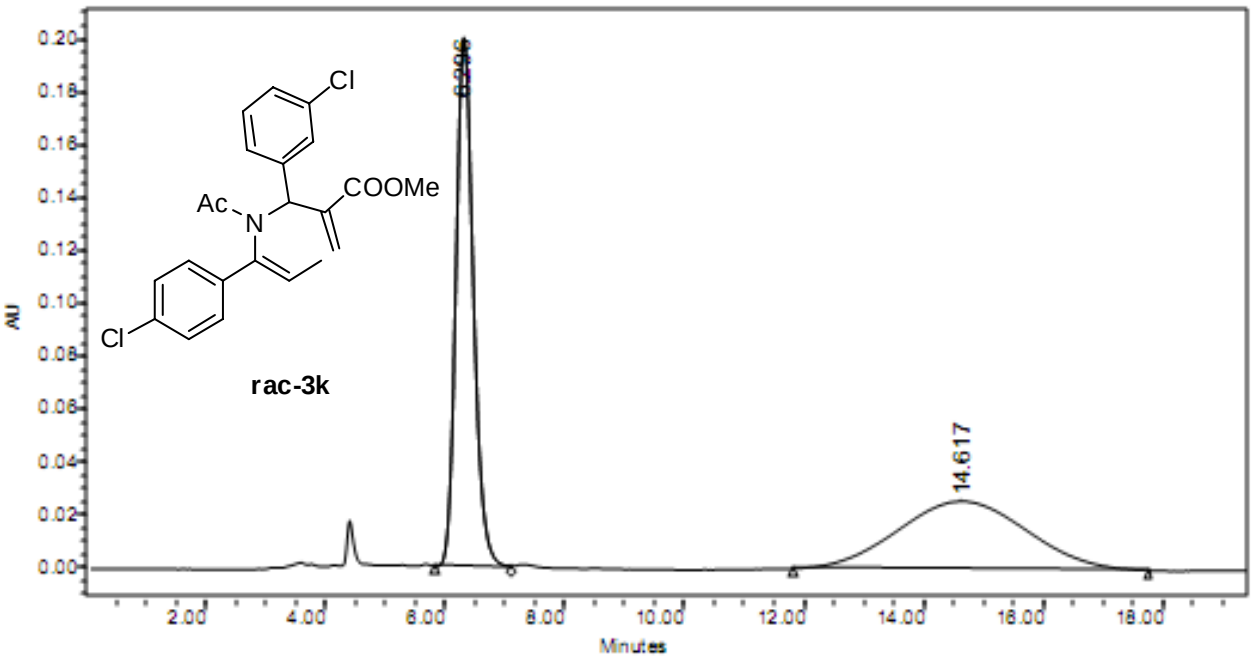
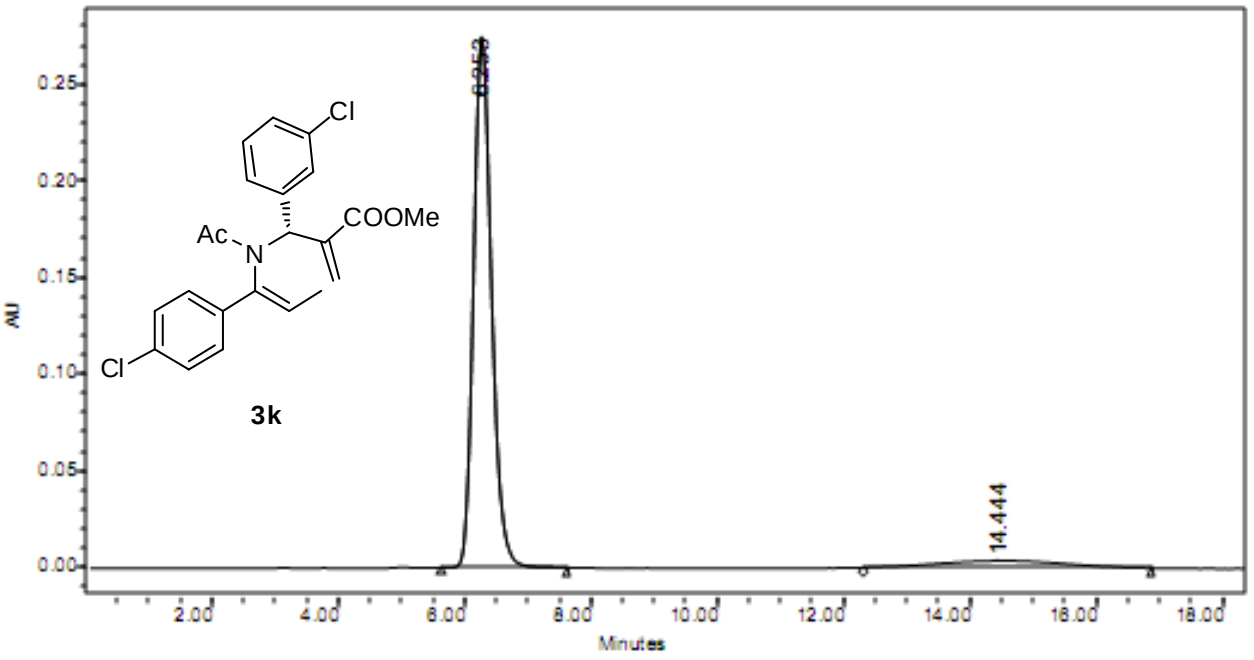
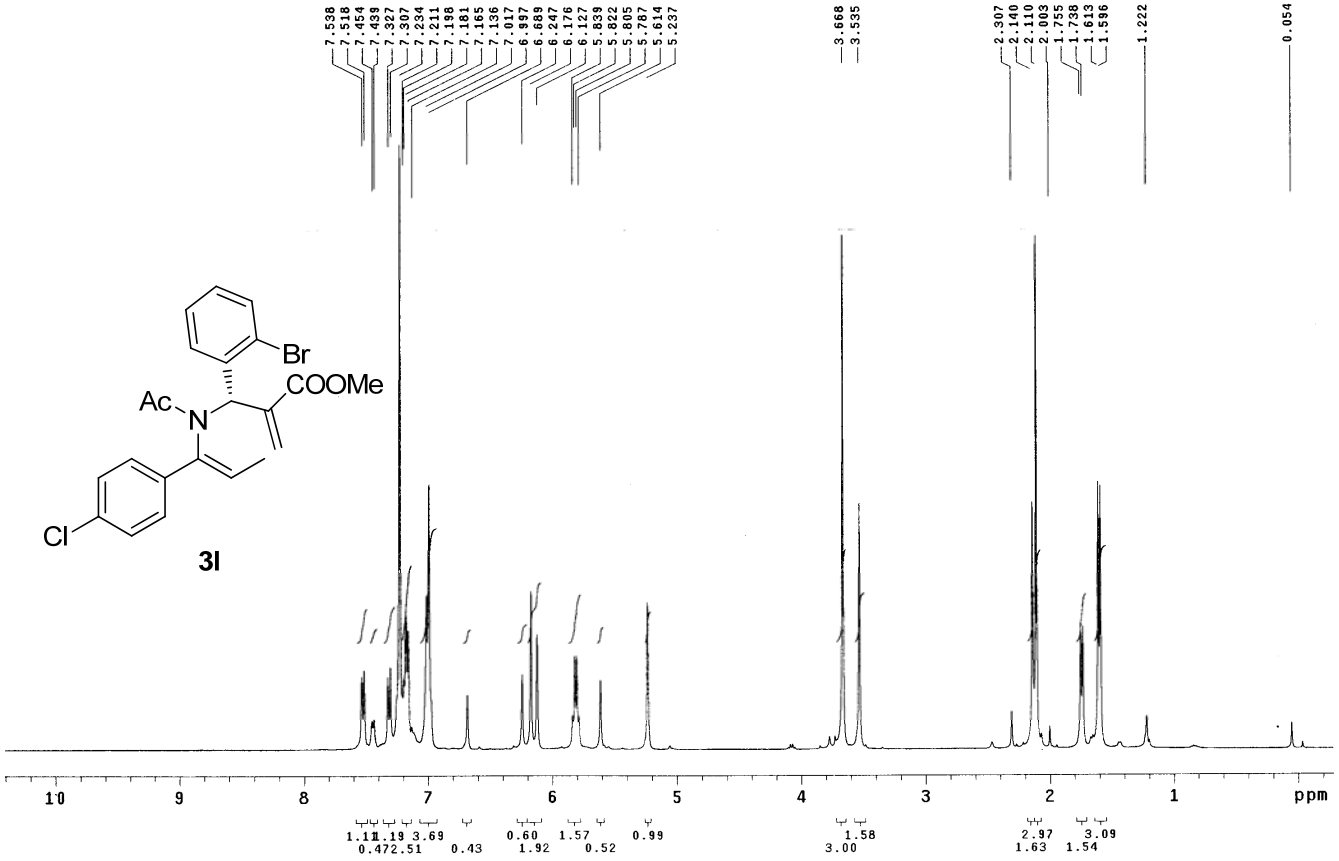
ppm



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	6.296	4070711	51.11	200240	88.58
2	14.617	3893221	48.89	25815	11.42



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	6.253	5438191	90.77	274673	98.63
2	14.444	552836	9.23	3824	1.37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7.234
7.211
7.188
7.181
7.165
7.156
7.017
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6.247
6.176
6.127
5.839
5.822
5.805
5.787
5.614
5.237
3.668
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COOMe
Ac
N
Cl
3l
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8
7
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5
4
3
2
1
ppm
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1.57
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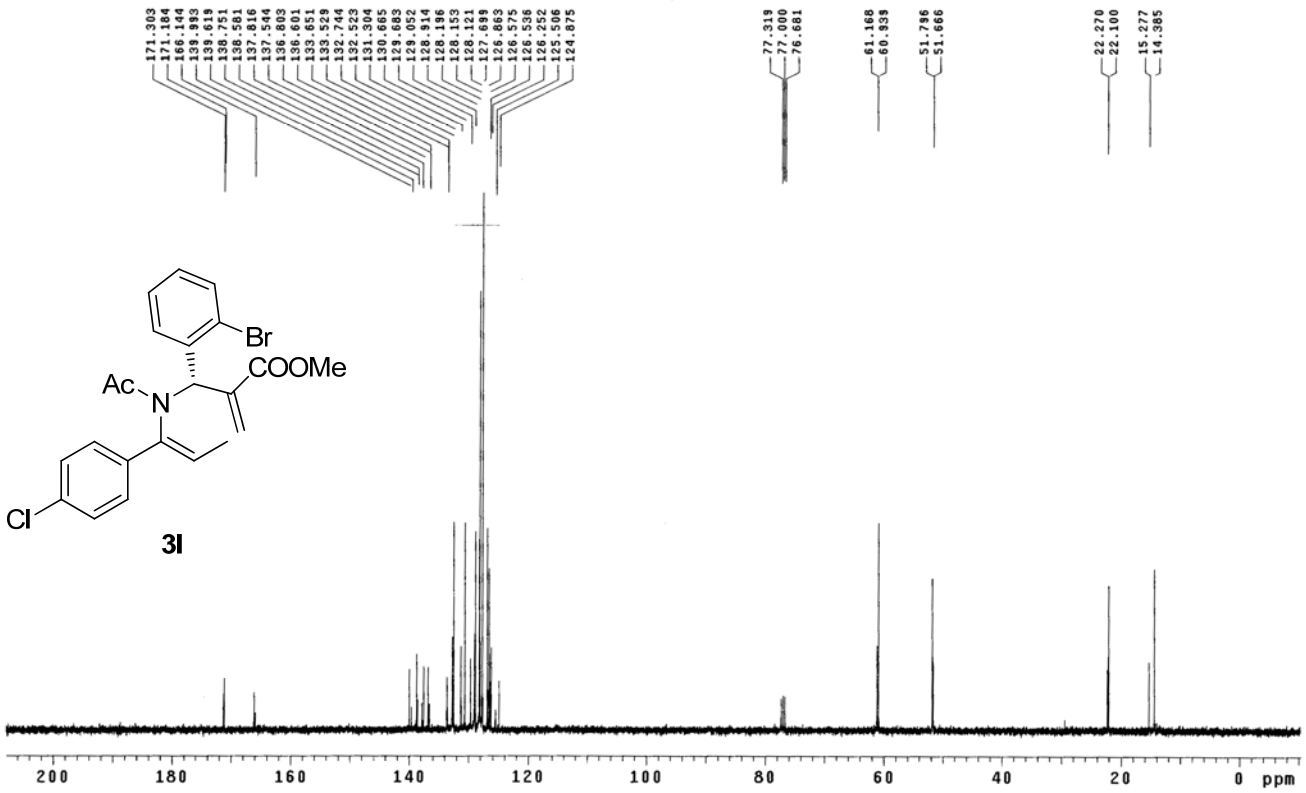
3.09
1.54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136.601
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132.744
132.523
131.304
130.665
129.683
129.052
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128.153
128.121
127.699
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126.575
126.536
126.252
125.506
124.875
77.319
77.000
76.681
61.168
60.933
51.796
51.666
22.270
22.100
15.277
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80
60
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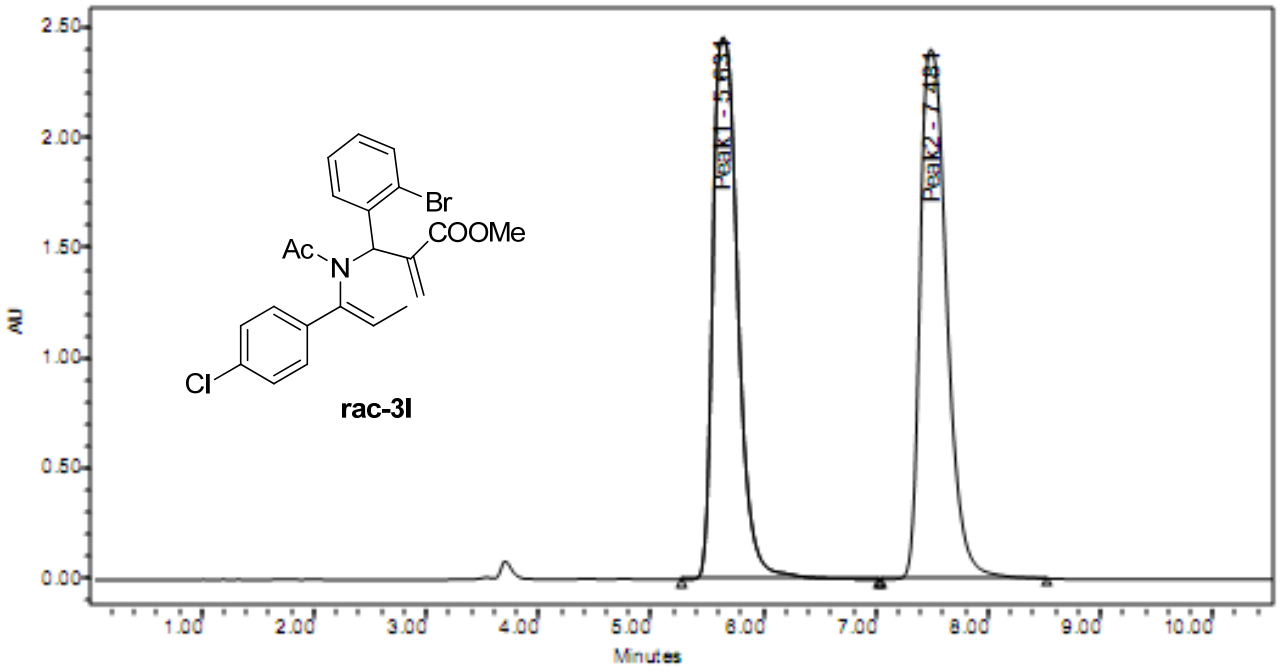
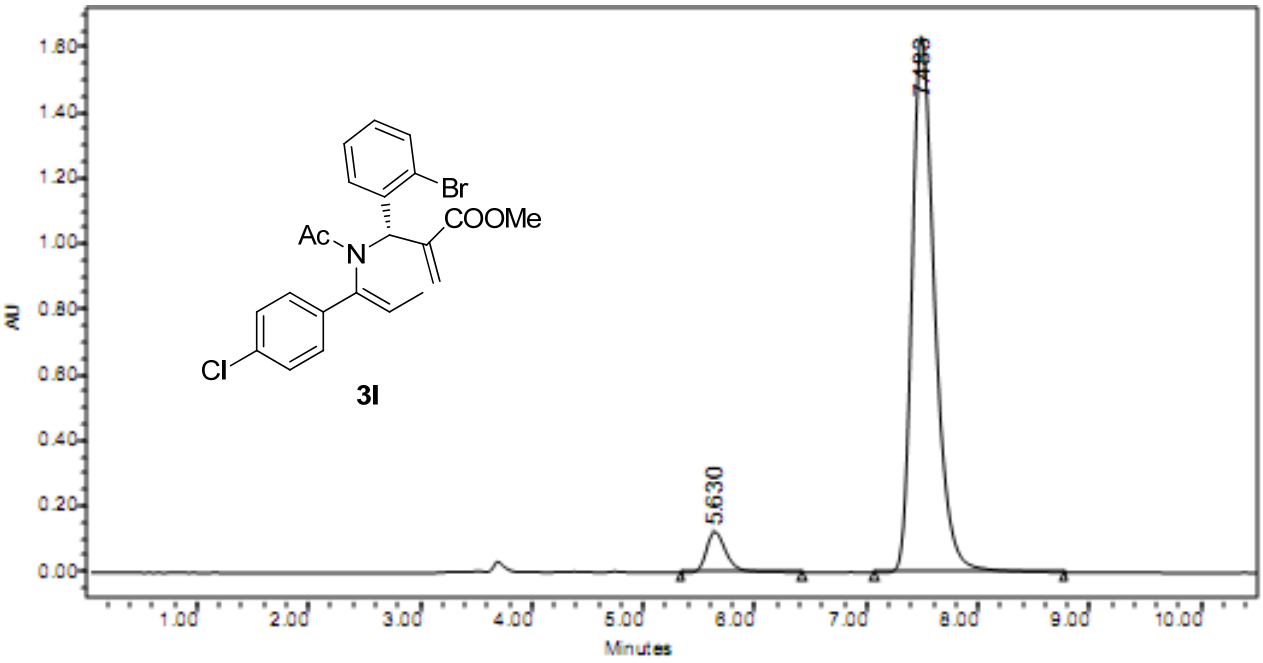
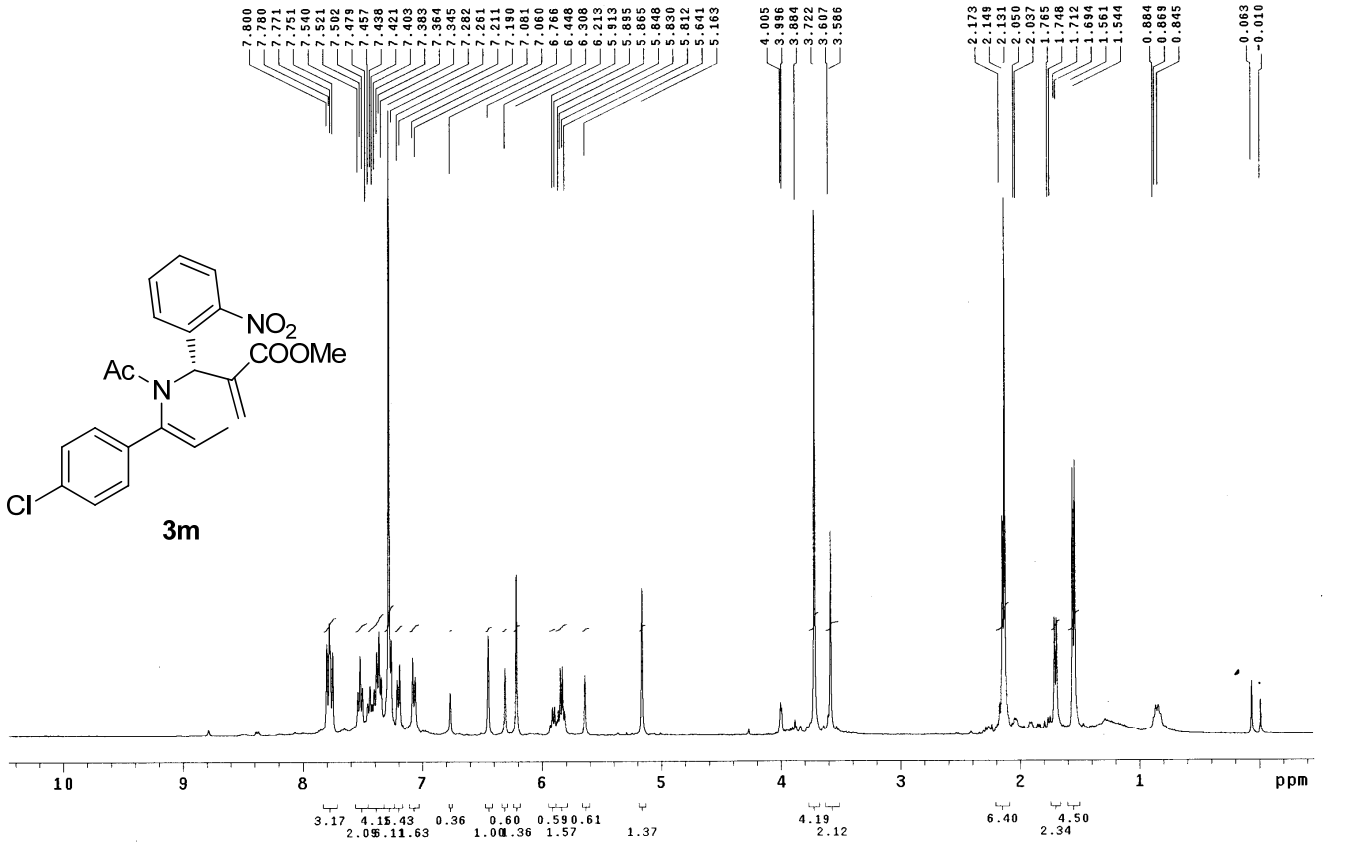
0
ppm



	Peak Name	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	Peak1	5.631	37975384	48.95	2460022	50.57
2	Peak2	7.481	39604065	51.05	2404676	49.43



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	5.630	1548188	6.27	124479	7.07
2	7.483	23154237	93.73	1634944	92.93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7.383
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7.345
7.282
7.261
7.211
7.190
7.081
7.060
6.766
6.448
6.308
6.213
5.913
5.895
5.865
5.848
5.830
5.812
5.641
5.163
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3.884
3.722
3.607
3.586
2.173
2.149
2.131
2.050
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1.544
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0.869
0.845
0.063
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COOMe
Ac
N
Cl
3m
10
9
8
7
6
5
4
3
2
1
ppm
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4.15
5.43
2.05
5.11
1.63
0.36
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0.59
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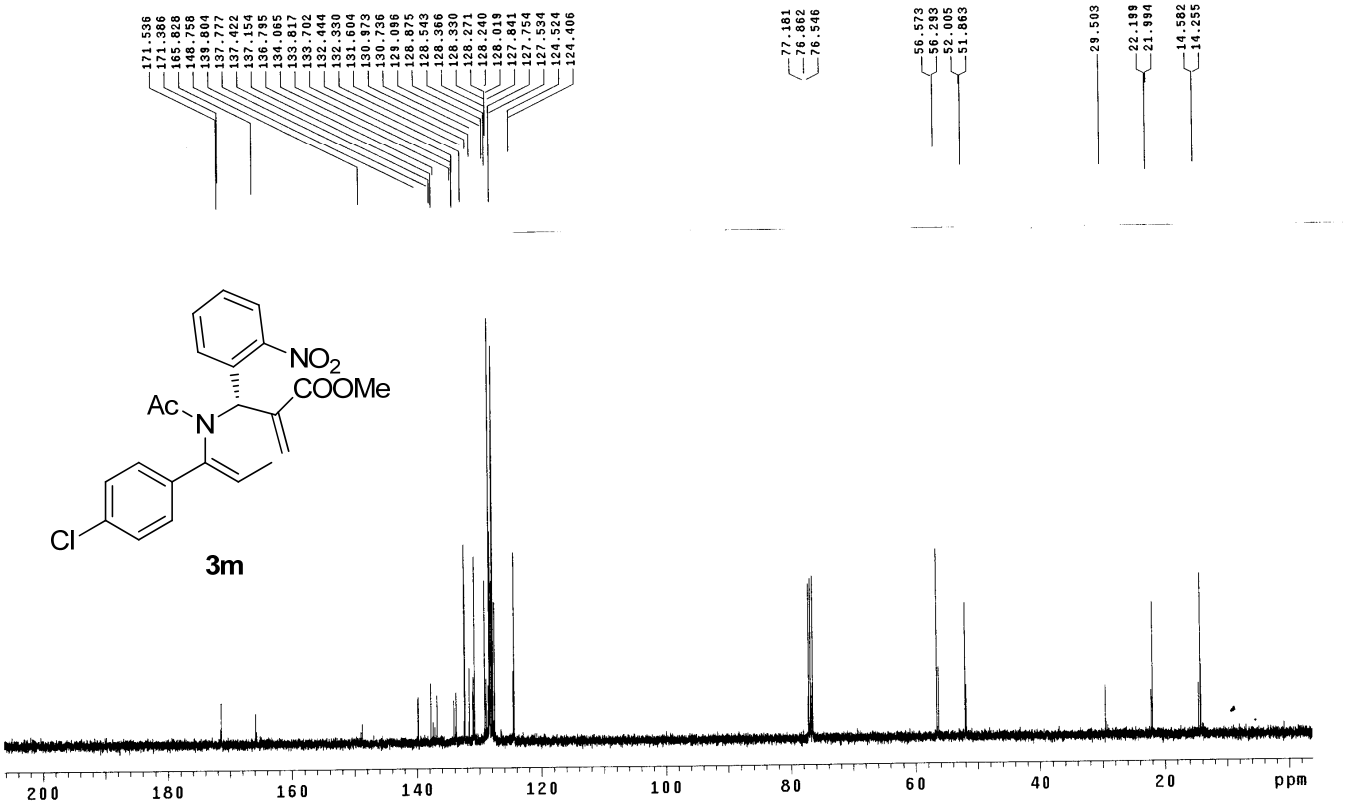
6.40
4.50
2.34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132.330
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128.330
128.271
128.240
128.019
127.841
127.754
127.534
124.524
124.406
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76.862
76.546
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52.005
51.863
29.503
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21.994
14.582
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COOMe
Ac
N
Cl
3m
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140
120
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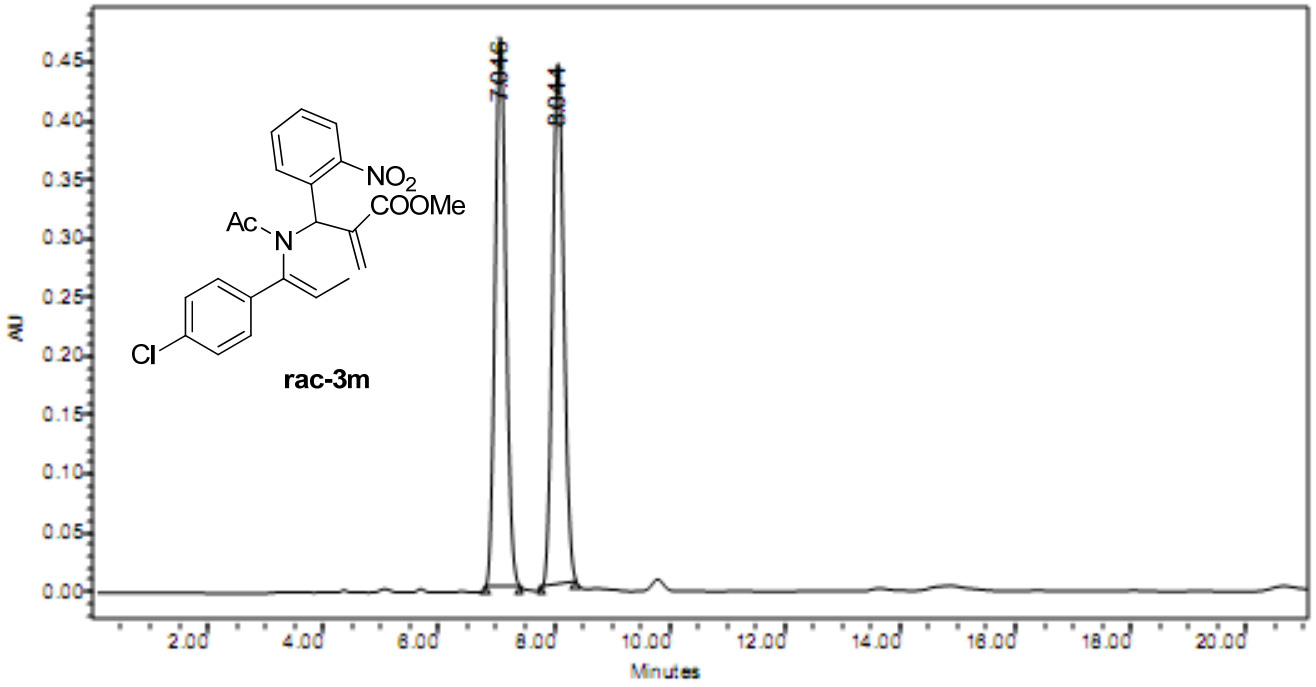
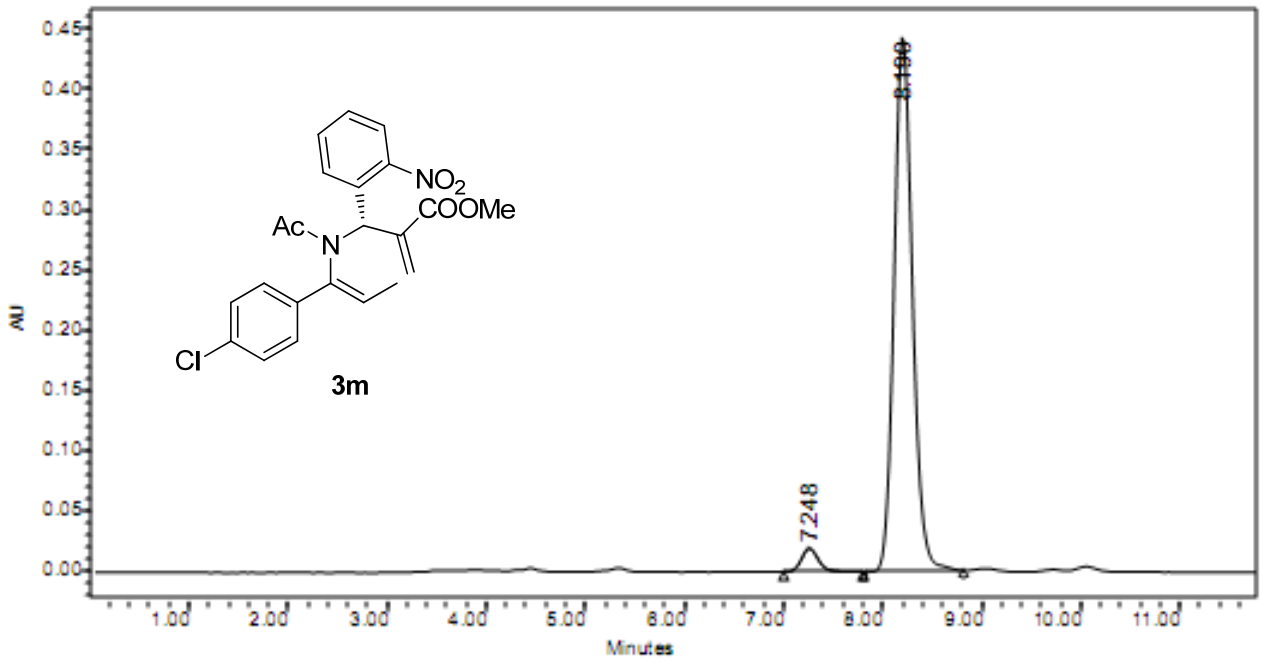
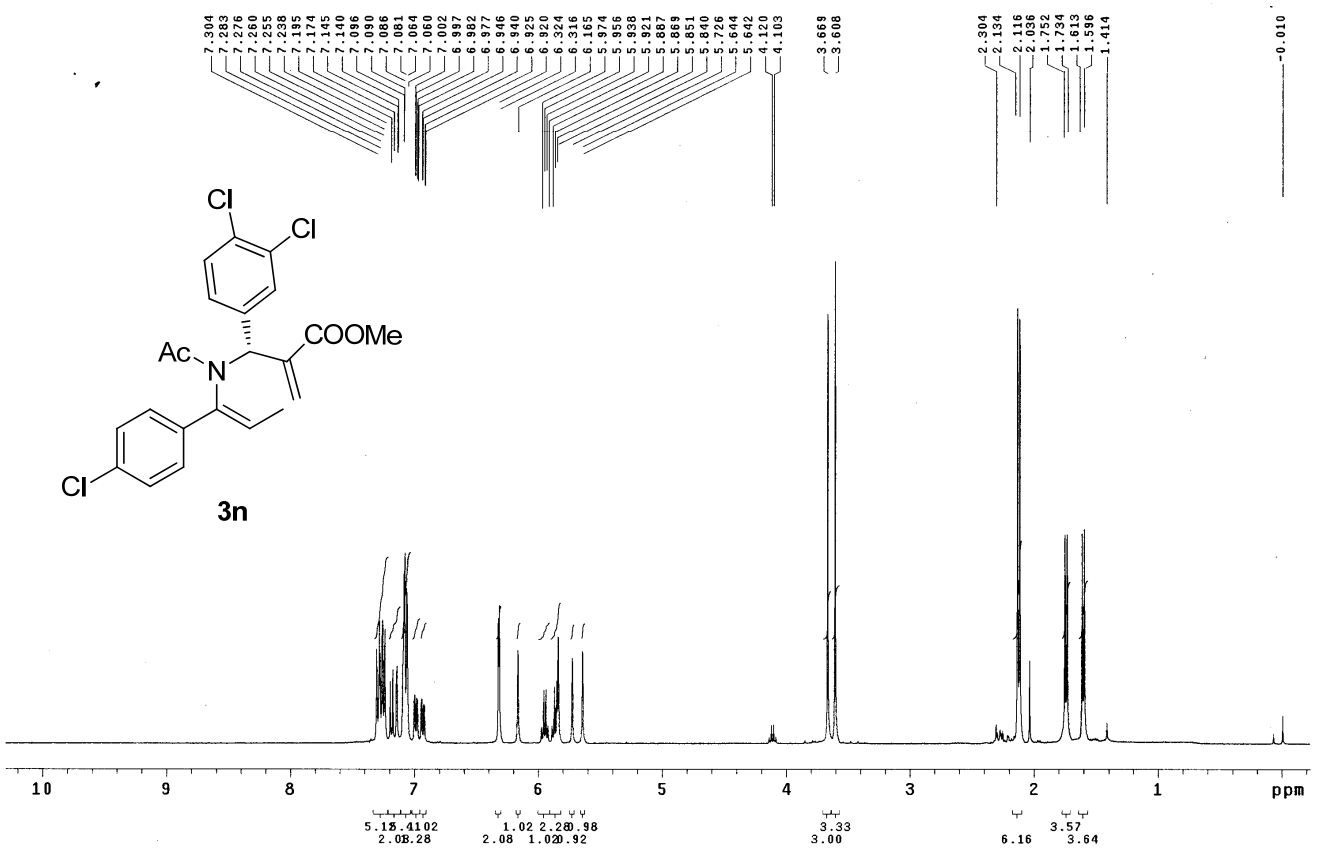
60
40
20
ppm



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	7.046	6081811	50.23	467639	51.33
2	8.044	6026445	49.77	443369	48.67



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	7.248	236824	3.97	19567	4.22
2	8.190	5735814	96.03	443935	95.78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7.064
7.060
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6.977
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5.840
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1.613
1.596
1.414
-0.010
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COOMe
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Cl
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8
7
6
5
4
3
2
1
ppm
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5.41
02
2.08
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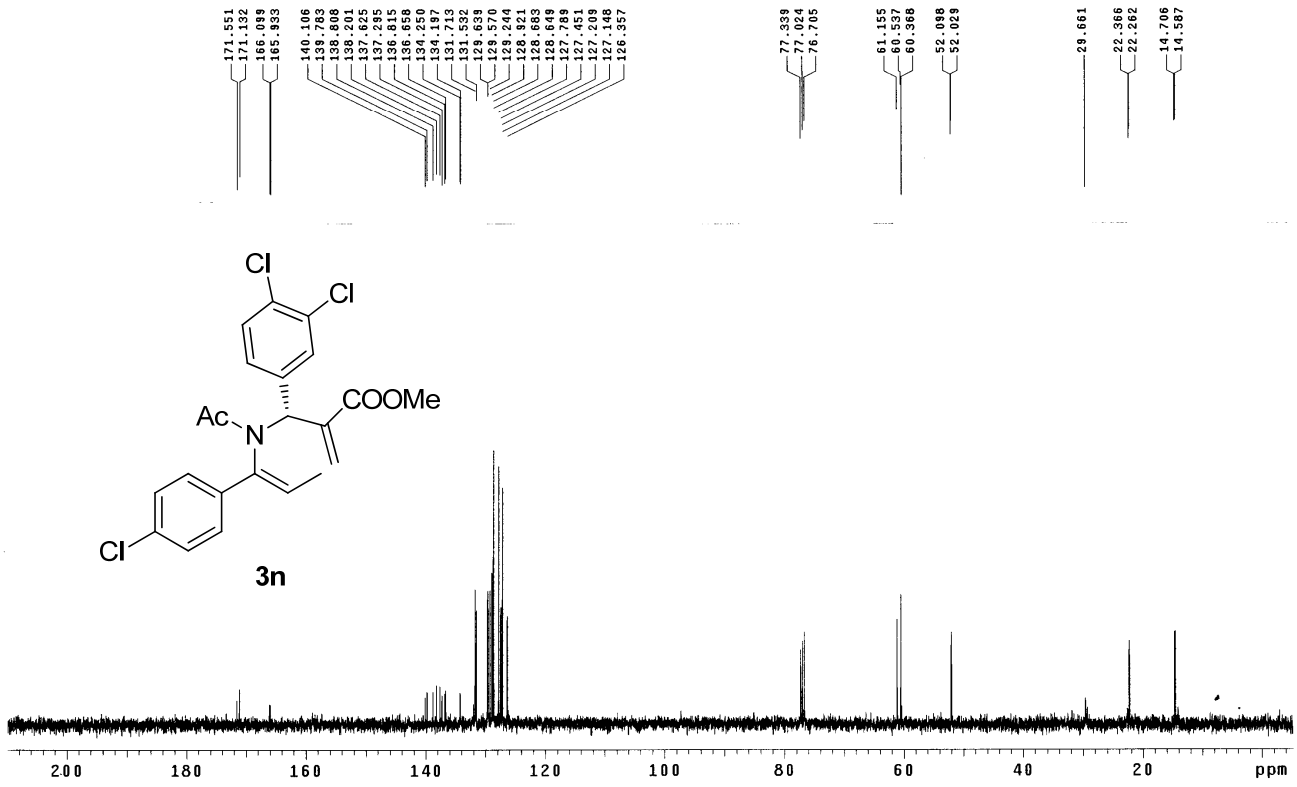
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77.024
76.705
61.155
60.537
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22.366
22.262
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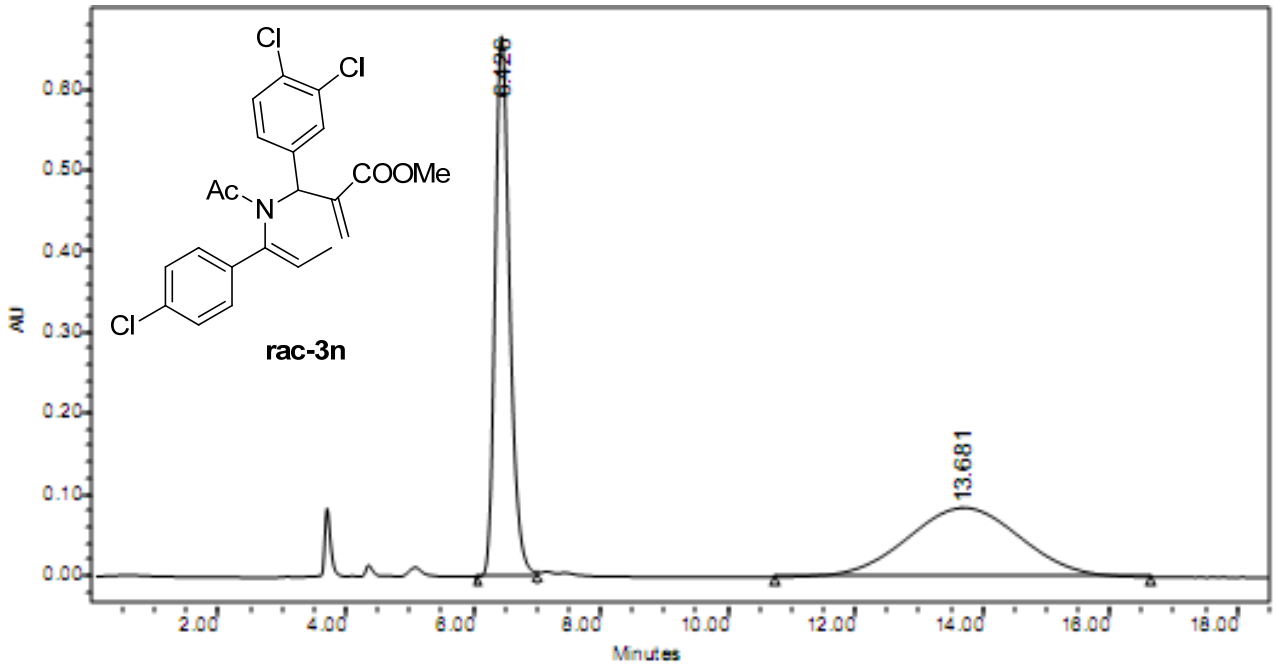
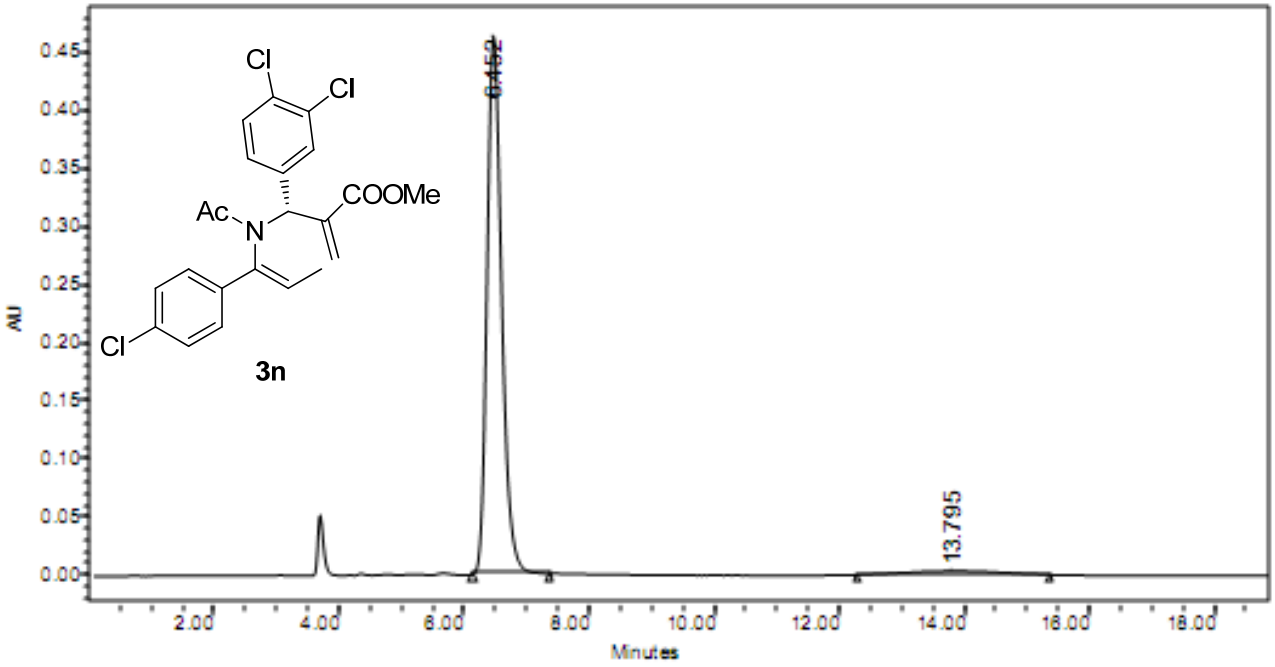
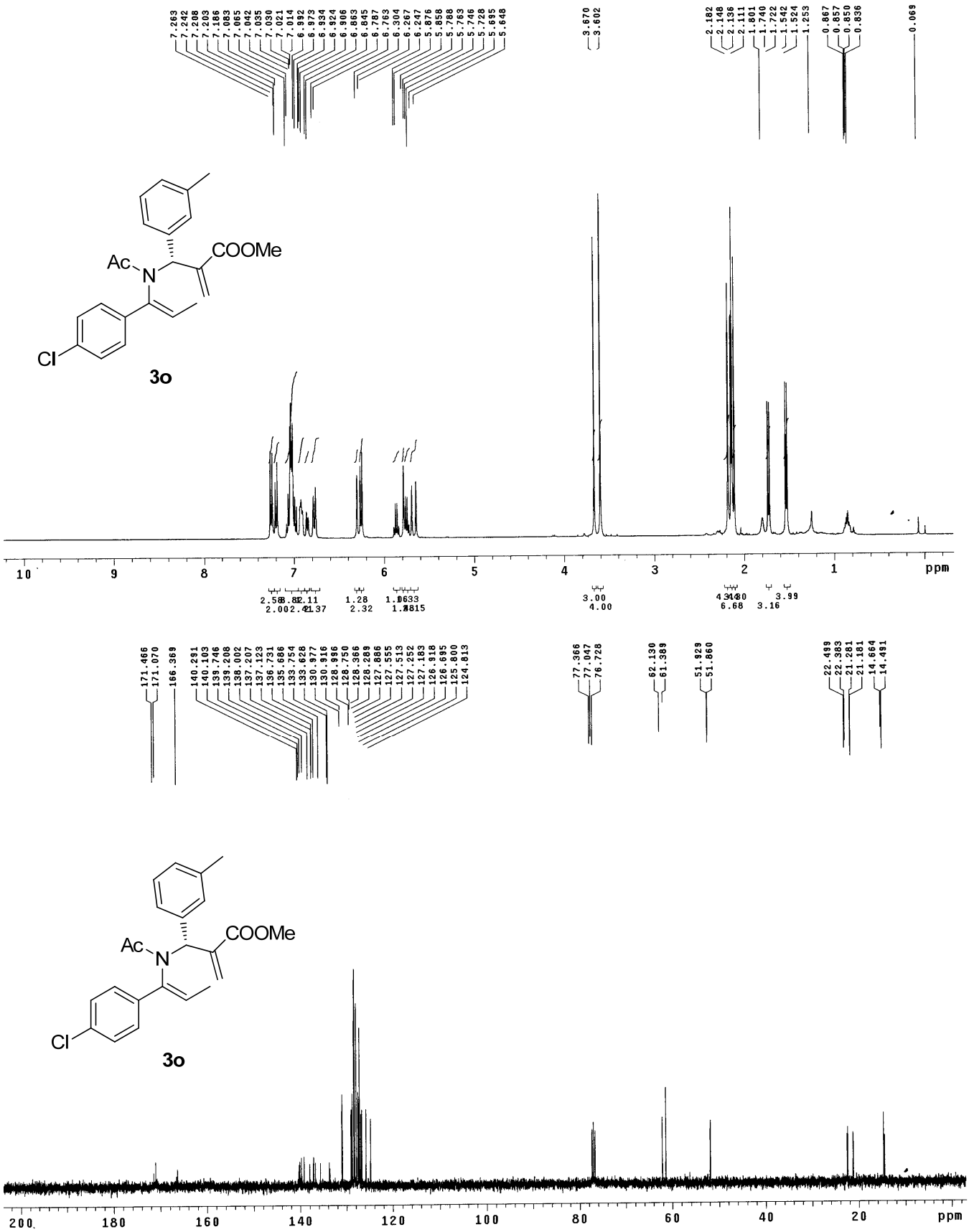
20
ppm



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	6.426	10687467	49.91	668039	88.64
2	13.681	10724424	50.09	85601	11.36



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	6.452	7514491	95.69	463600	99.30
2	13.795	338760	4.31	3265	0.70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7.030
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7.014
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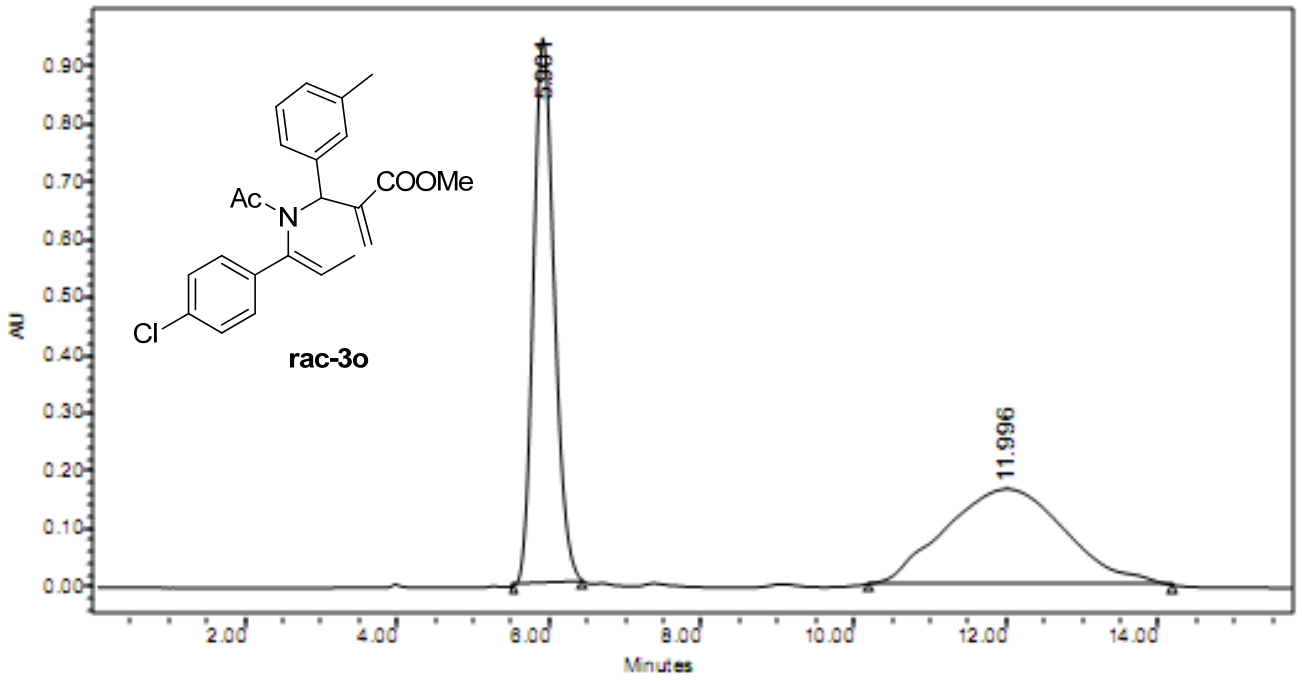
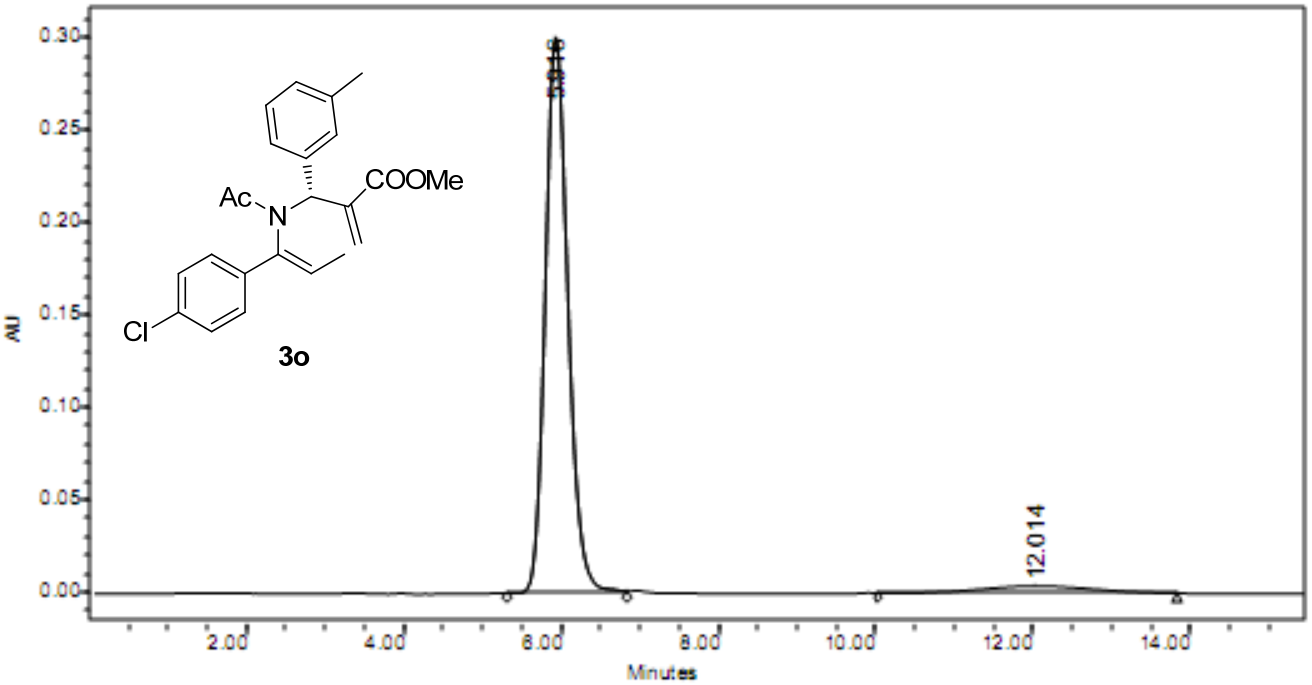
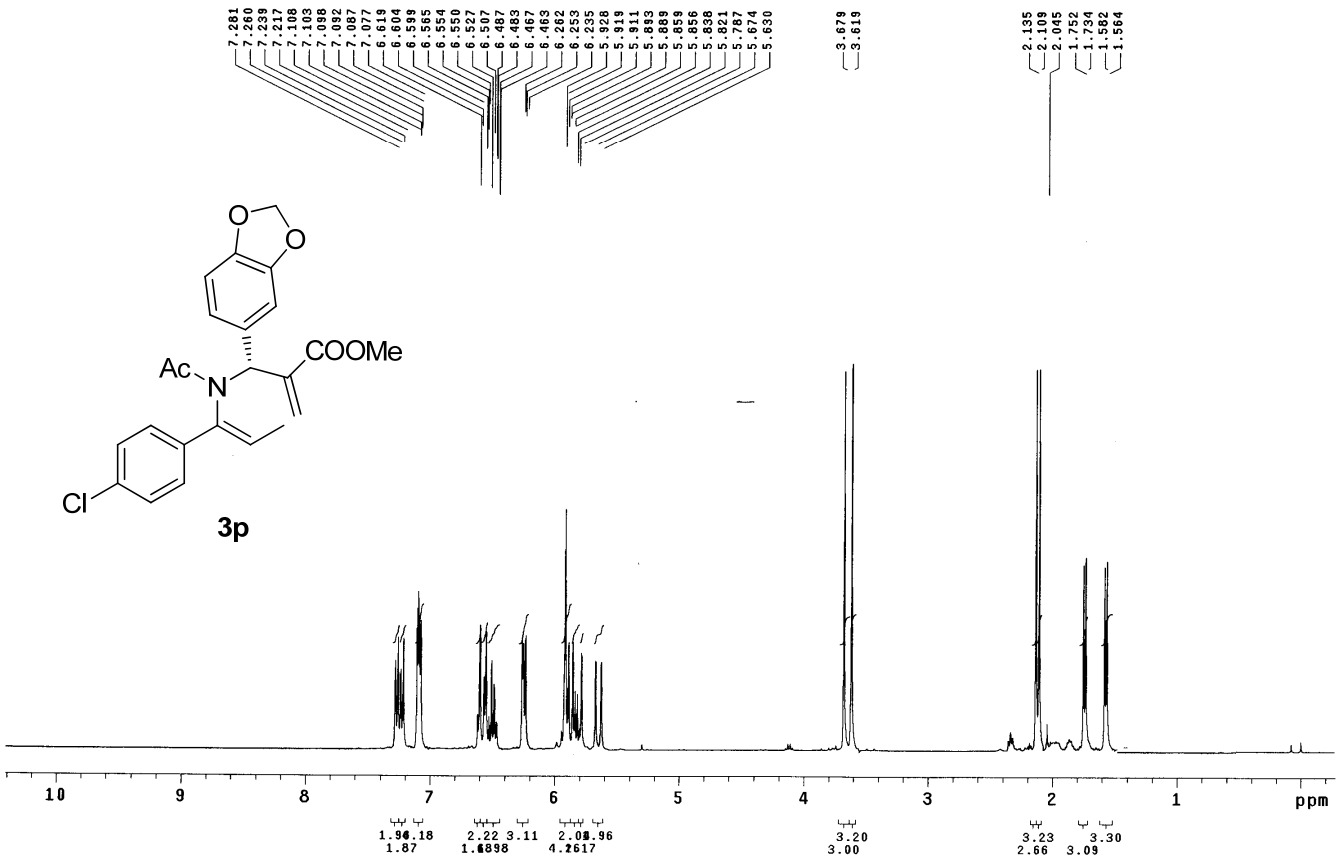


	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	5.901	17930111	49.59	943638	85.11
2	11.996	18224114	50.41	165124	14.89



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	5.916	6042501	93.46	300658	98.67
2	12.014	422939	6.54	4042	1.33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4
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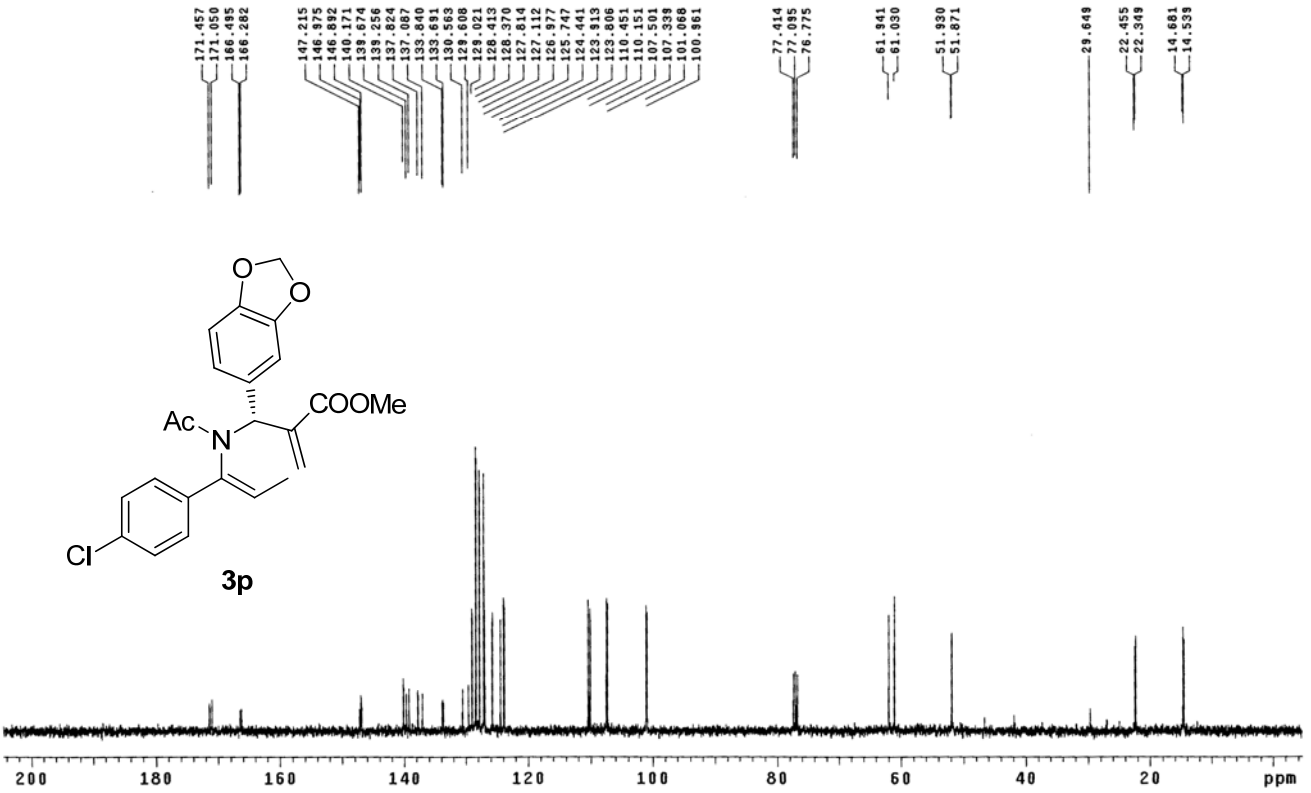
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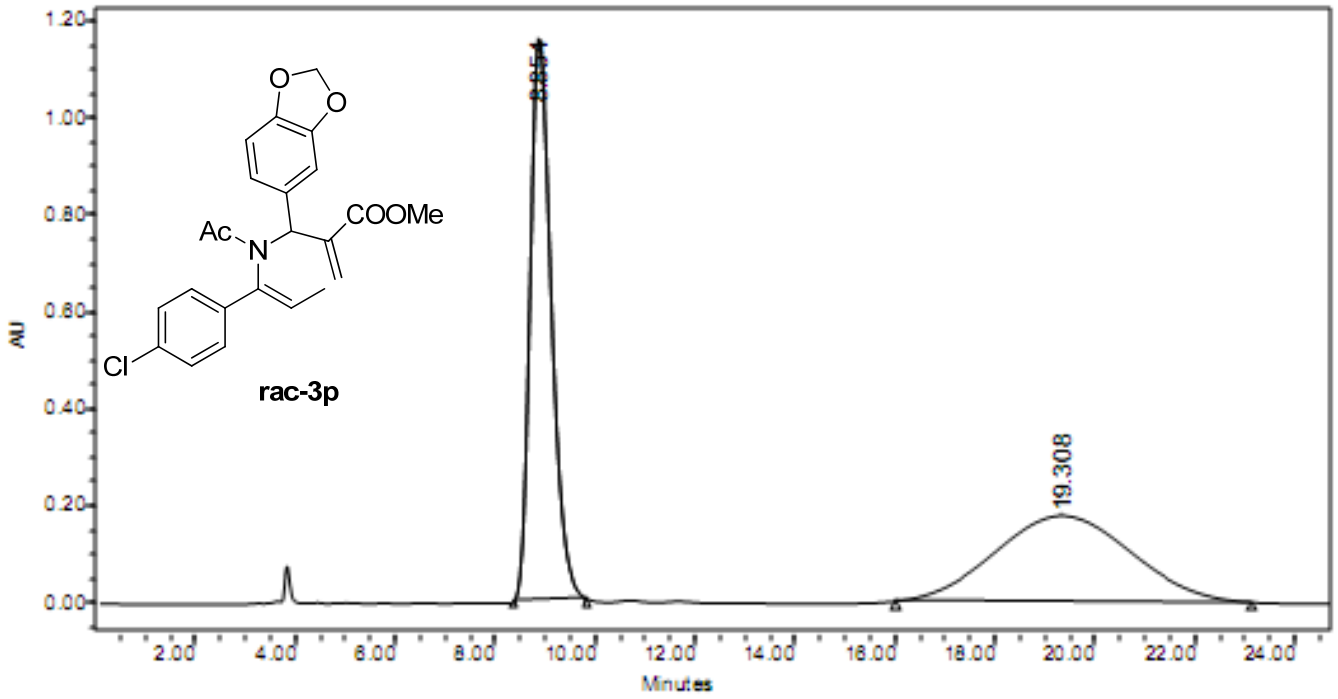
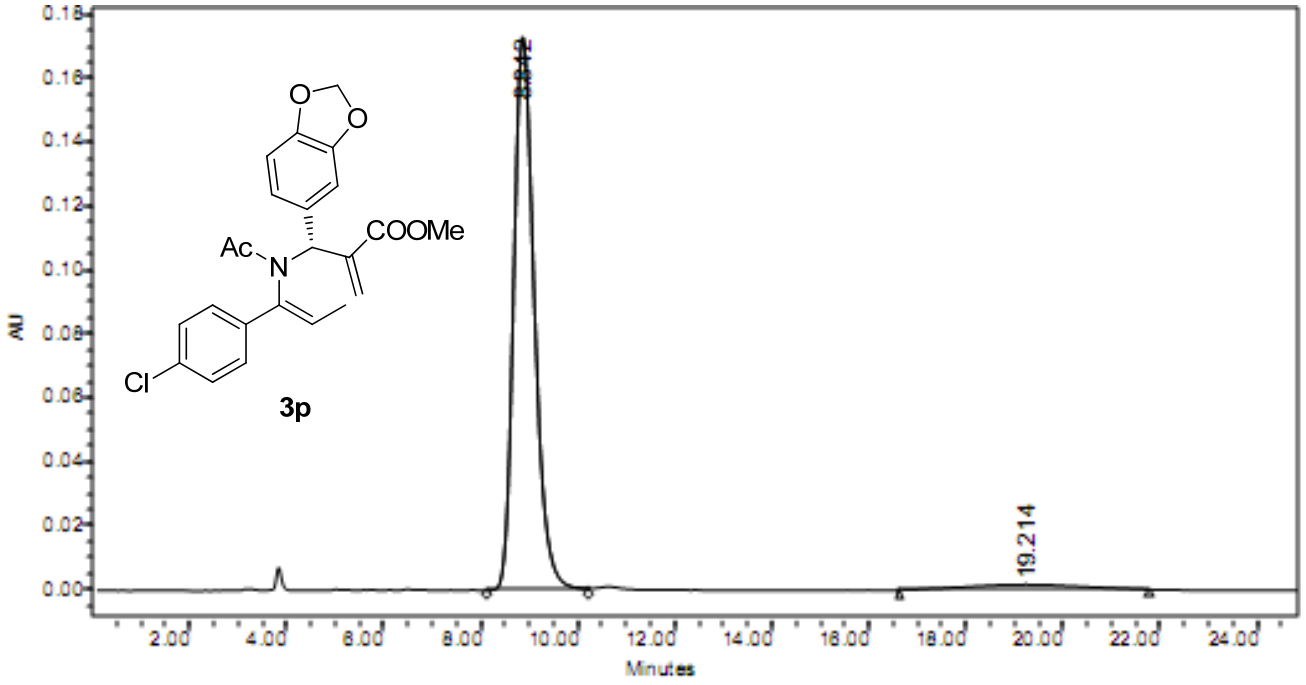
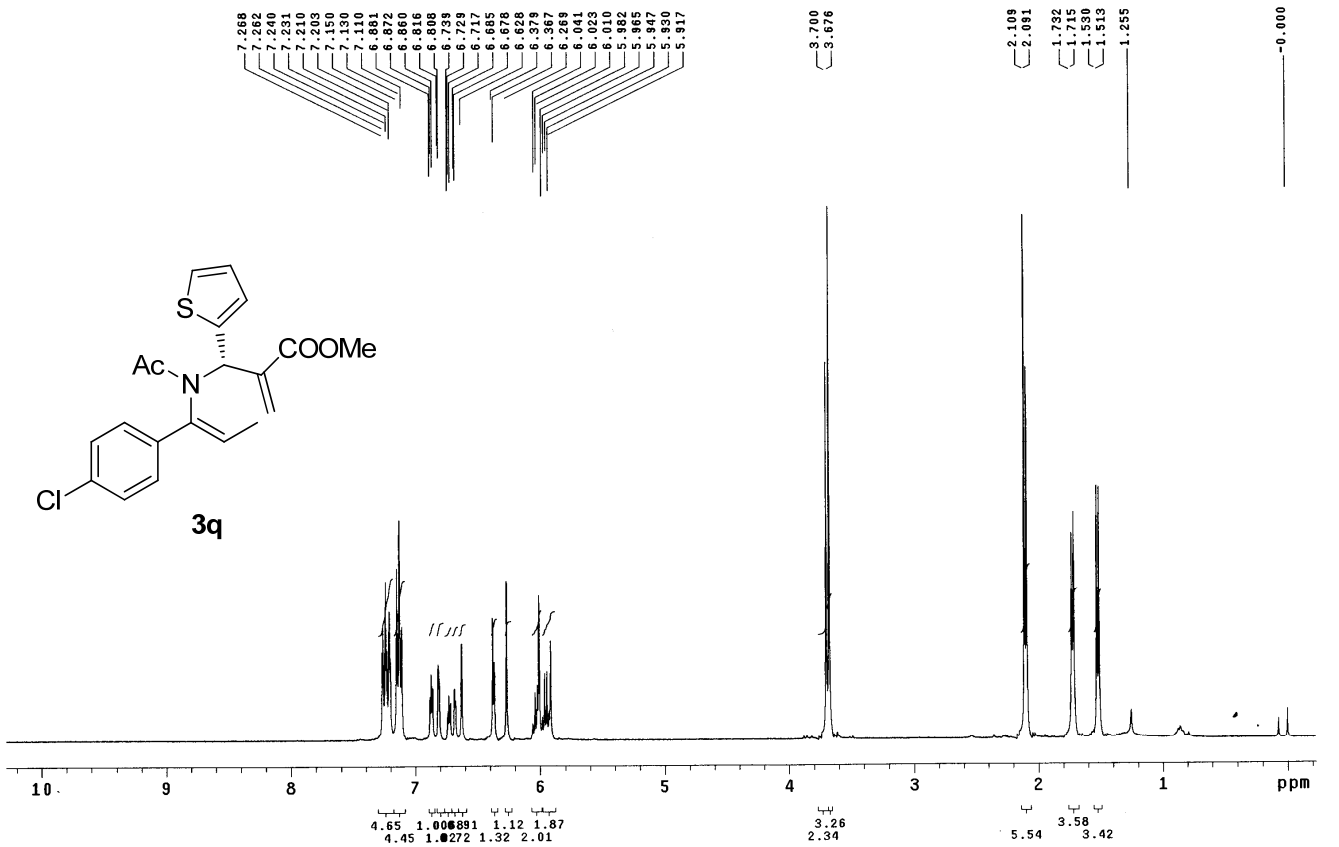


	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	8.854	34211022	50.37	1157552	86.72
2	19.308	33710088	49.63	177269	13.28



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	8.842	5120365	94.78	173115	99.02
2	19.214	282267	5.22	1719	0.98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N
COOMe
Cl
3q
10
9
8
7
6
5
4
3
2
1
ppm
4.65
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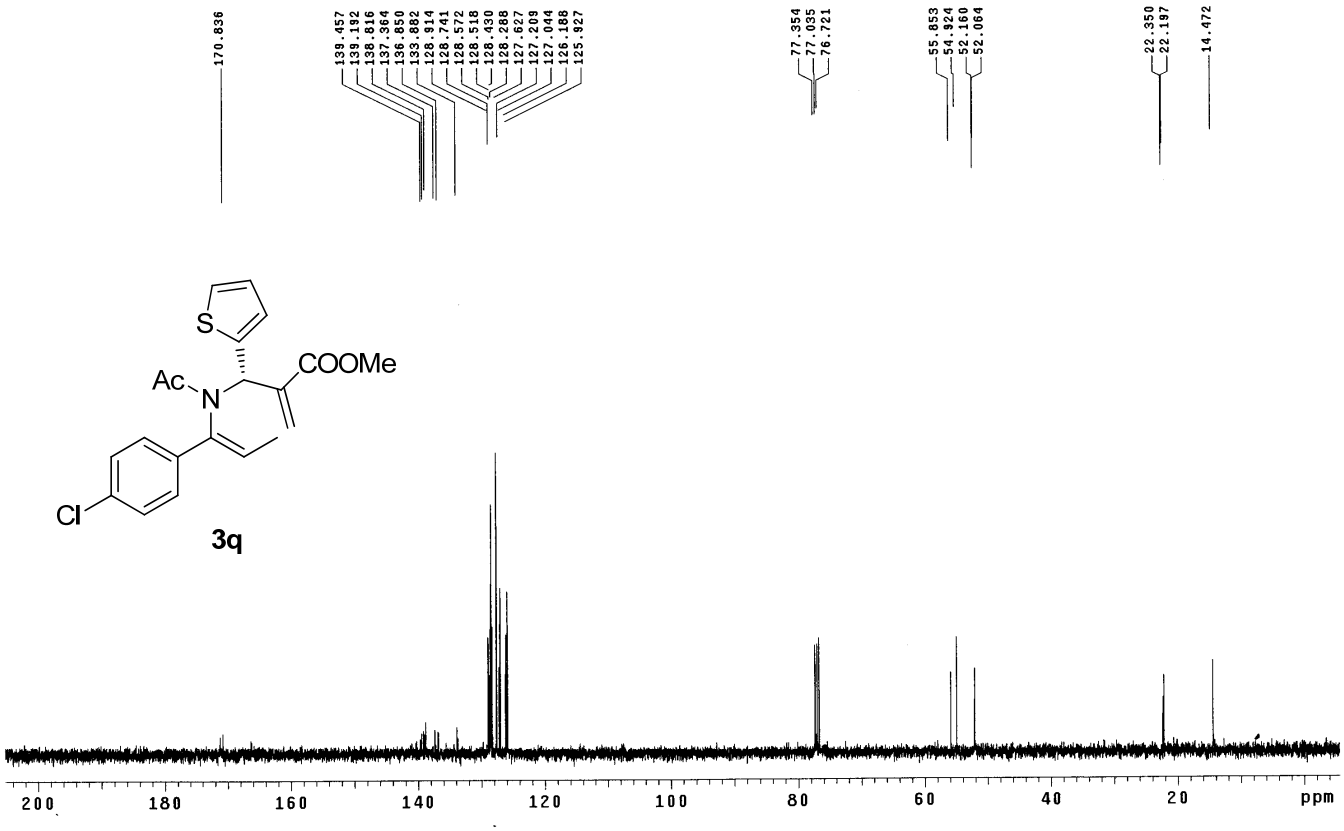
1.0
1.12
1.32
1.87
2.01
3.26
2.34
5.54
3.58
3.42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128.572
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128.288
127.627
127.209
127.044
126.188
125.927
77.354
77.035
76.721
55.853
54.924
52.160
52.064
22.350
22.197
14.472
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Ac
N
COOMe
Cl
3q
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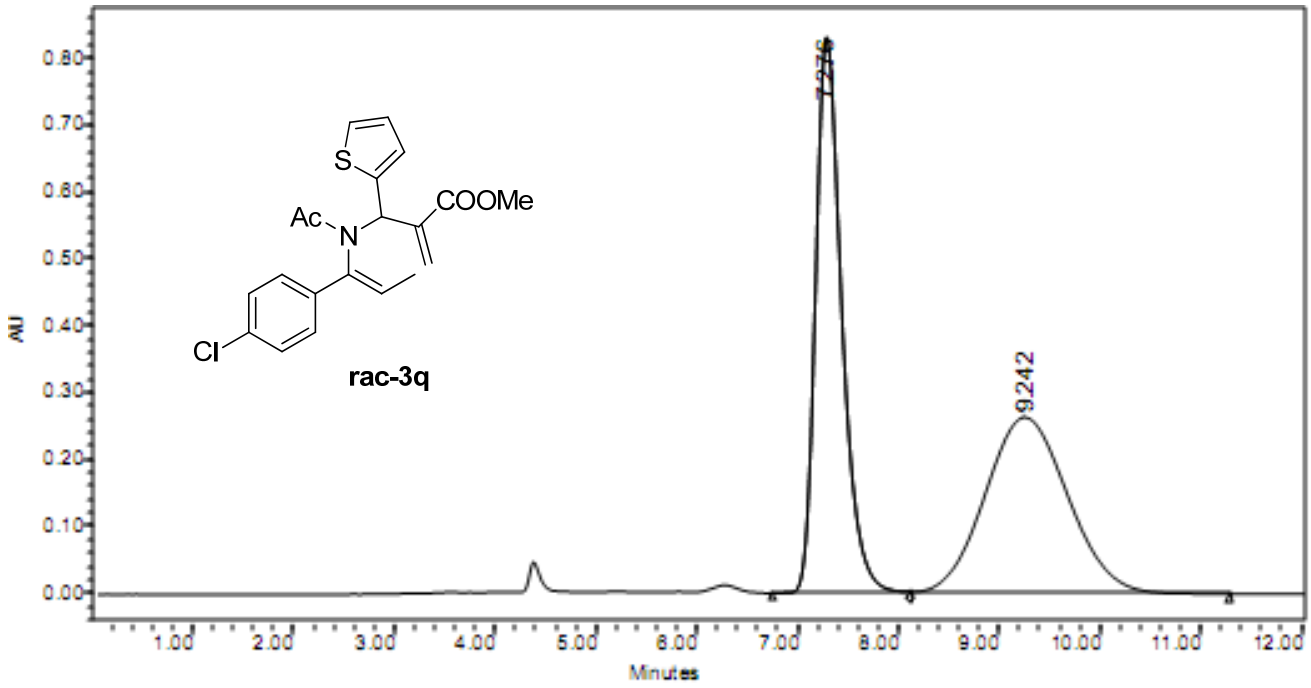
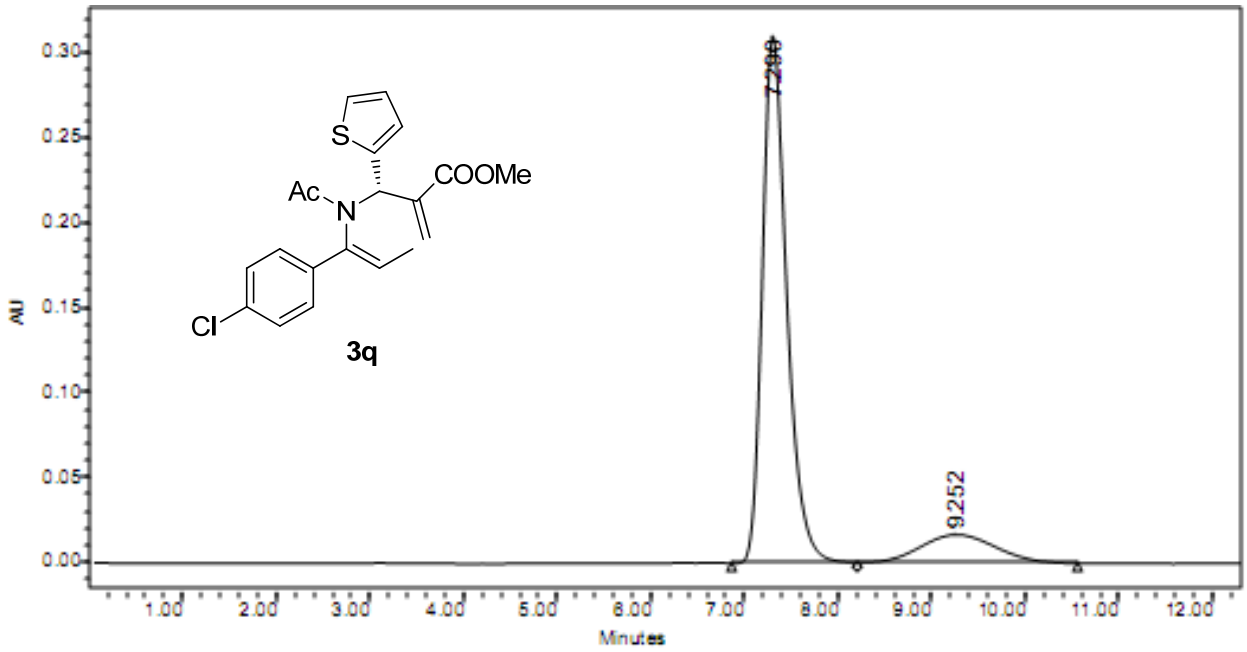
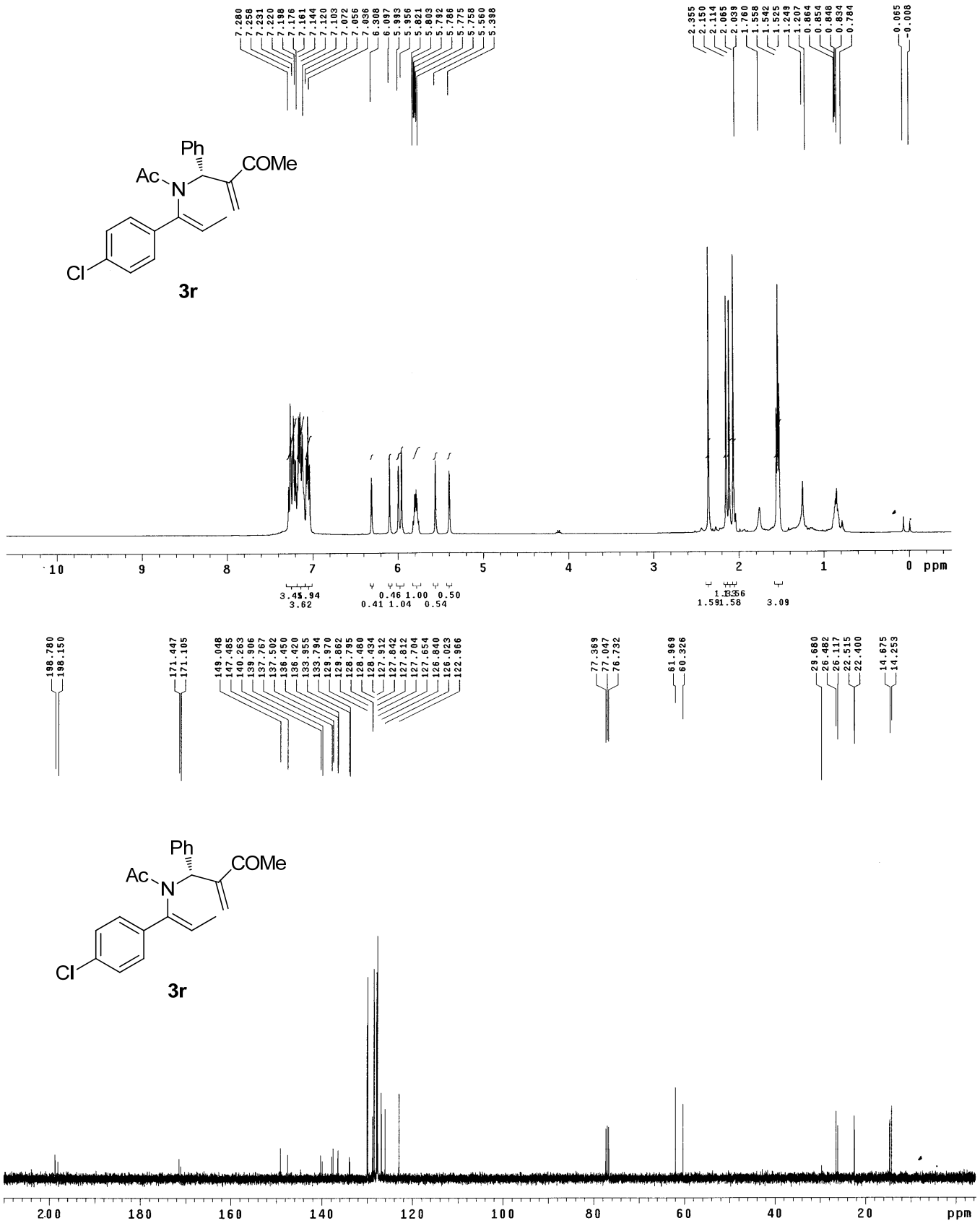
140
120
100
80
60
40
20
ppm



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	7.276	15094153	49.94	832711	75.94
2	9.242	15128701	50.06	263774	24.06



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	7.290	5638198	85.38	311088	94.85
2	9.252	965720	14.62	16887	5.15


Ph
Ac
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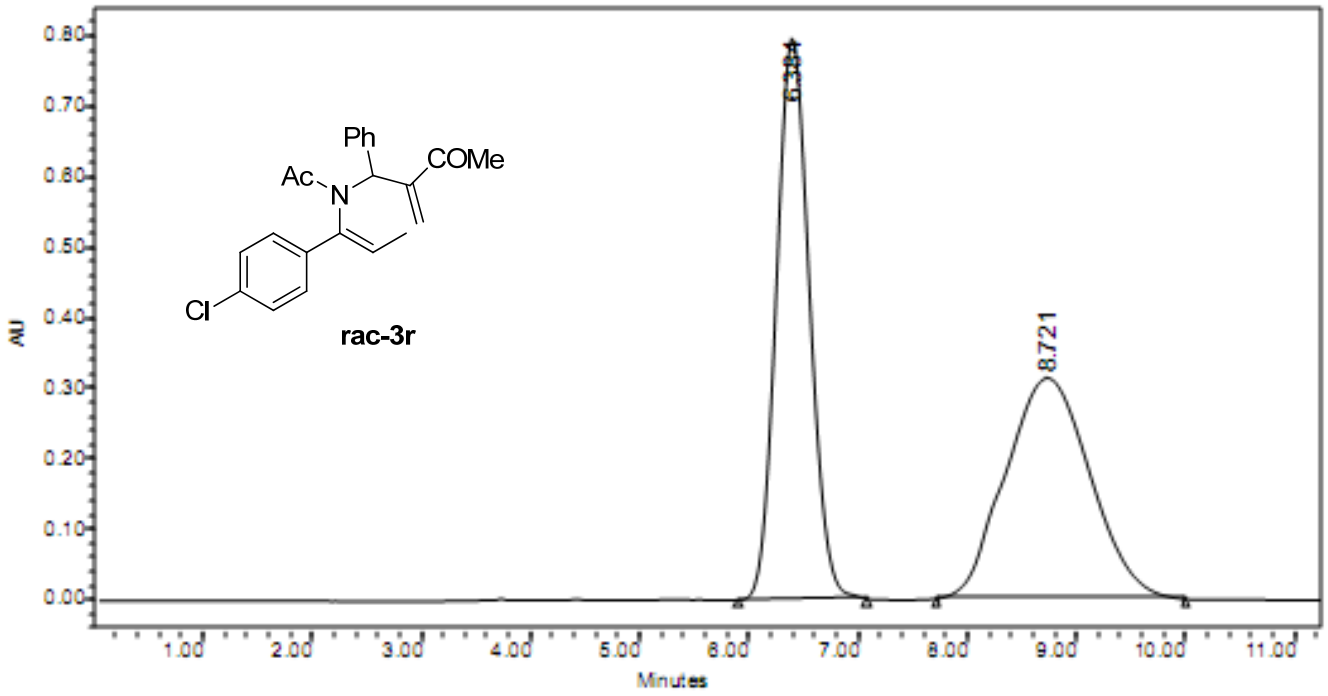
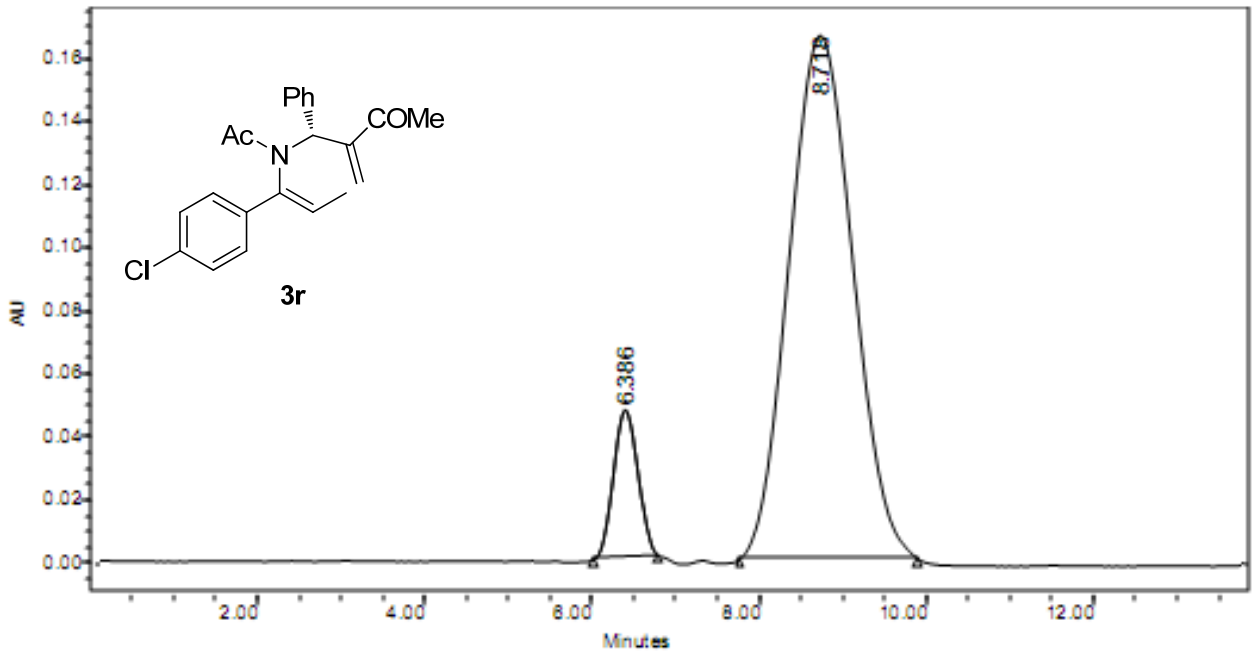
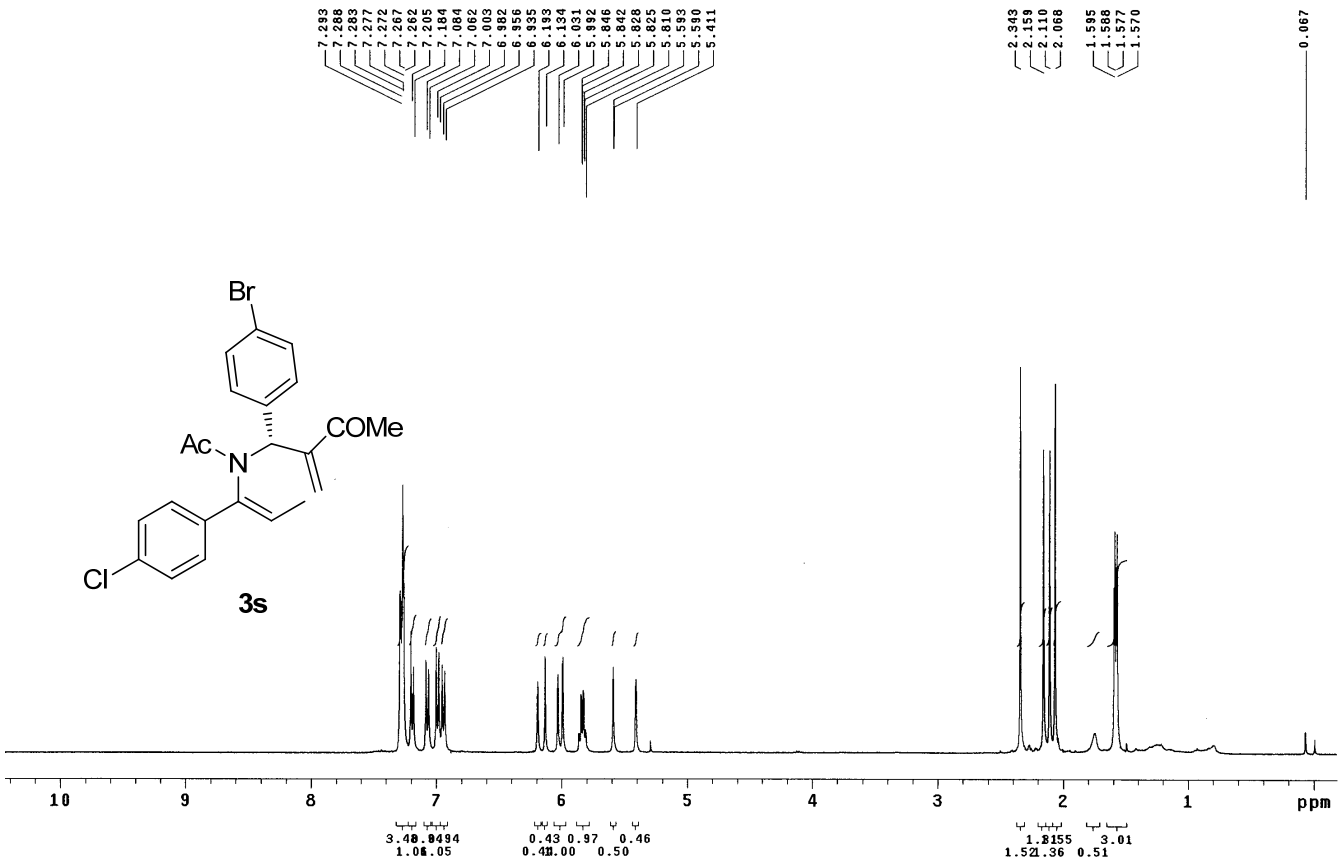
COMe
Cl
3r
ppm
Ph
Ac
N
COMe
Cl
3r
ppm



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	6.384	16813683	49.85	796031	71.77
2	8.721	16913321	50.15	313170	28.23



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	6.386	954416	9.77	46745	21.98
2	8.718	8809722	90.23	165962	78.02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7.062
7.003
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6.956
6.935
6.193
6.134
6.031
5.992
5.846
5.842
5.828
5.825
5.810
5.593
5.590
5.411
2.343
2.159
2.110
2.068
1.595
1.588
1.577
1.570
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COMe
Ac
N
Cl
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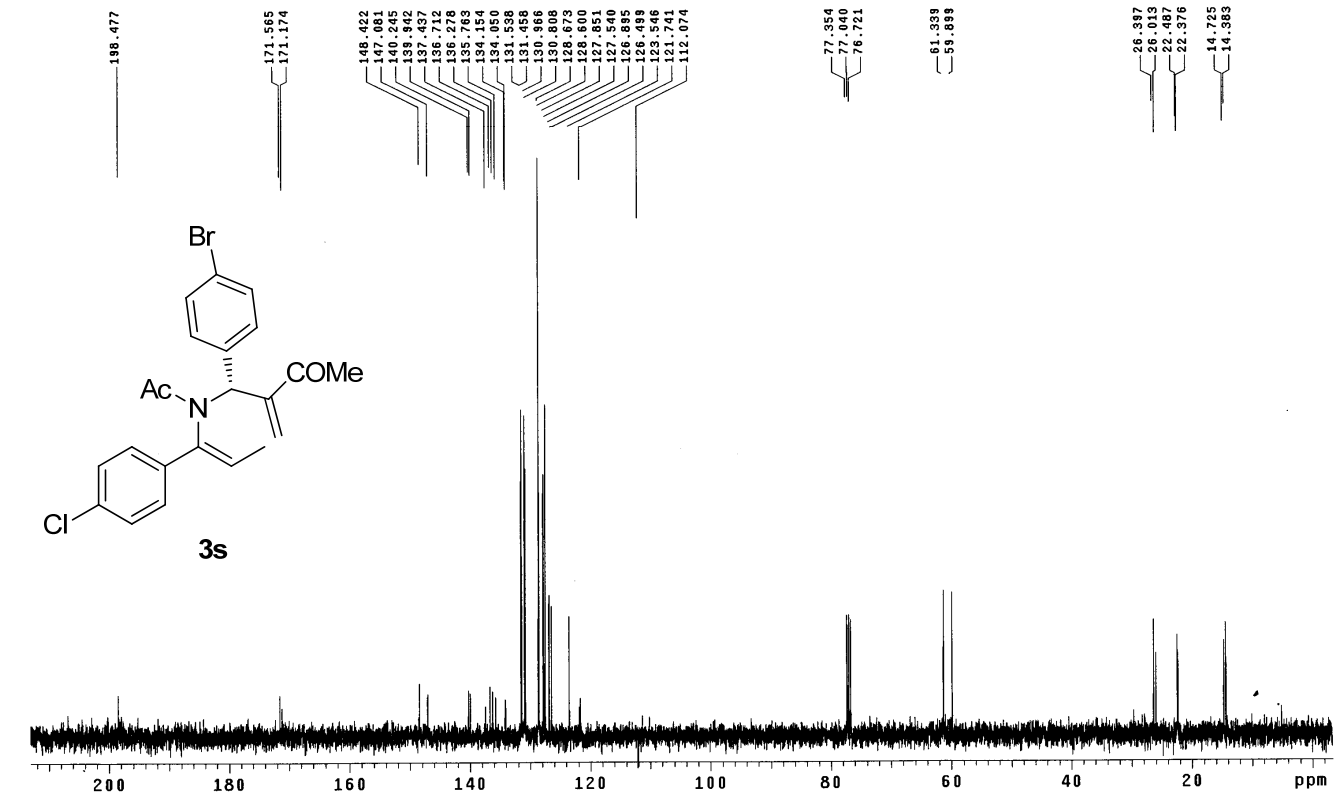
7
6
5
4
3
2
1
ppm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140.245
139.842
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135.763
134.454
134.050
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131.458
130.966
130.808
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128.600
127.851
127.540
126.895
126.499
123.546
121.741
112.074
77.354
77.040
76.721
61.339
59.899
26.397
26.013
22.487
22.376
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COMe
Ac
N
Cl
3s
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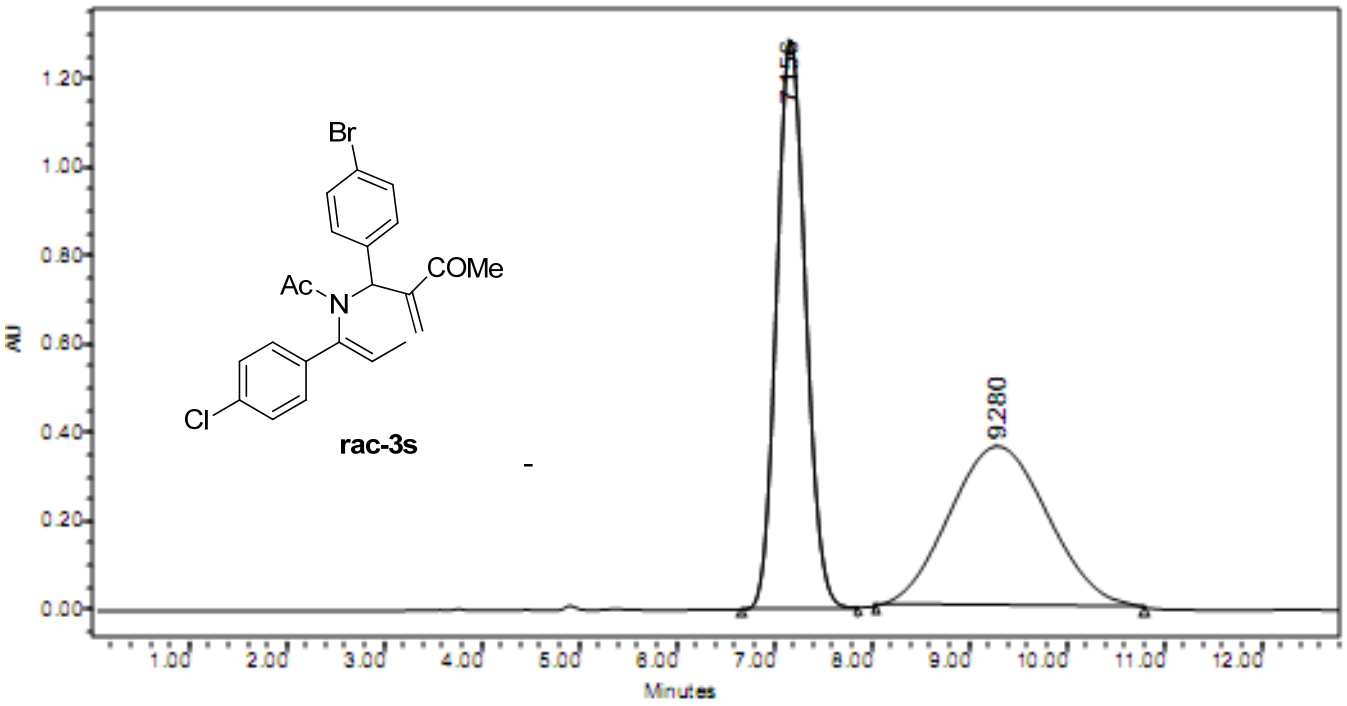
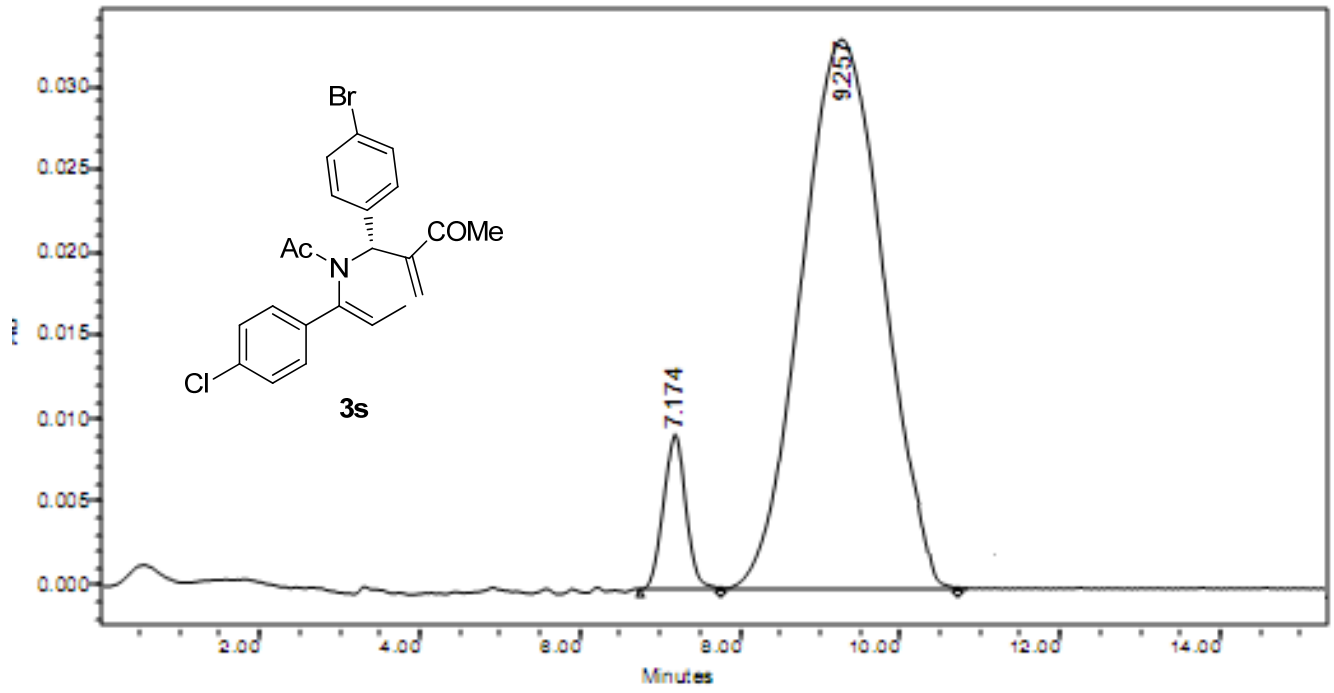
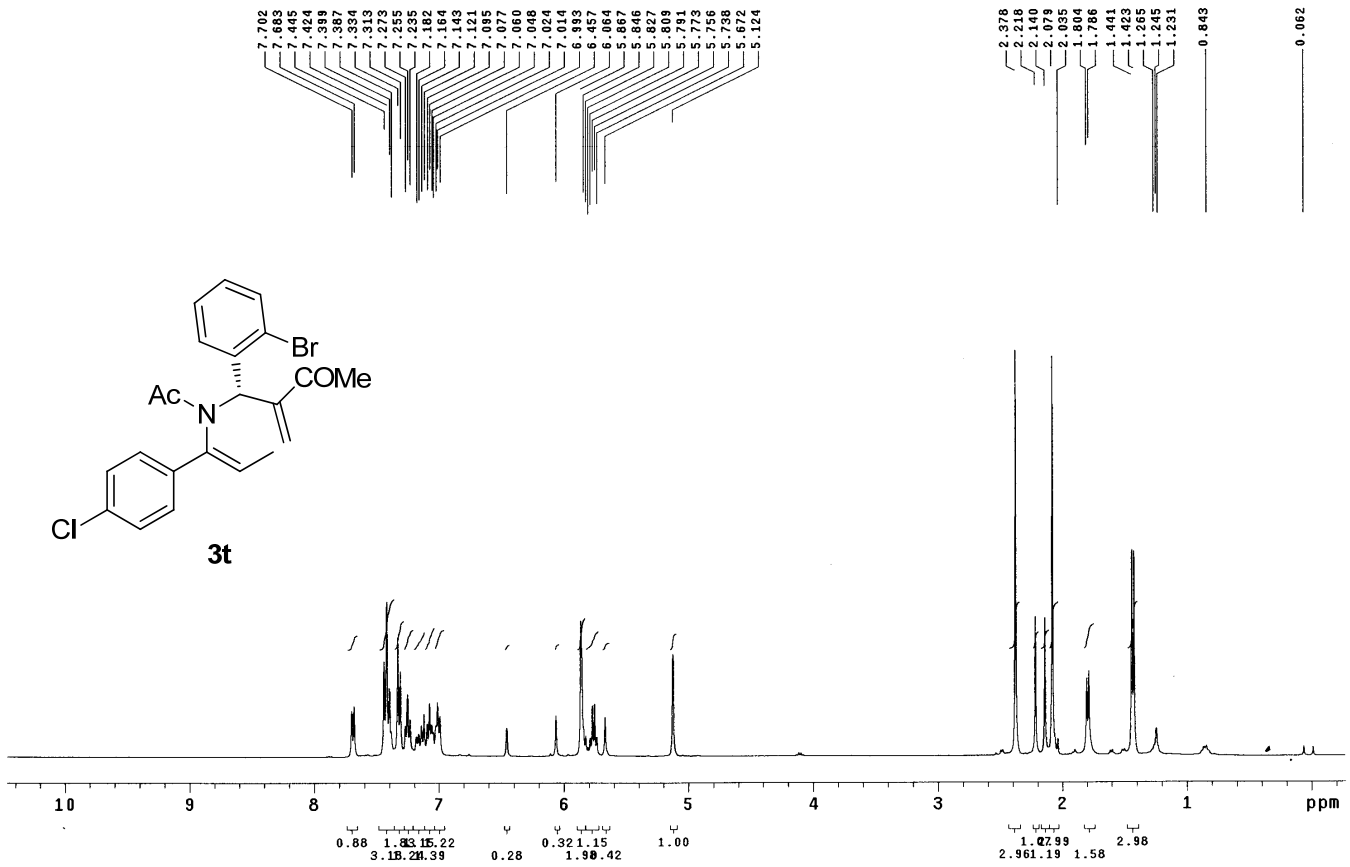
140
120
100
80
60
40
20
ppm



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	7.156	26758879	51.09	1289703	78.07
2	9.280	25614069	48.91	362363	21.93



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	7.174	181974	7.03	9309	21.94
2	9.257	2405836	92.97	33123	78.06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7.313
7.273
7.255
7.235
7.182
7.164
7.143
7.121
7.095
7.077
7.060
7.048
7.024
7.014
6.983
6.457
6.064
5.867
5.846
5.827
5.809
5.791
5.773
5.756
5.738
5.672
5.124
2.378
2.218
2.140
2.079
2.035
1.804
1.786
1.441
1.423
1.265
1.245
1.231
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0.062
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COMe
Ac
N
Cl
3t
10
9
8
7
6
5
4
3
2
1
ppm
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1.83
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5.22
3.15
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0.32
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1.00
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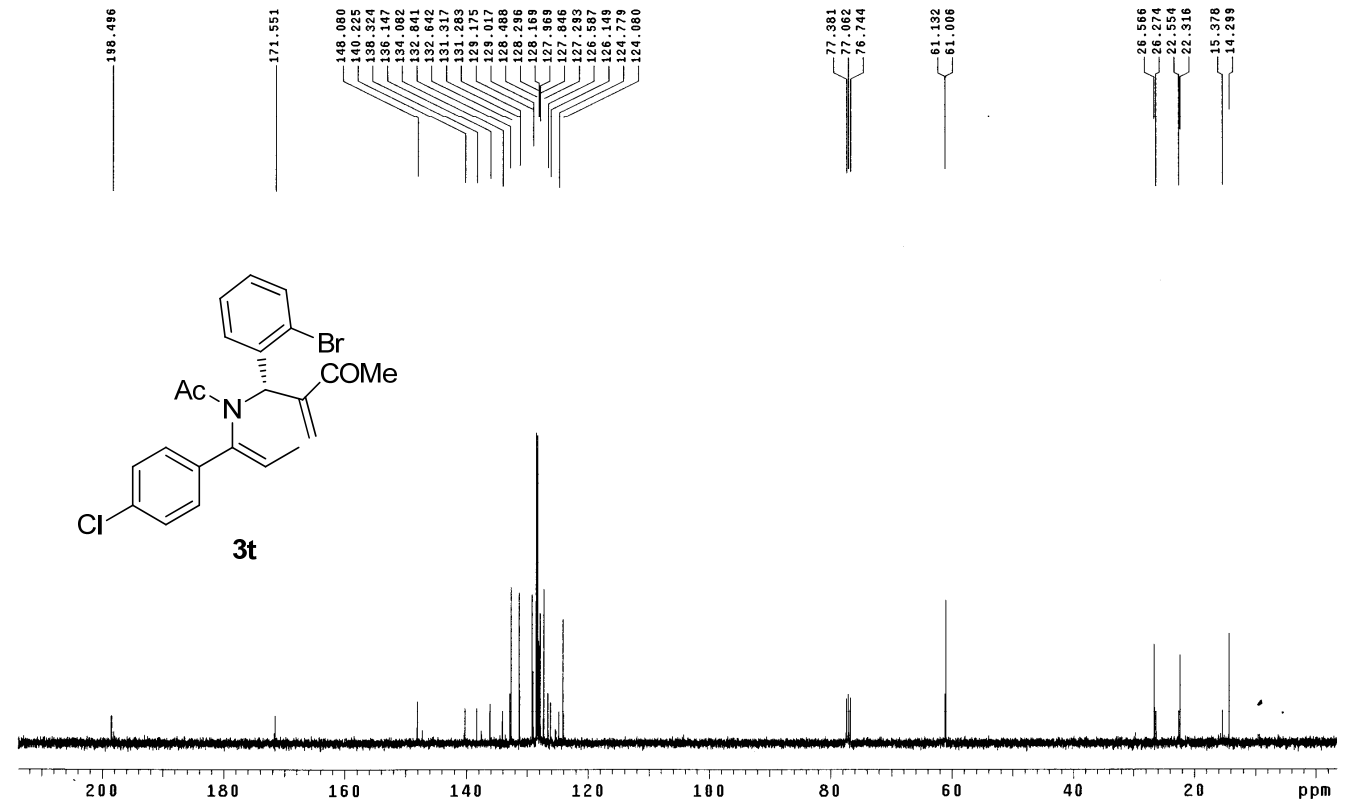
1.19
1.58
2.98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128.296
128.169
127.969
127.846
127.293
126.587
126.149
124.779
124.080
77.381
77.062
76.744
61.132
61.006
26.566
26.274
22.554
22.316
15.378
14.299
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COMe
Ac
N
Cl
3t
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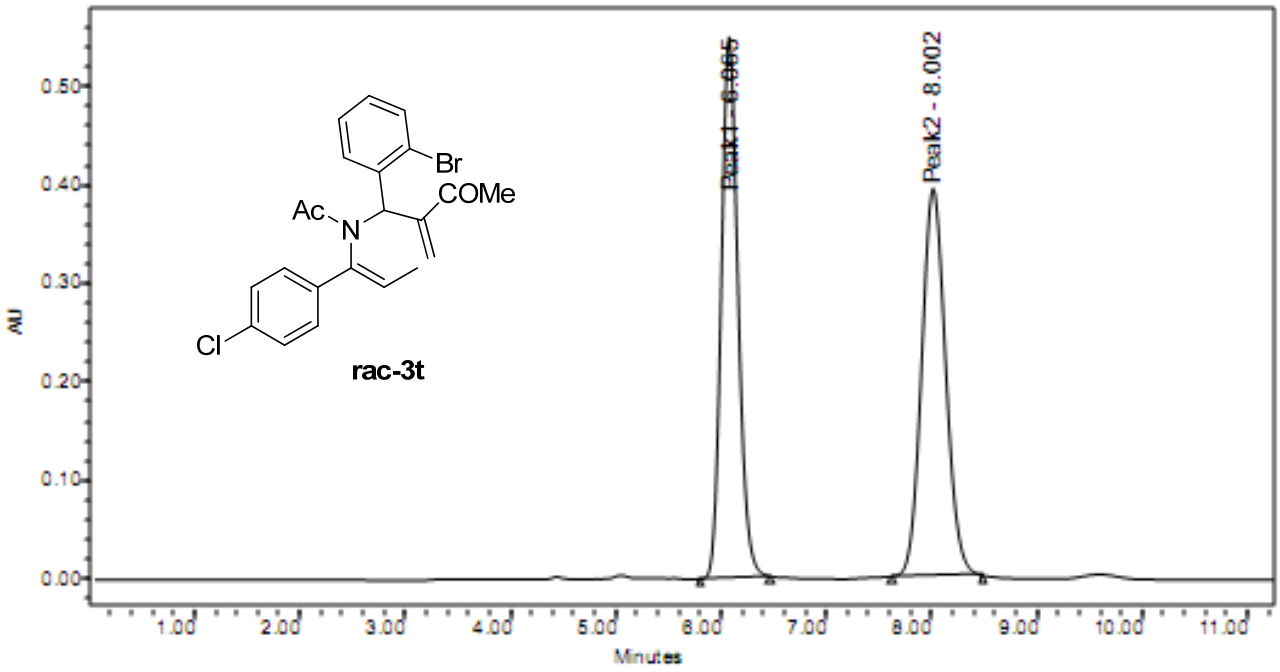
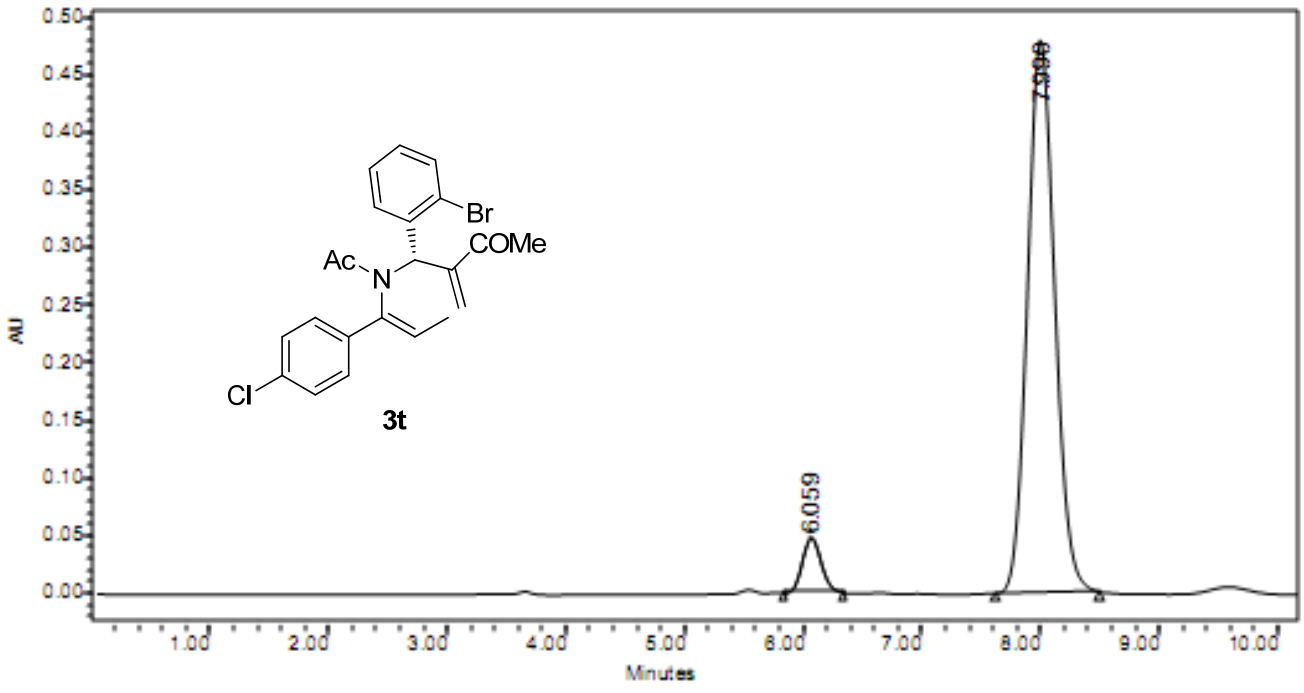
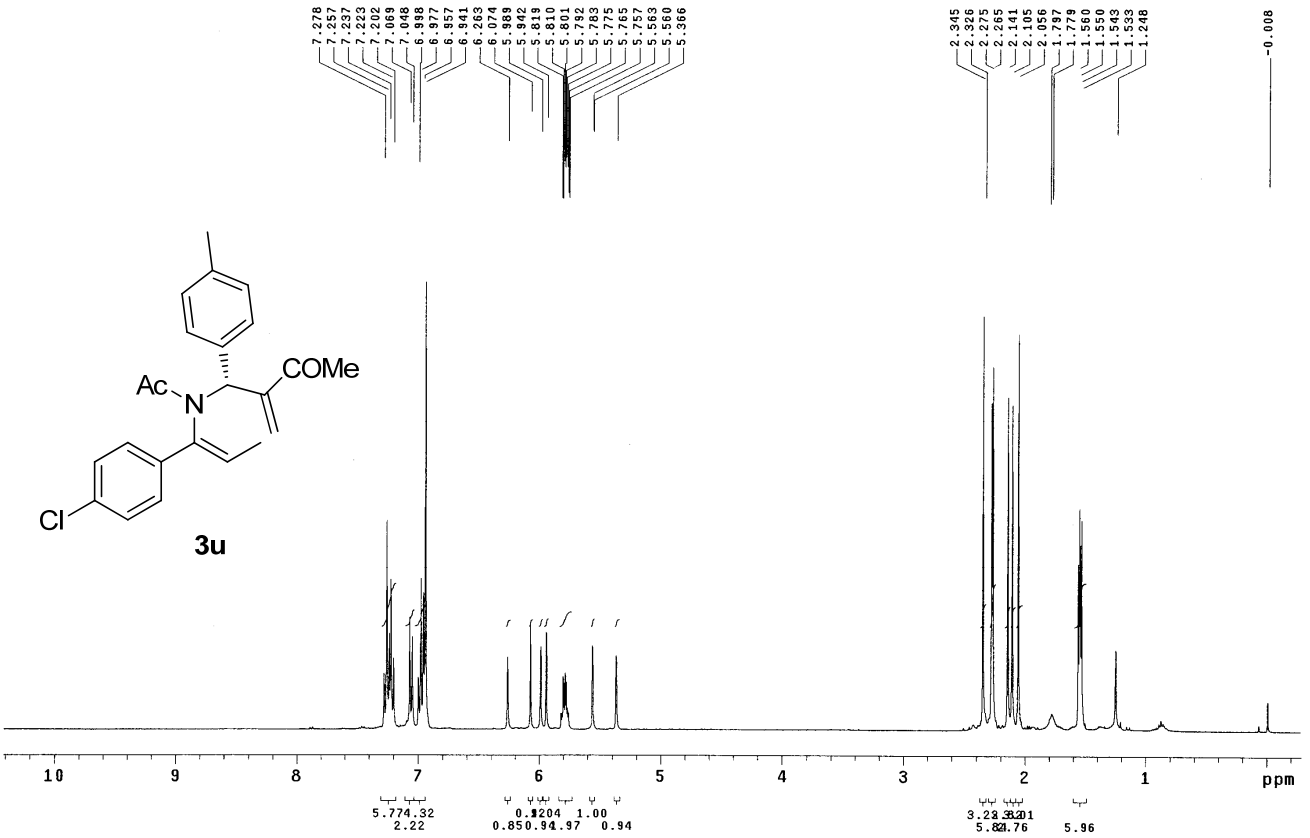
140
120
100
80
60
40
20
ppm



	Peak Name	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	Peak1	6.065	6037236	49.16	548634	58.22
2	Peak2	8.002	6244644	50.84	393696	41.78



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	6.059	527155	6.45	47848	9.06
2	7.990	7646613	93.55	480174	90.94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7.237
7.223
7.202
7.069
7.048
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6.977
6.957
6.941
6.263
6.074
5.989
5.942
5.819
5.810
5.801
5.792
5.783
5.775
5.765
5.757
5.563
5.560
5.366
2.345
2.326
2.275
2.265
2.141
2.105
2.056
1.797
1.779
1.560
1.550
1.543
1.533
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Ac
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COMe
Cl
3u
10
9
8
7
6
5
4
3
2
1
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0.94
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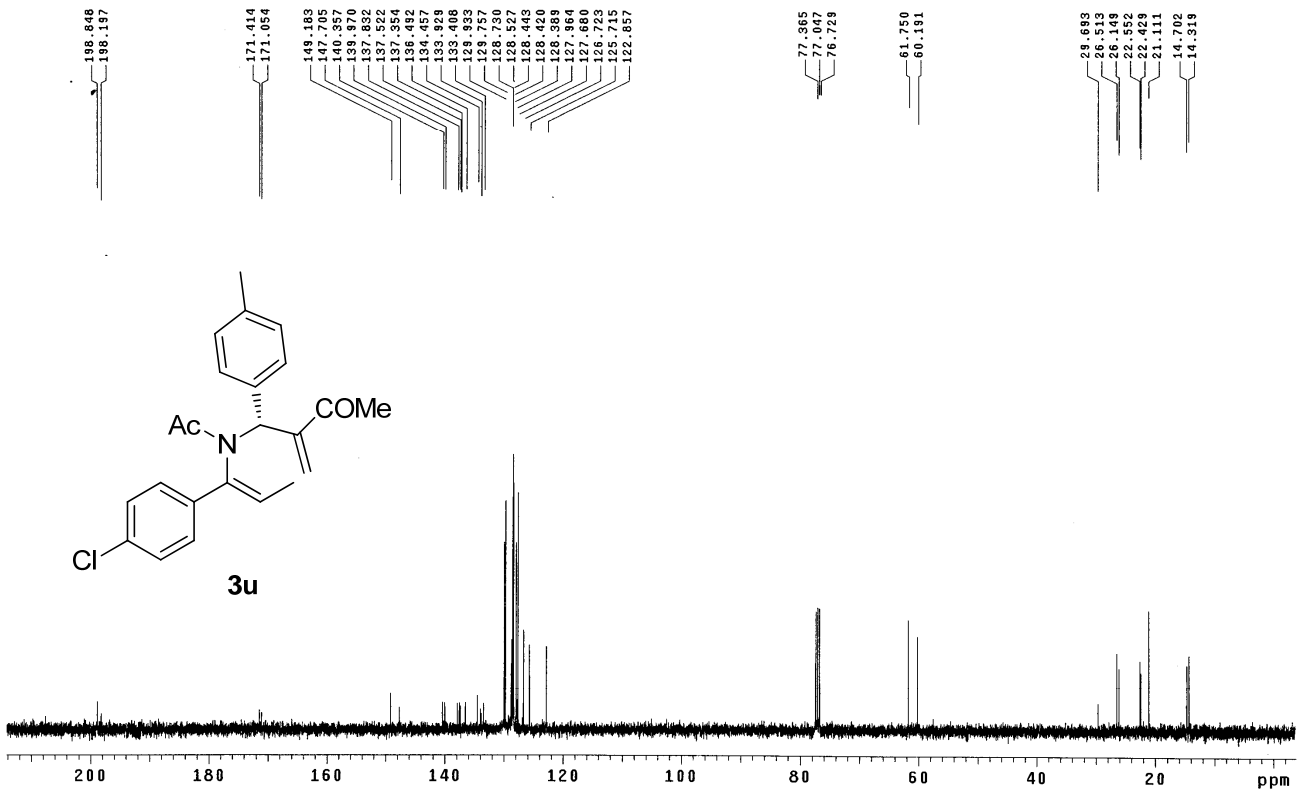
4.76
5.96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133.929
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129.933
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128.730
128.527
128.443
128.420
128.389
127.964
127.680
126.723
125.715
122.857
77.365
77.047
76.729
61.750
60.191
28.683
26.513
26.149
22.552
22.429
21.111
14.702
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Ac
N
COMe
Cl
3u
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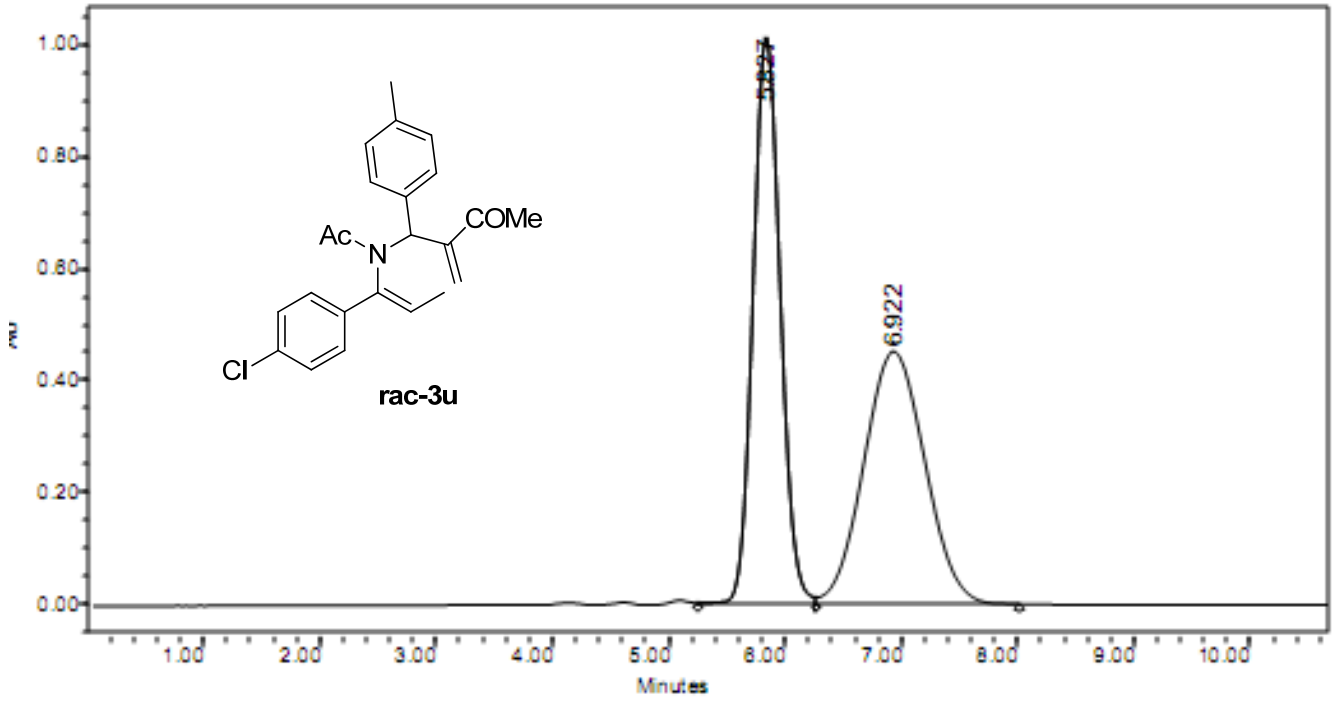
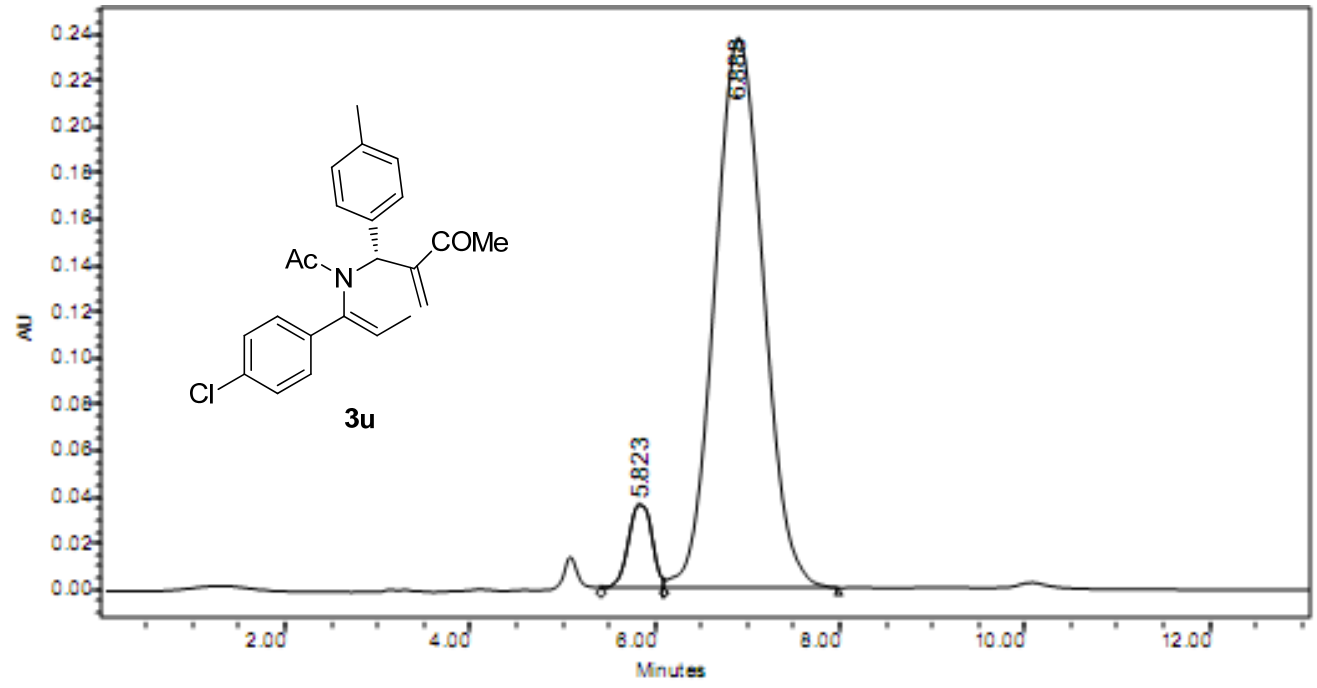
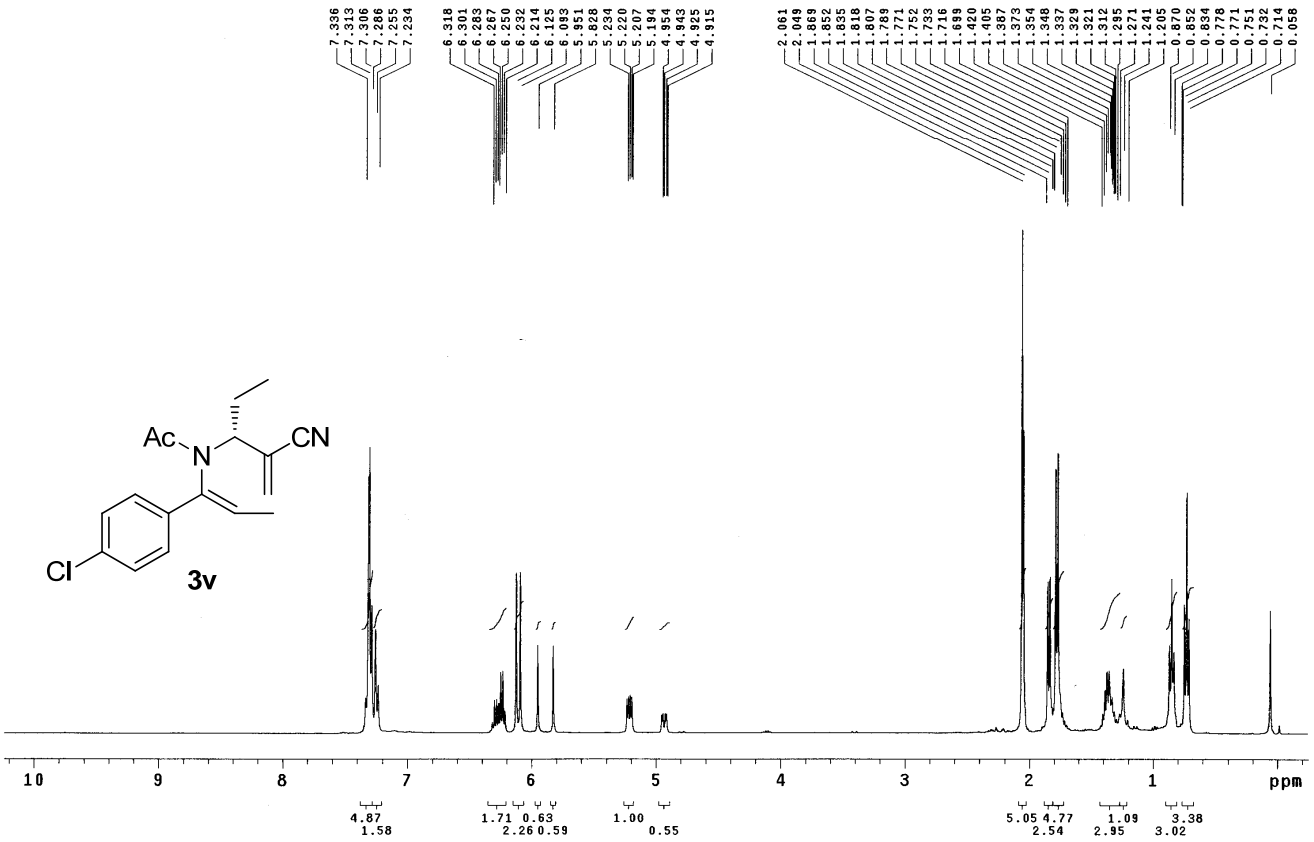
140
120
100
80
60
40
20
ppm



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	5.827	16544143	49.94	1013745	69.15
2	6.922	16586335	50.06	452288	30.85



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	5.823	678519	7.21	36720	13.36
2	6.888	8734055	92.79	238164	86.64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6.267
6.250
6.232
6.214
6.125
6.083
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5.828
5.234
5.220
5.207
5.194
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4.943
4.925
4.915
2.061
2.049
1.869
1.852
1.835
1.818
1.807
1.789
1.771
1.752
1.733
1.716
1.699
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1.405
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1.354
1.348
1.337
1.329
1.321
1.312
1.295
1.271
1.241
1.205
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0.852
0.834
0.778
0.771
0.751
0.732
0.714
0.058
Ac
N
CN
Cl
3v
10
9
8
7
6
5
4
3
2
1
ppm
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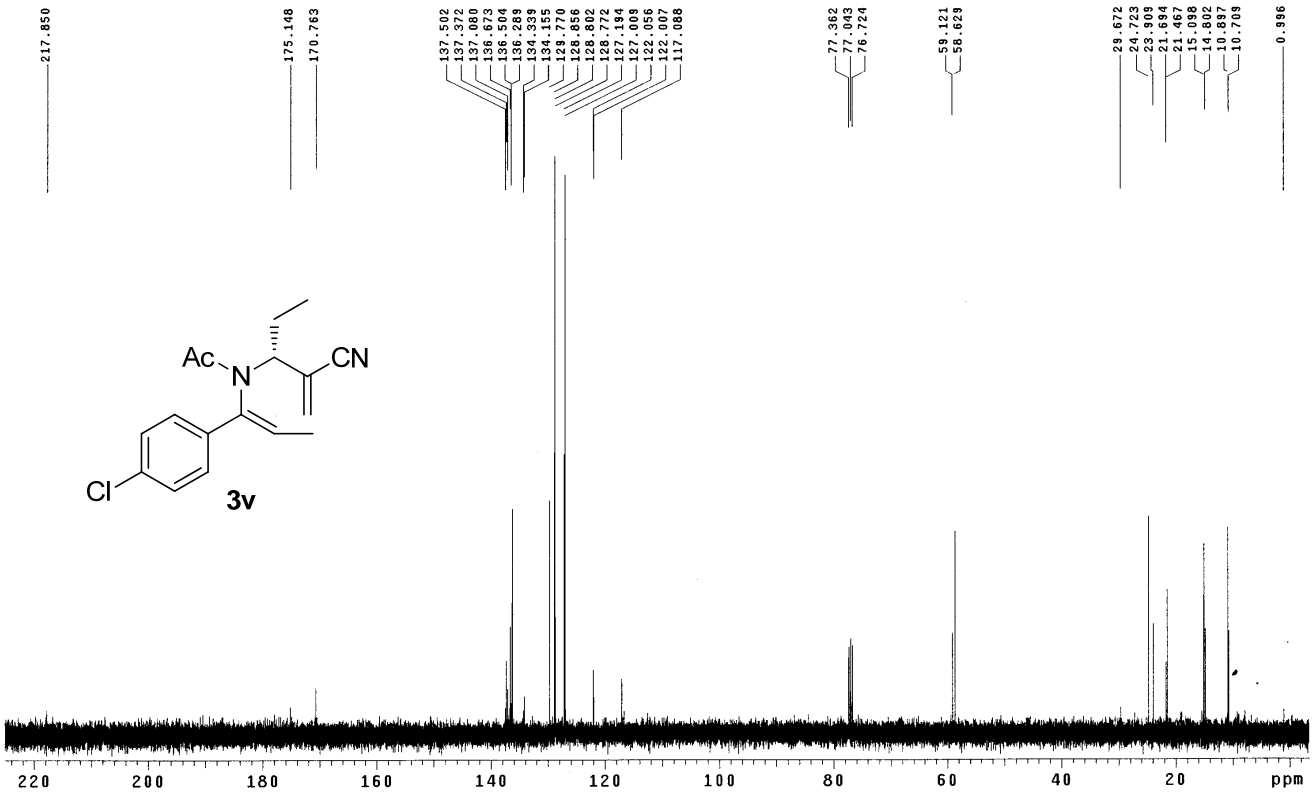
3.02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127.008
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122.007
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77.362
77.043
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58.623
29.672
24.723
23.909
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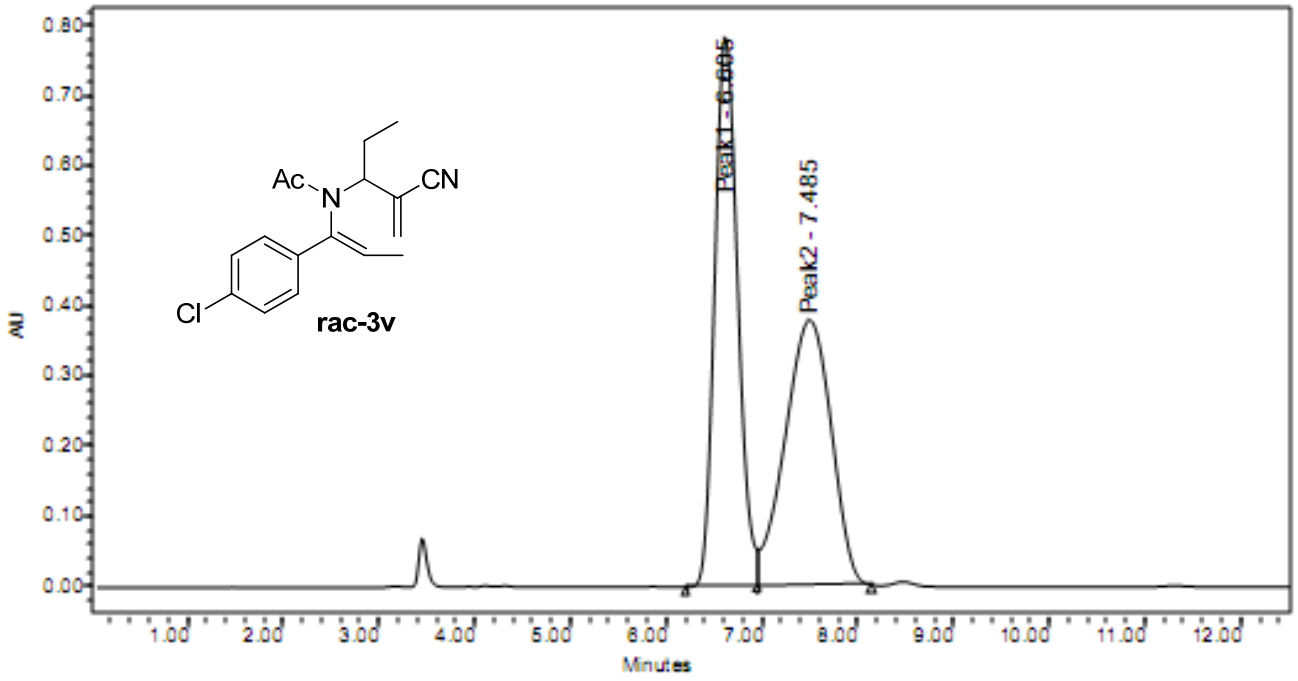
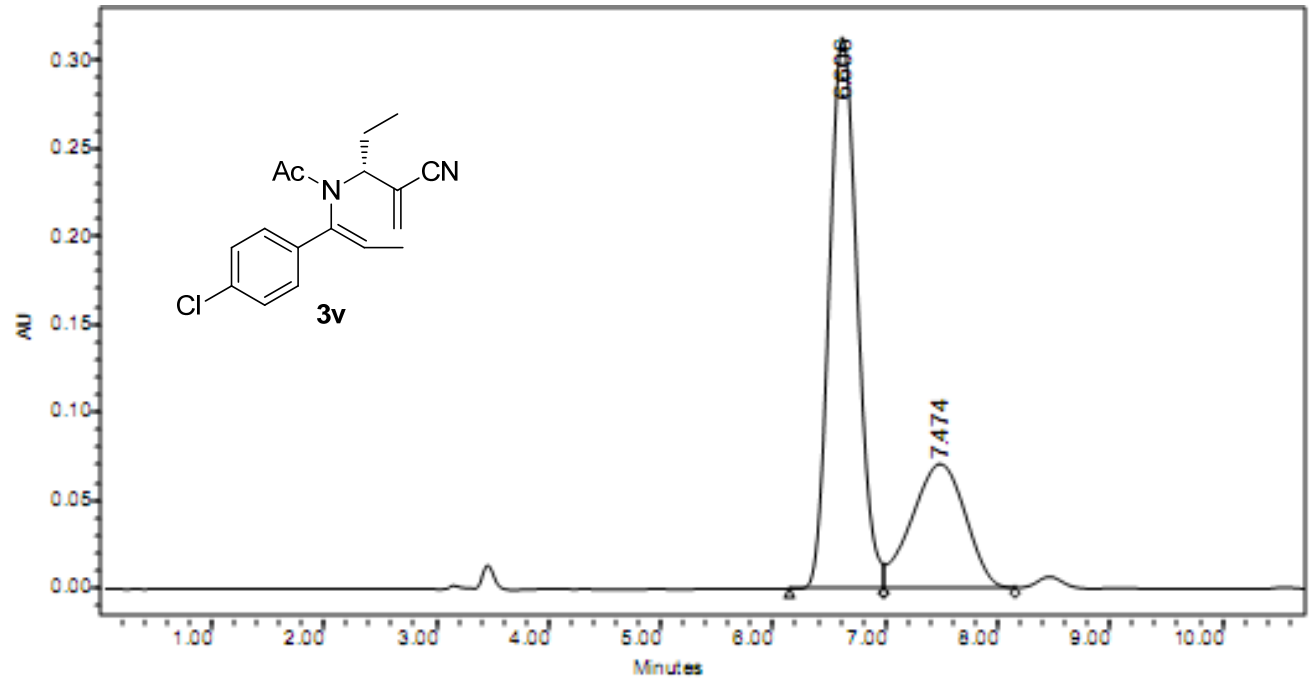
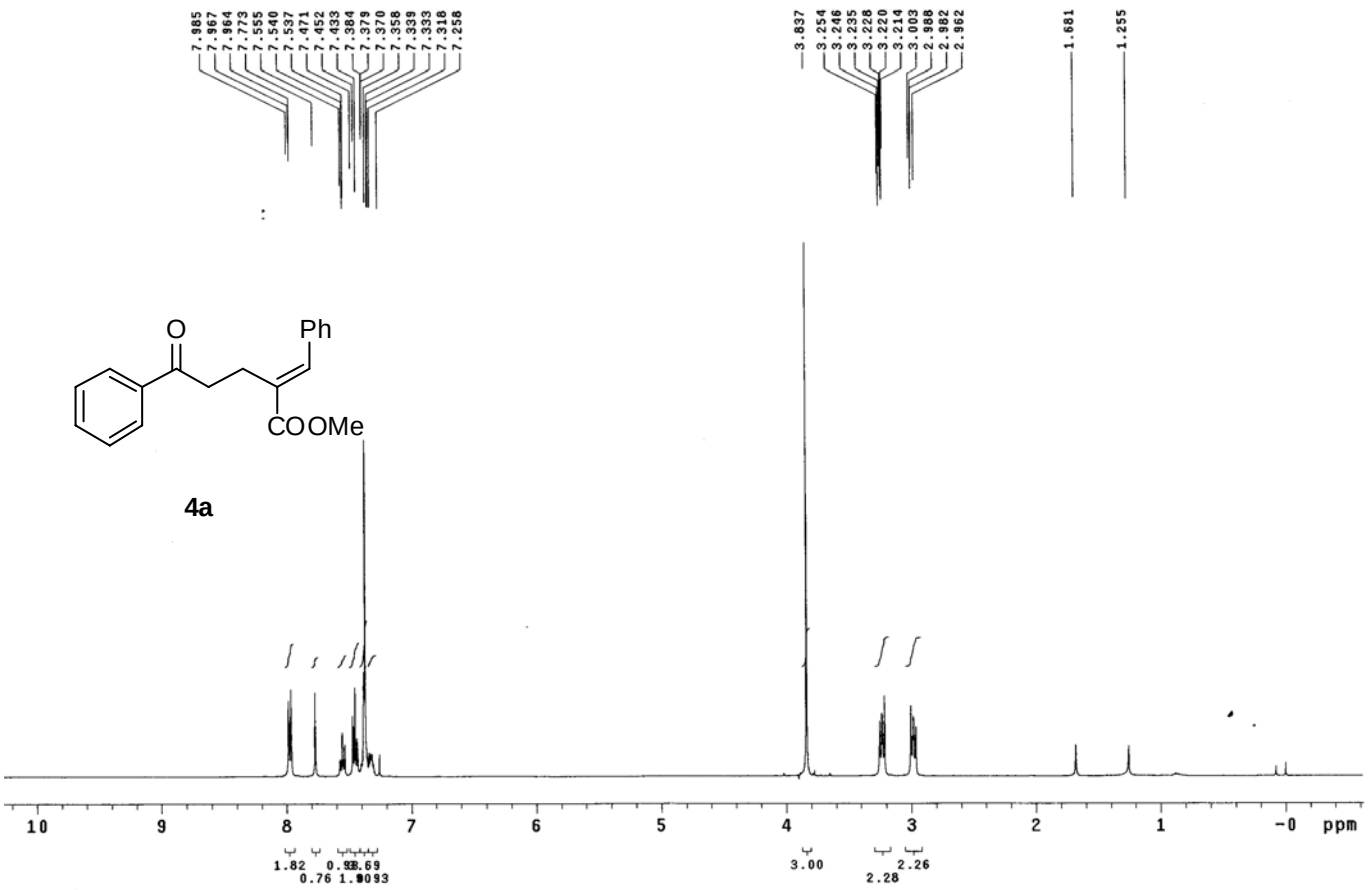
20
ppm



	Peak Name	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	Peak1	6.605	13062983	50.60	784433	67.41
2	Peak2	7.485	12753668	49.40	379273	32.59



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	6.606	5409835	69.42	314090	81.53
2	7.474	2383337	30.58	71137	18.47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7.379
7.370
7.358
7.339
7.333
7.318
7.258
3.837
3.254
3.246
3.235
3.228
3.220
3.214
3.003
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2.982
2.962
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O
Ph
COOMe
4a
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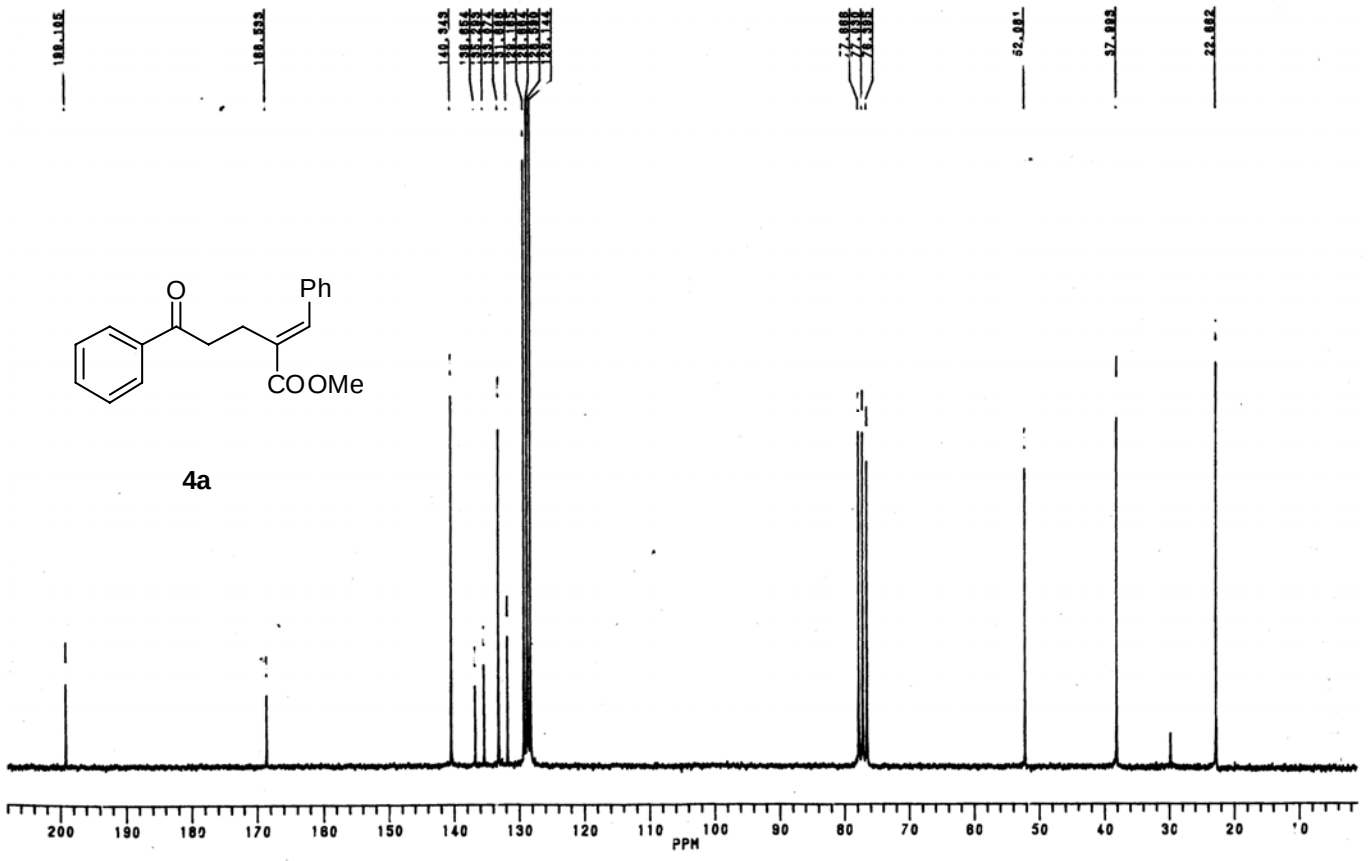
2.26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Ph
COOMe
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60
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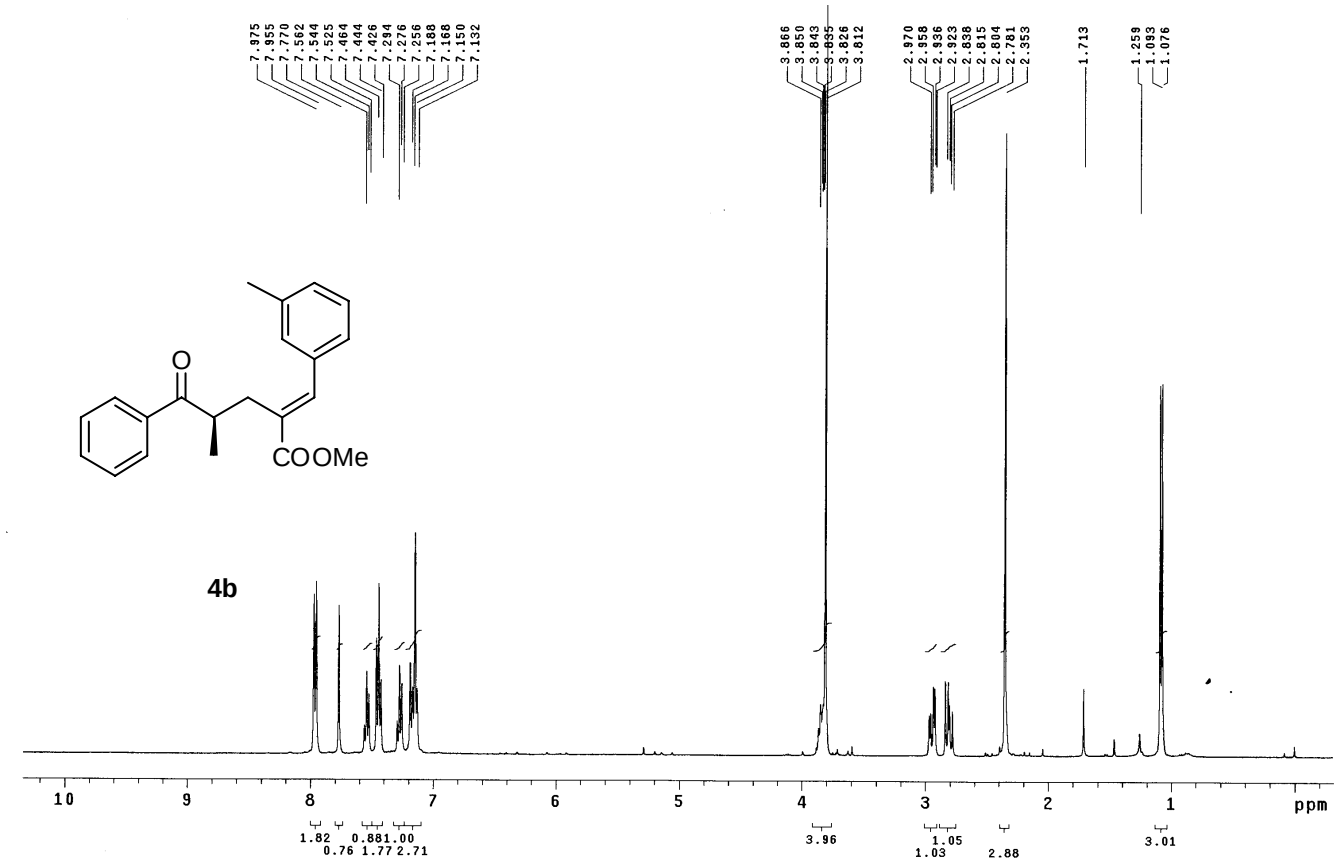
30
20
10
PPM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7.444
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7.188
7.168
7.150
7.132
3.866
3.850
3.843
3.826
3.812
2.970
2.958
2.936
2.923
2.838
2.815
2.804
2.781
2.353
1.713
1.259
1.093
1.076
O
COOMe
4b
10
9
8
7
6
5
4
3
2
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3.96
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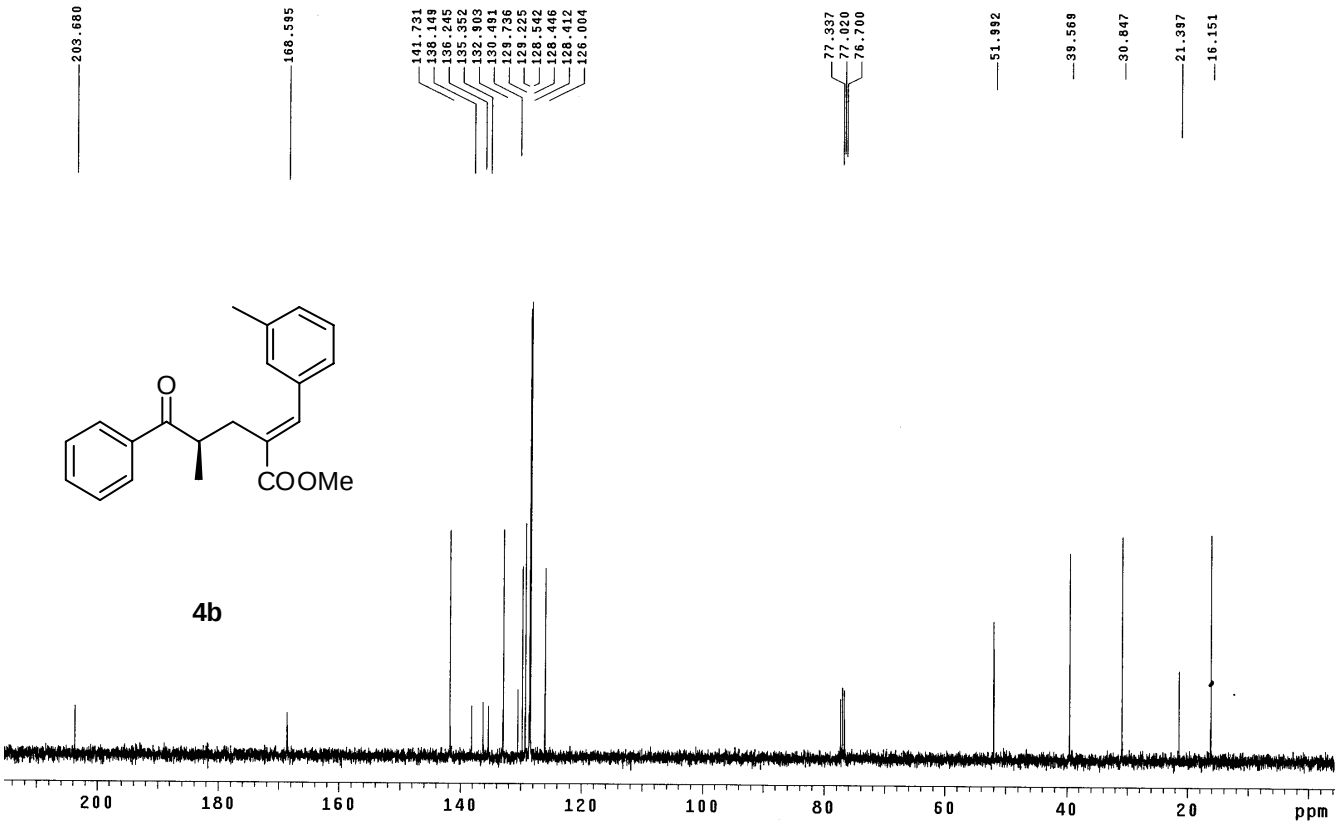
1.03
2.88
3.01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128.412
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77.337
77.020
76.700
51.992
39.569
30.847
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16.151
O
COOMe
4b
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140
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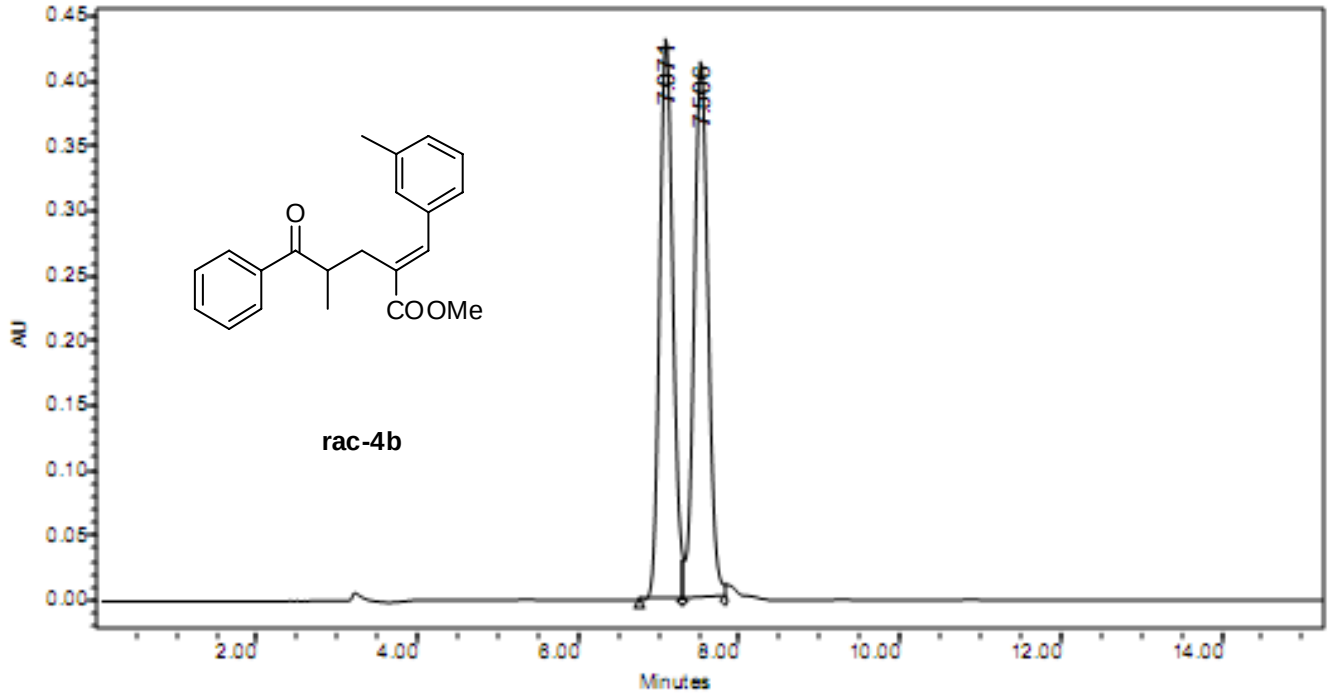
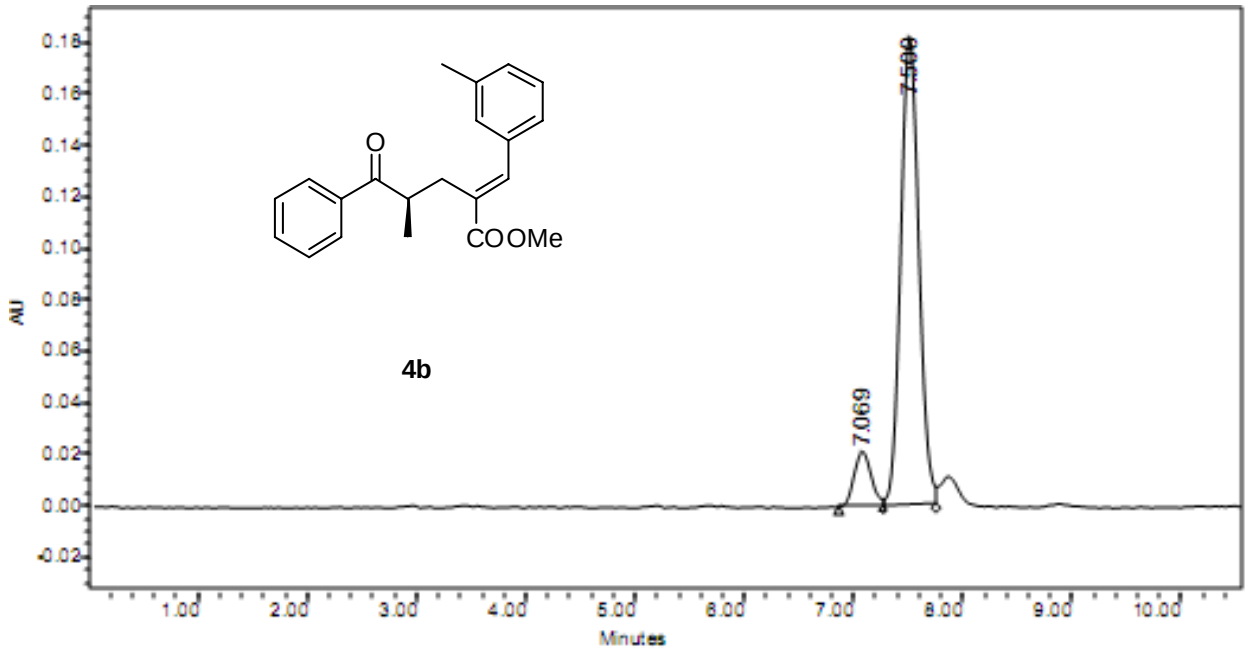
60
40
20
ppm



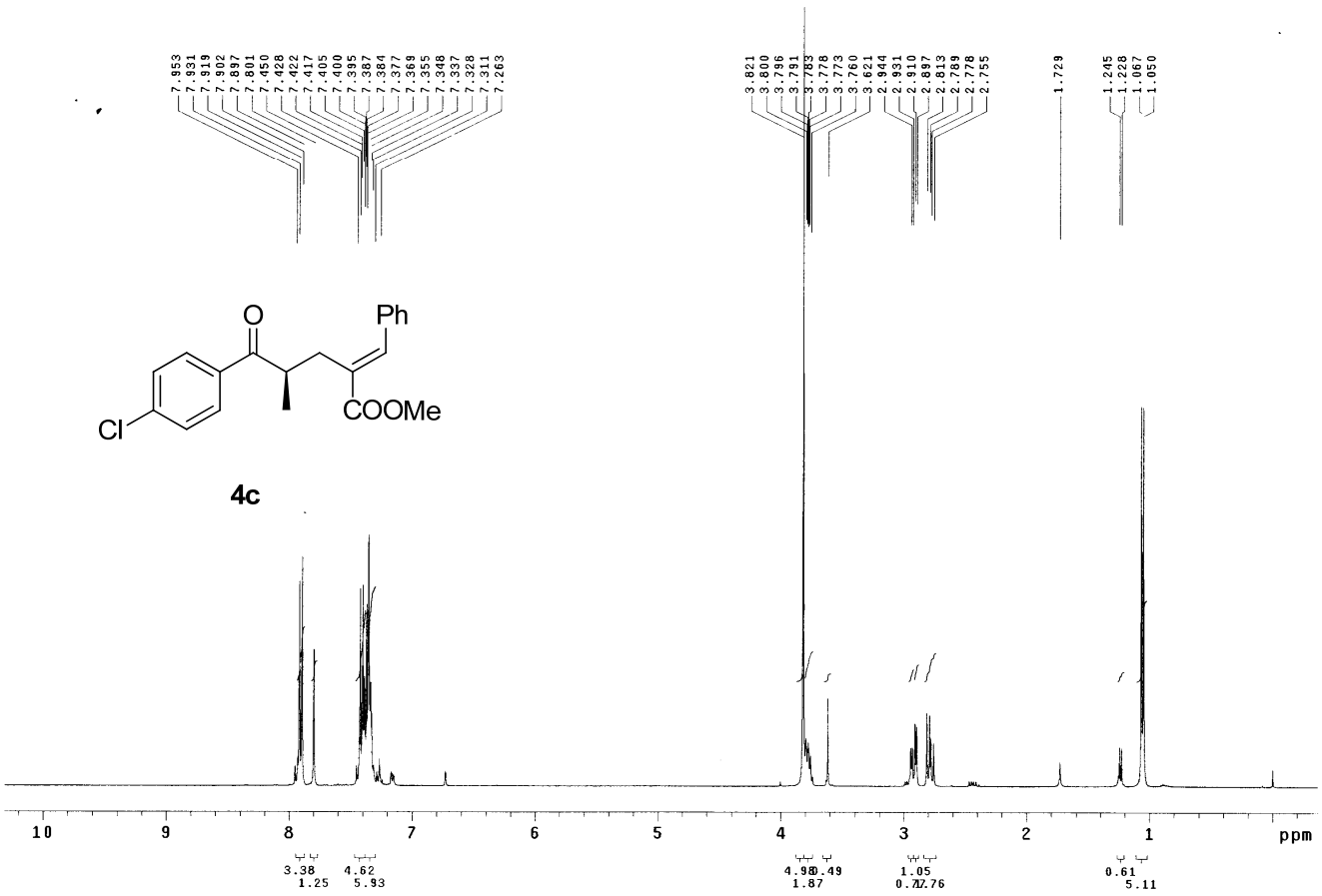
rac-4b

	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	7.071	4992765	49.94	432366	51.08
2	7.506	5004028	50.06	414088	48.92



4b

	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	7.069	234095	9.99	21400	10.51
2	7.500	2109706	90.01	182253	89.49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7.428
7.422
7.417
7.405
7.400
7.395
7.387
7.384
7.377
7.369
7.355
7.348
7.337
7.328
7.311
7.263
3.821
3.800
3.796
3.791
3.783
3.778
3.773
3.760
3.621
2.944
2.931
2.910
2.897
2.813
2.789
2.778
2.755
1.729
1.245
1.228
1.067
1.050
O
Ph
Cl
COOMe
4c
10
9
8
7
6
5
4
3
2
1
ppm
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4.62
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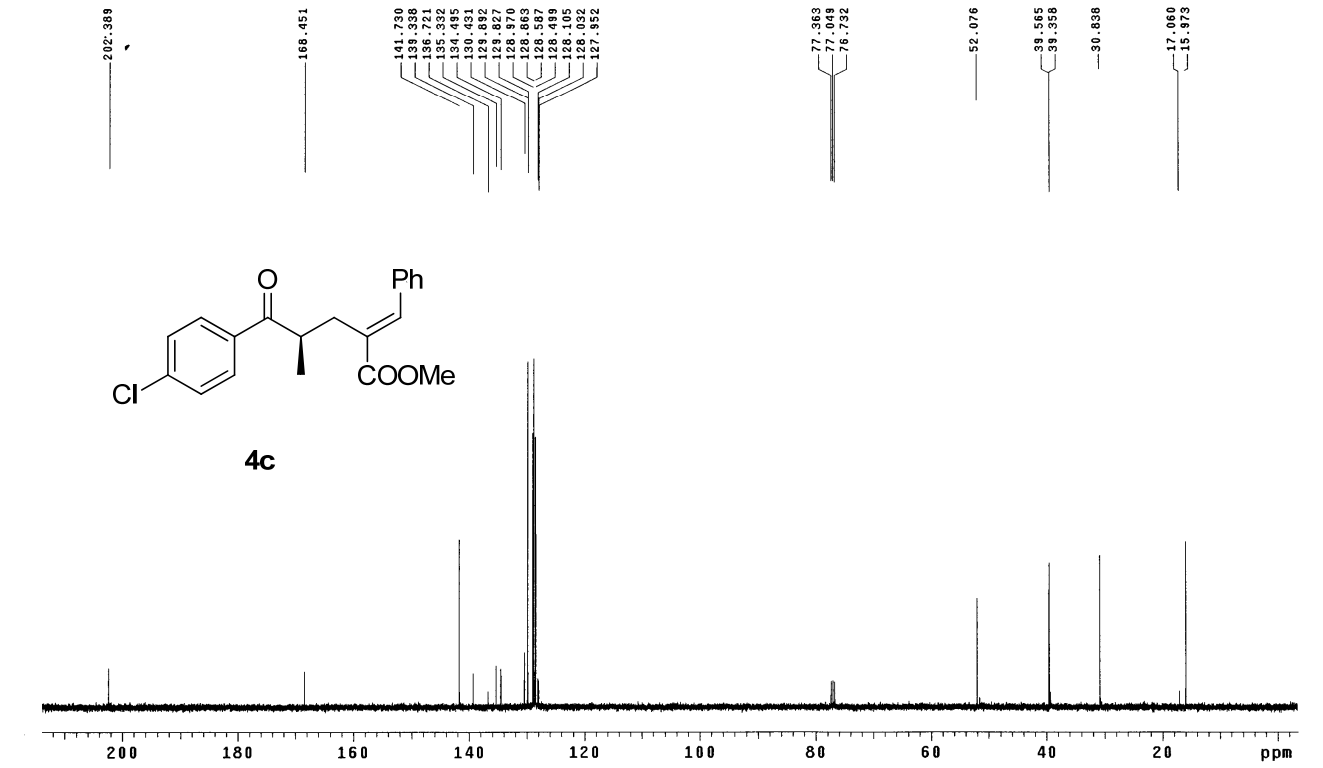
1.05
0.76
0.61
5.11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141.730
139.338
138.721
135.332
134.495
130.431
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128.498
128.105
128.032
127.952
77.363
77.049
76.732
52.076
39.565
39.358
30.838
17.060
15.973
O
Ph
Cl
COOMe
4c
200
180
160
140
120
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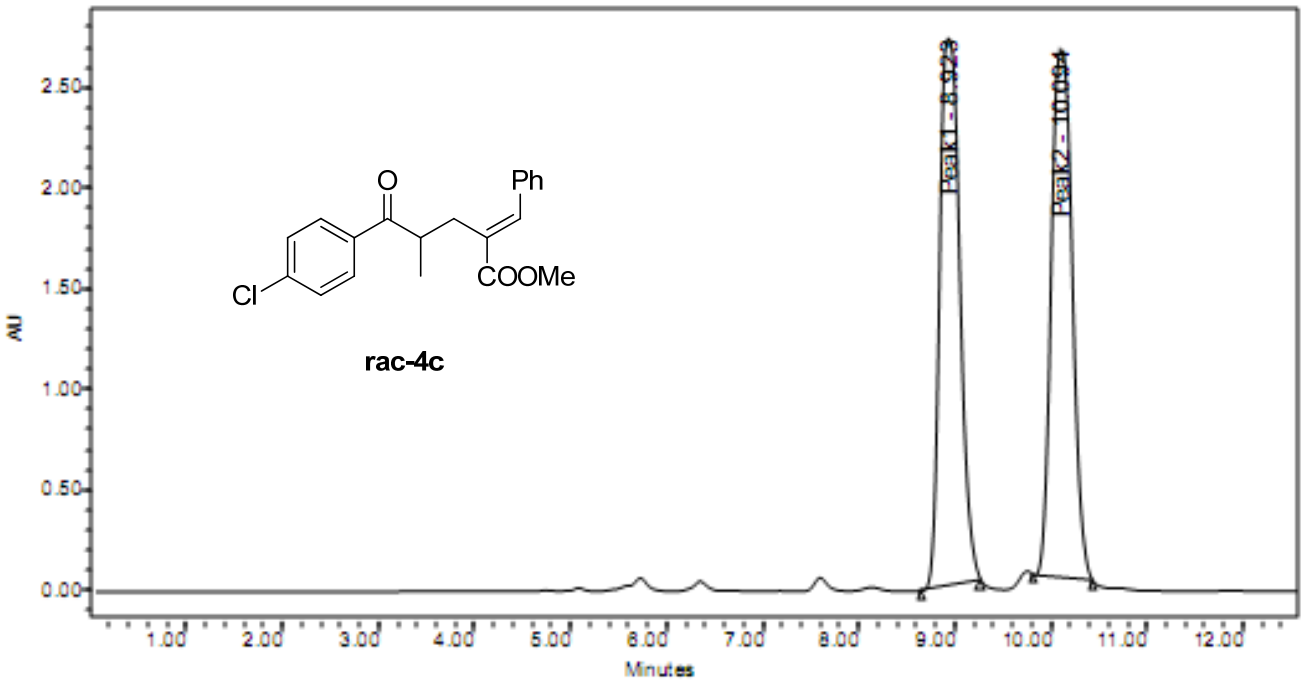
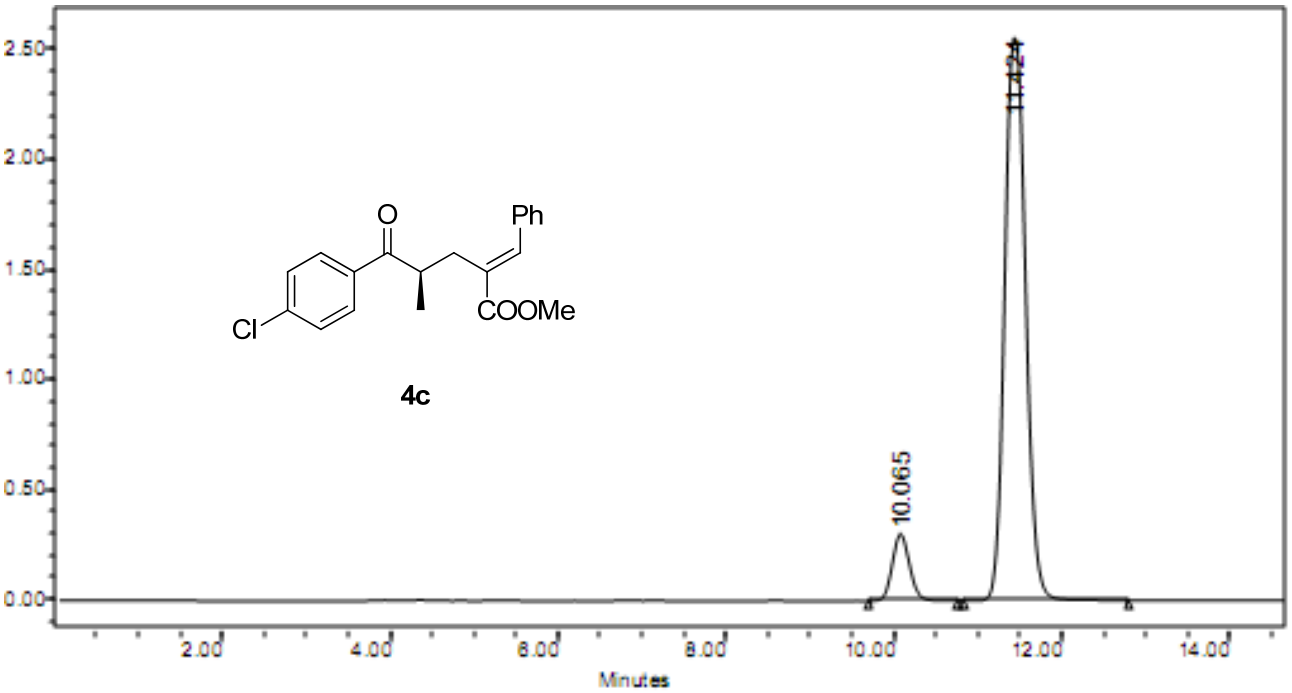
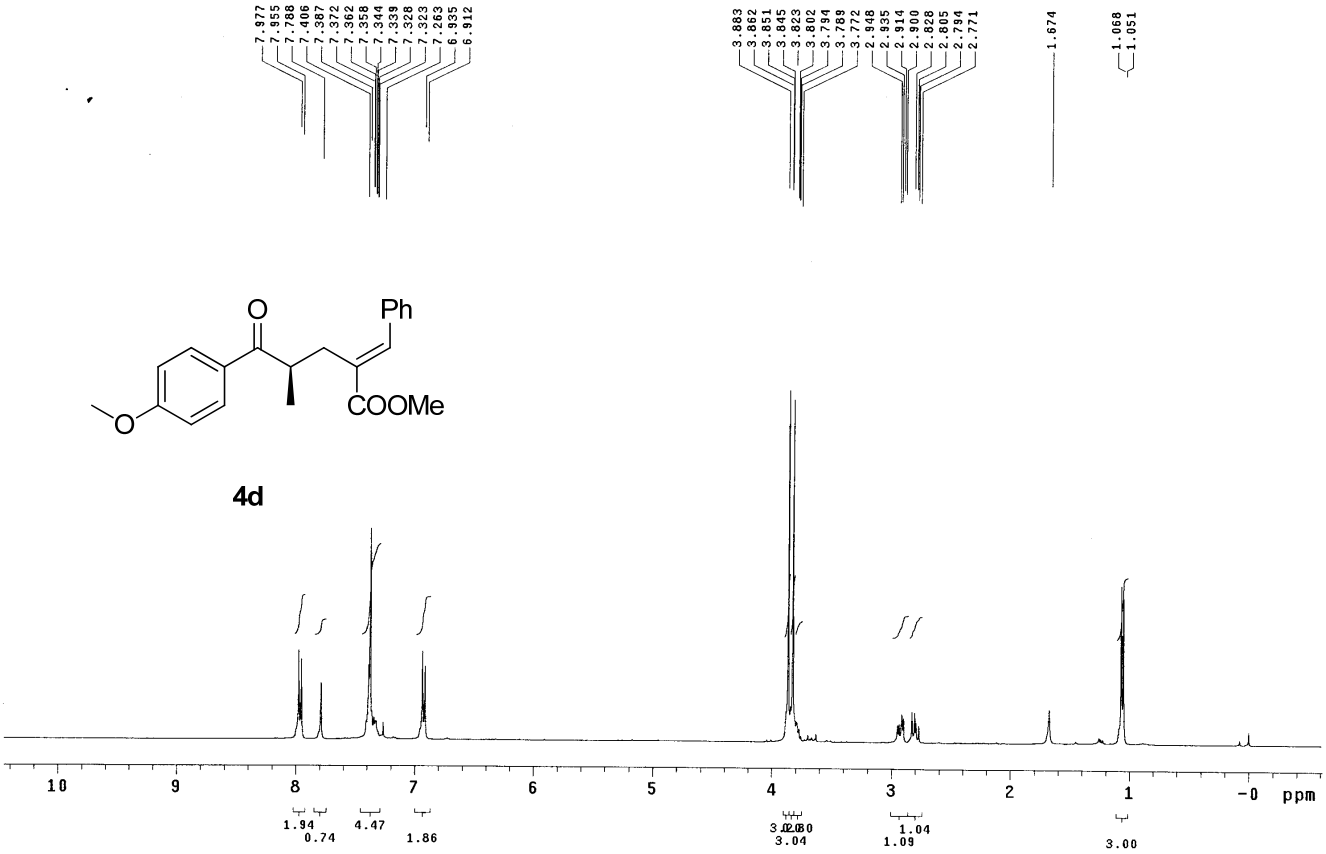
80
60
40
20
ppm



	Peak Name	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	Peak1	8.923	39877019	50.07	2728467	50.93
2	Peak2	10.094	39770637	49.93	2628932	49.07



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	10.065	4290031	8.84	302310	10.56
2	11.424	44237992	91.16	2559382	89.44


7.977
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7.387
7.372
7.362
7.358
7.344
7.339
7.328
7.323
7.263
6.935
6.912
3.883
3.862
3.851
3.845
3.823
3.802
3.784
3.769
3.772
2.948
2.935
2.914
2.900
2.828
2.805
2.794
2.771
1.674
1.068
1.051
O
Ph
COOMe
O
4d
10
9
8
7
6
5
4
3
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1
-0
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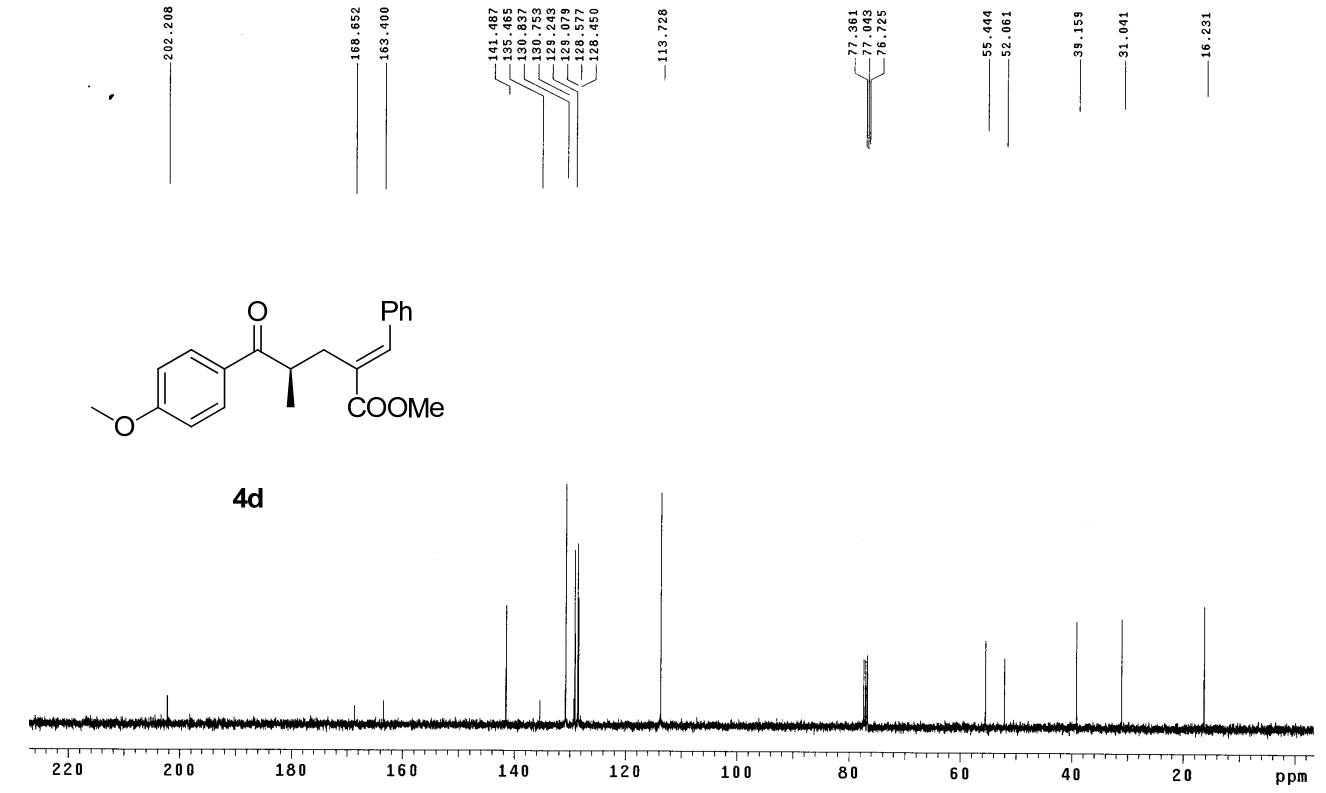
1.04
1.09
3.00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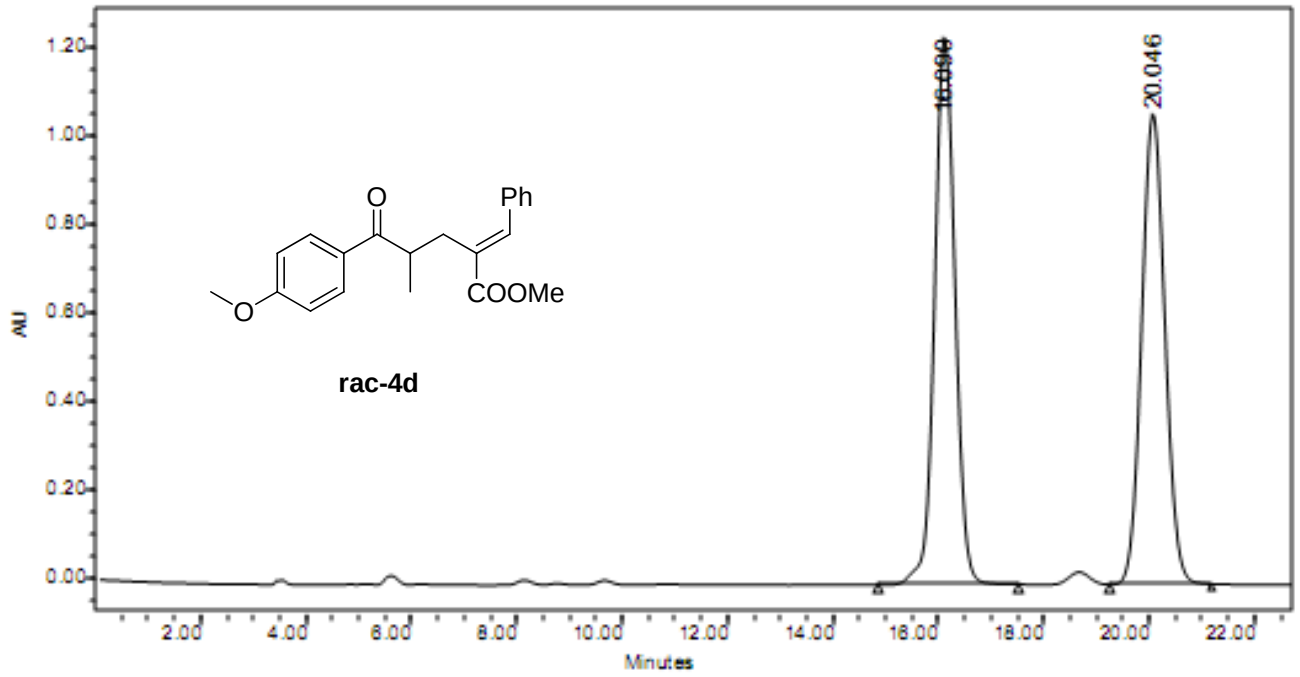
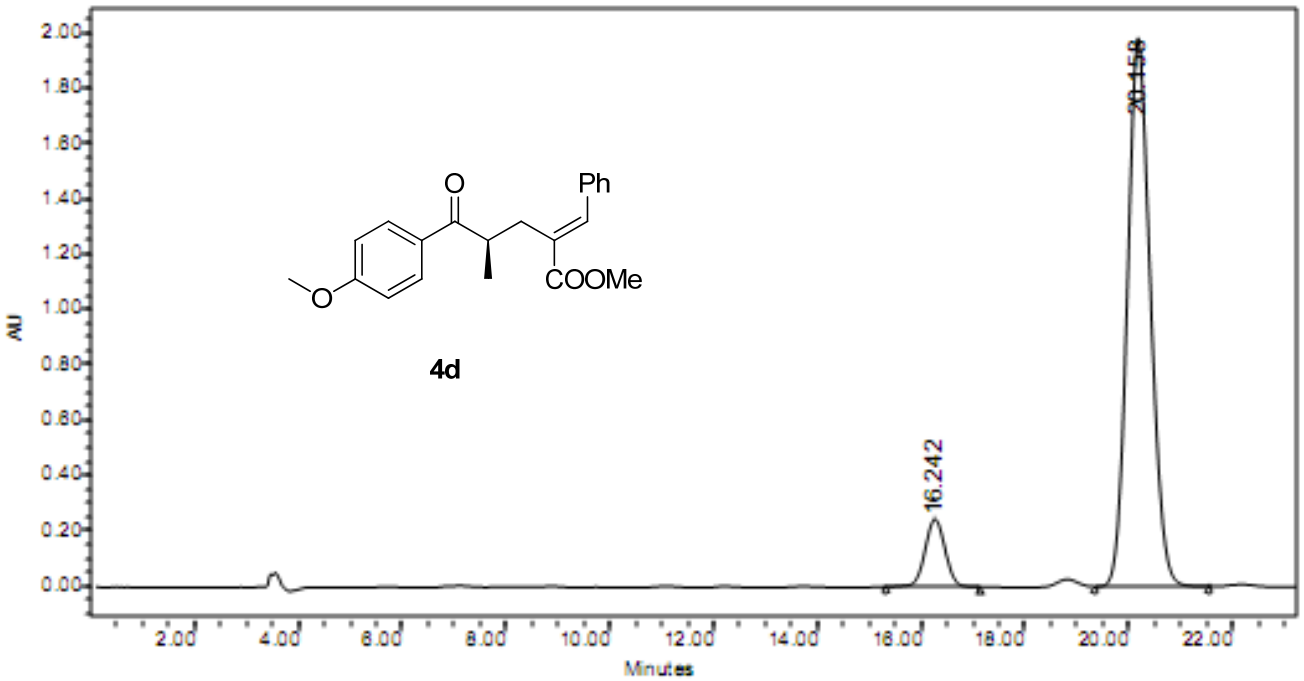
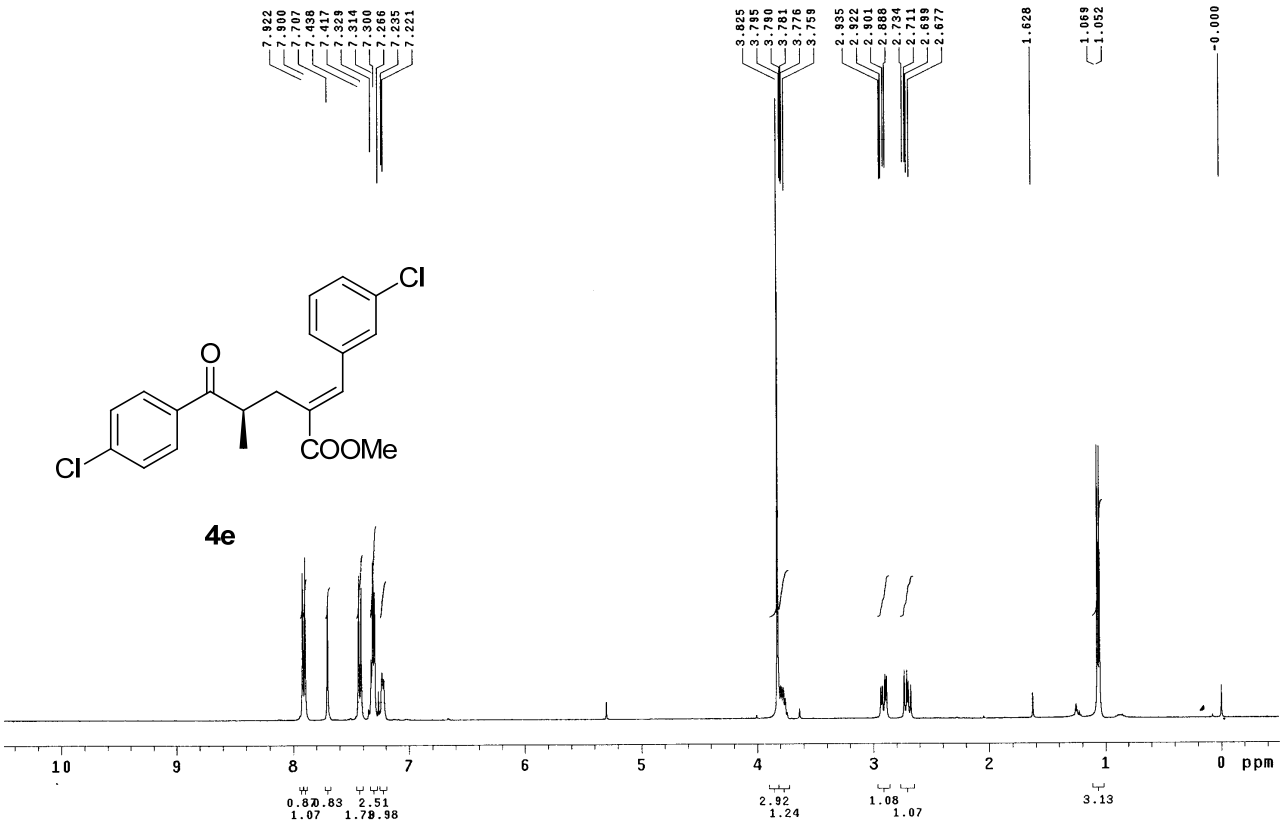
163.400
141.487
135.465
130.837
130.753
129.243
129.079
128.577
128.450
113.728
77.361
77.043
76.725
55.444
52.061
39.159
31.041
16.231
O
Ph
COOMe
O
4d
220
200
180
160
140
120
100
80
60
40
20
ppm



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	16.090	33391538	50.59	1235977	53.80
2	20.046	32606759	49.41	1061423	46.20



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	16.242	6640225	9.78	244365	10.96
2	20.158	61226339	90.22	1985217	89.04


7.922
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7.707
7.438
7.417
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7.314
7.300
7.266
7.235
7.221
3.825
3.795
3.790
3.781
3.776
3.759
2.935
2.922
2.901
2.888
2.734
2.711
2.699
2.677
1.628
1.068
1.052
-0.000
Cl
O
COOMe
Cl
4e
10
9
8
7
6
5
4
3
2
1
0 ppm
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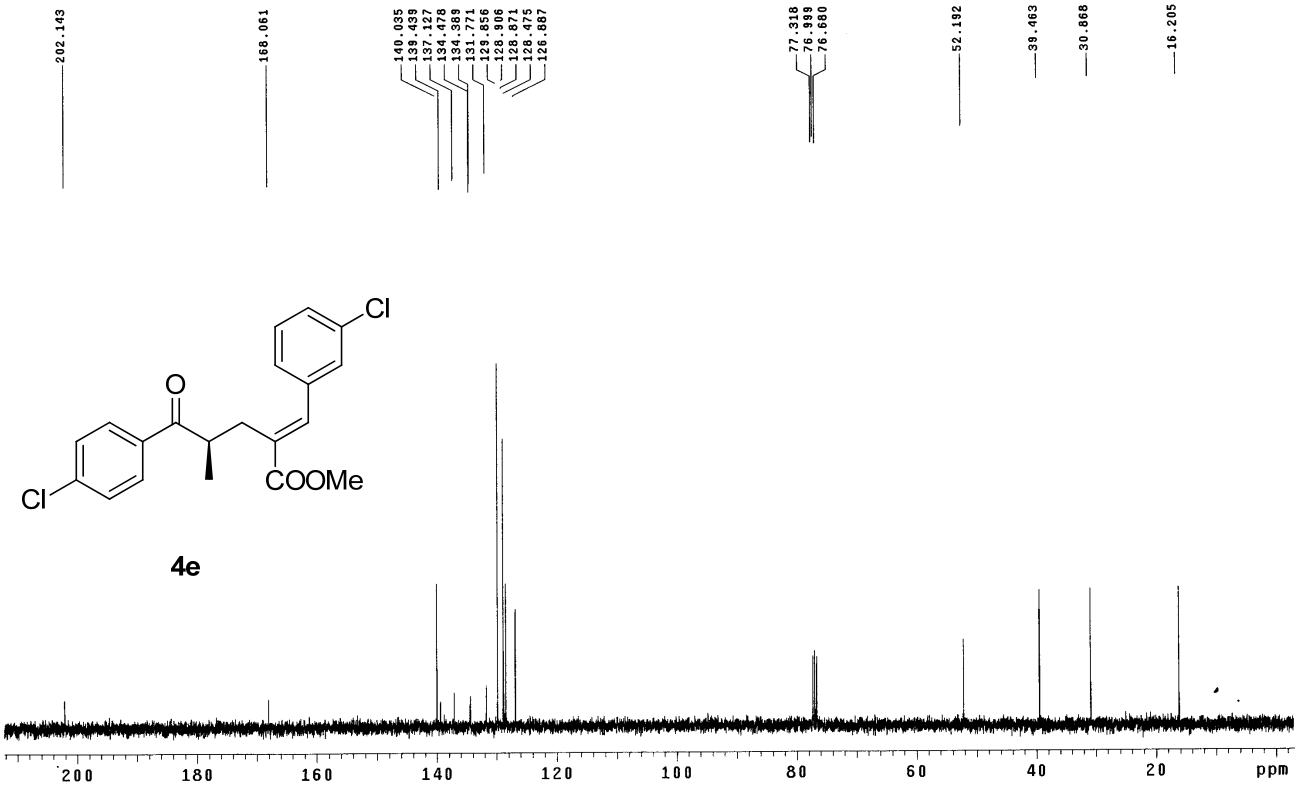
0.83
2.51
1.07
1.78
8.98
2.92
1.24
1.08
1.07
3.13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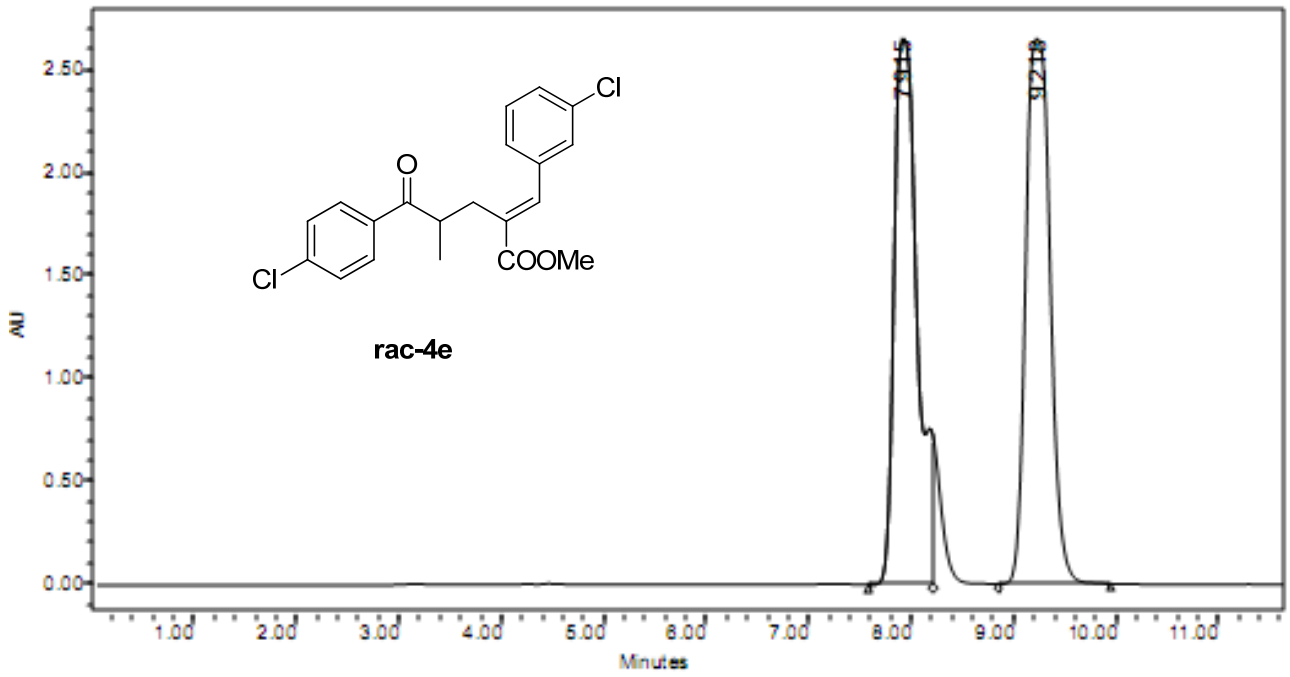
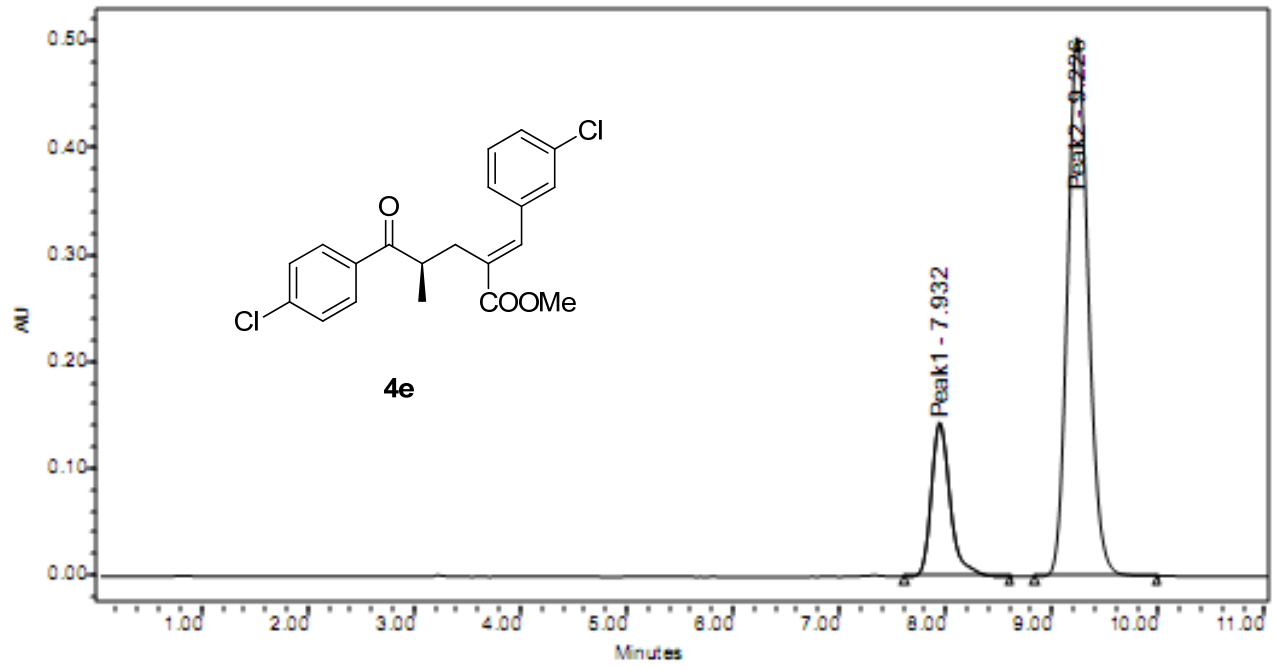
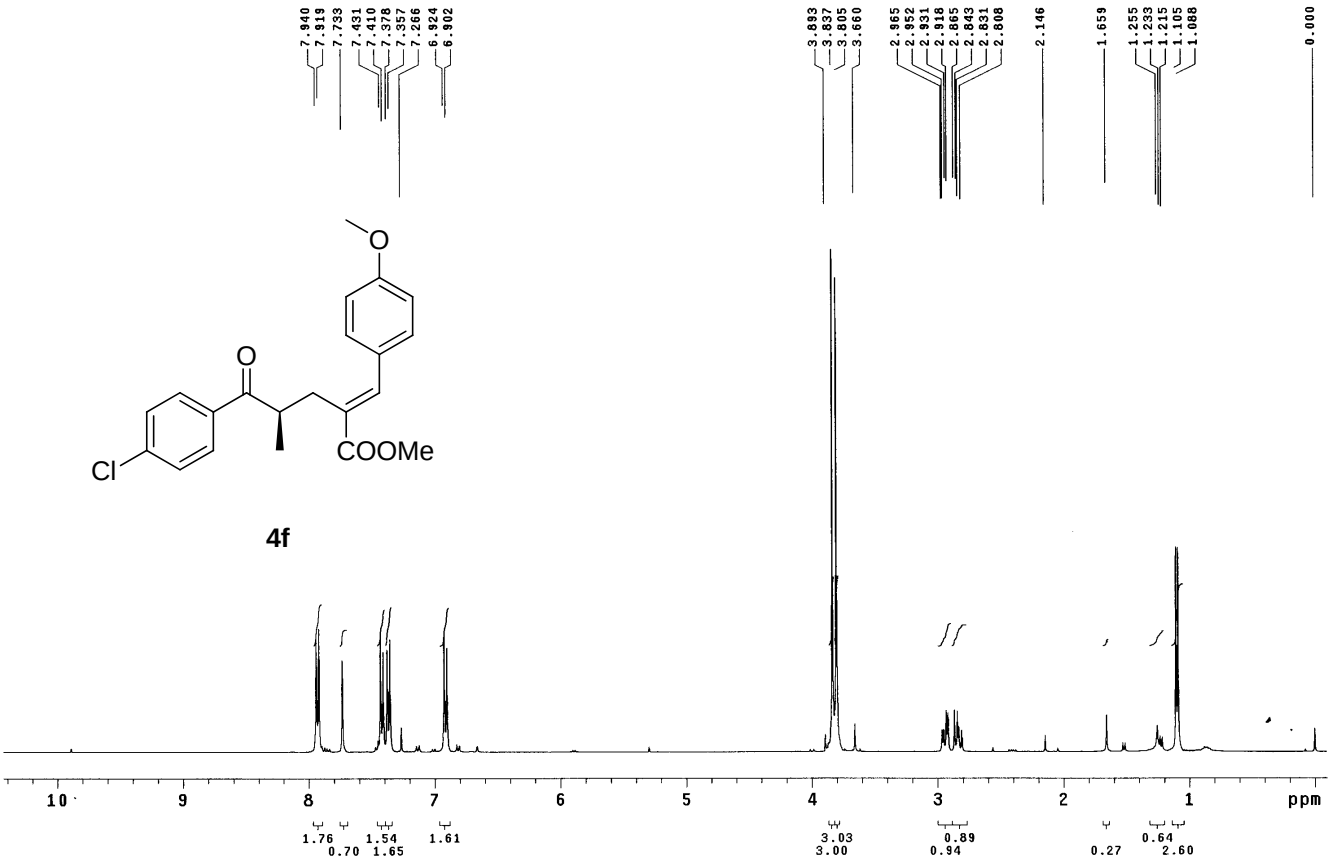
137.127
134.478
134.389
131.771
129.856
128.906
128.871
128.475
126.887
77.318
76.999
76.680
52.192
39.463
30.868
16.205
Cl
O
COOMe
Cl
4e
200
180
160
140
120
100
80
60
40
20
ppm



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	7.915	42367098	49.10	2654056	50.03
2	9.218	43919333	50.90	2651250	49.97



	Peak Name	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	Peak1	7.932	1783050	20.67	143721	22.15
2	Peak2	9.226	6843331	79.33	505151	77.85


7.940
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7.431
7.410
7.378
7.357
7.266
6.924
6.902
3.893
3.837
3.805
3.660
2.965
2.952
2.931
2.918
2.865
2.843
2.831
2.808
2.146
1.659
1.255
1.233
1.215
1.105
1.088
0.000
O
O
Cl
COOMe
10
9
8
7
6
5
4
3
2
1
ppm
1.76
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1.65
1.61
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3.00
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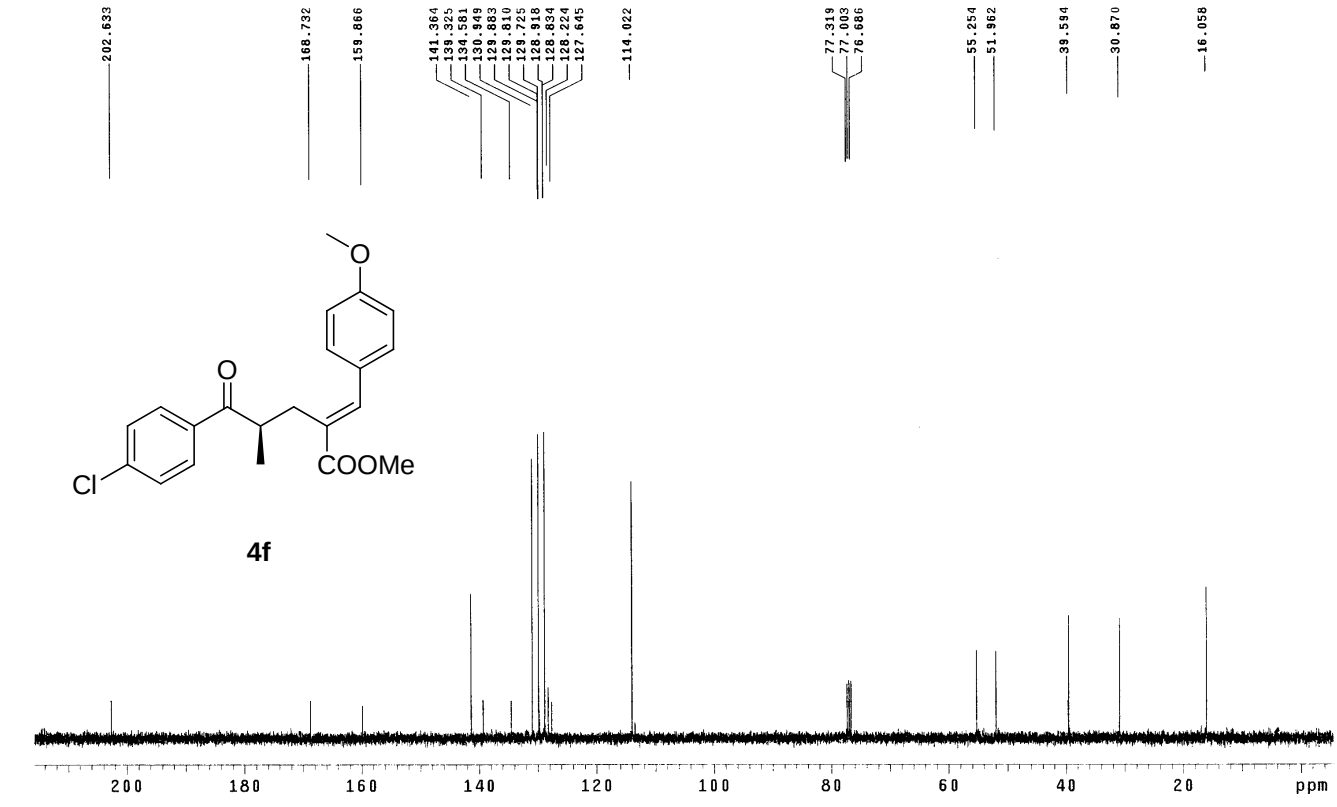
0.94
0.27
0.64
2.60

4f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130.949
129.883
129.810
129.725
128.918
128.834
128.224
127.645
114.022
77.319
77.003
76.686
55.254
51.962
39.594
30.870
16.058
O
O
Cl
COOMe
200
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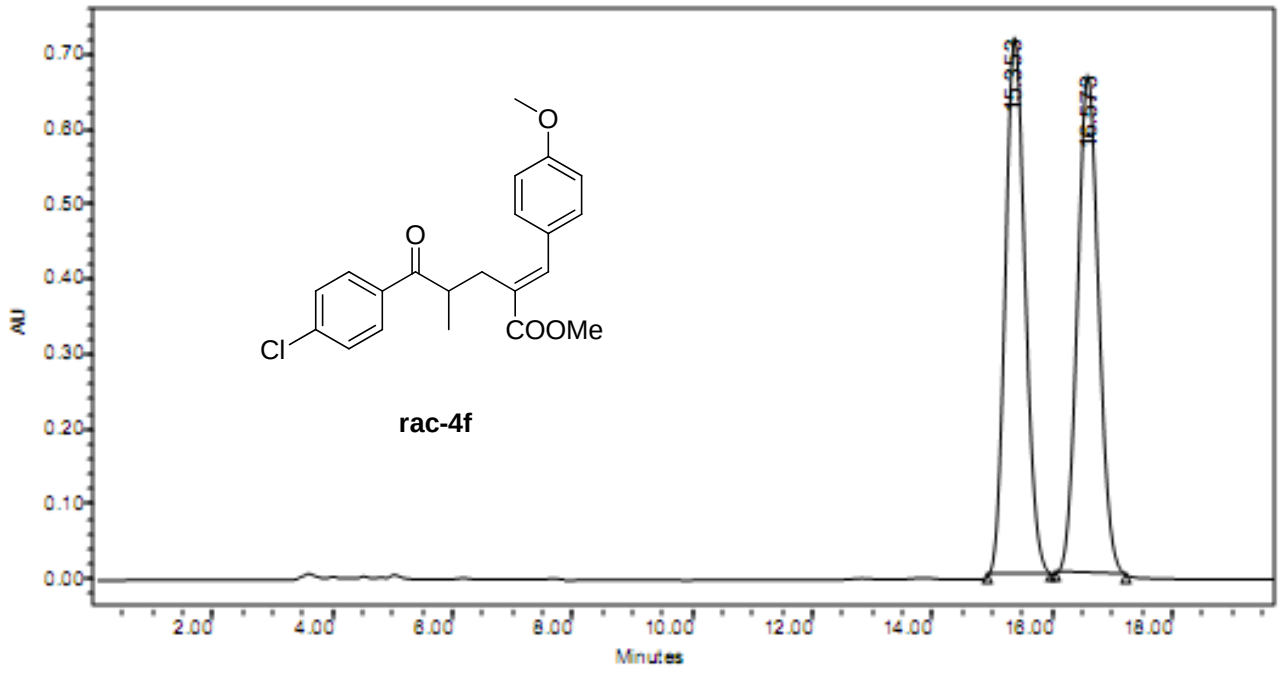
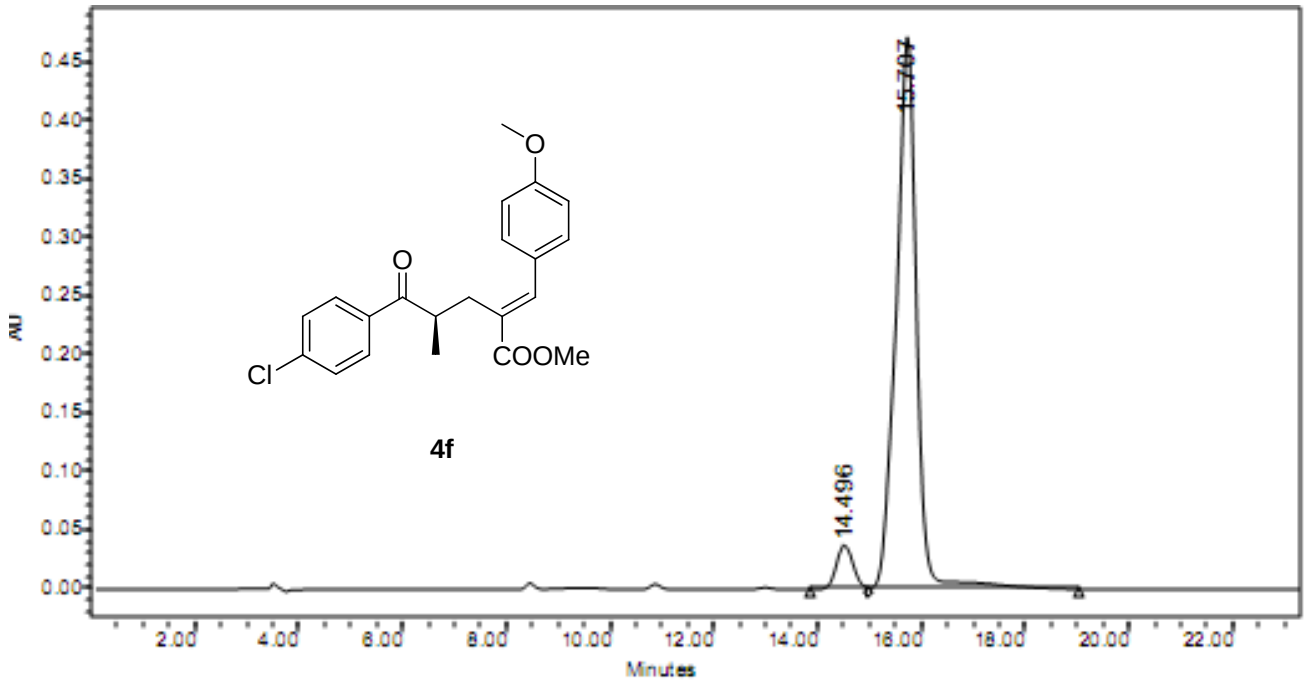
160
140
120
100
80
60
40
20
ppm

4f



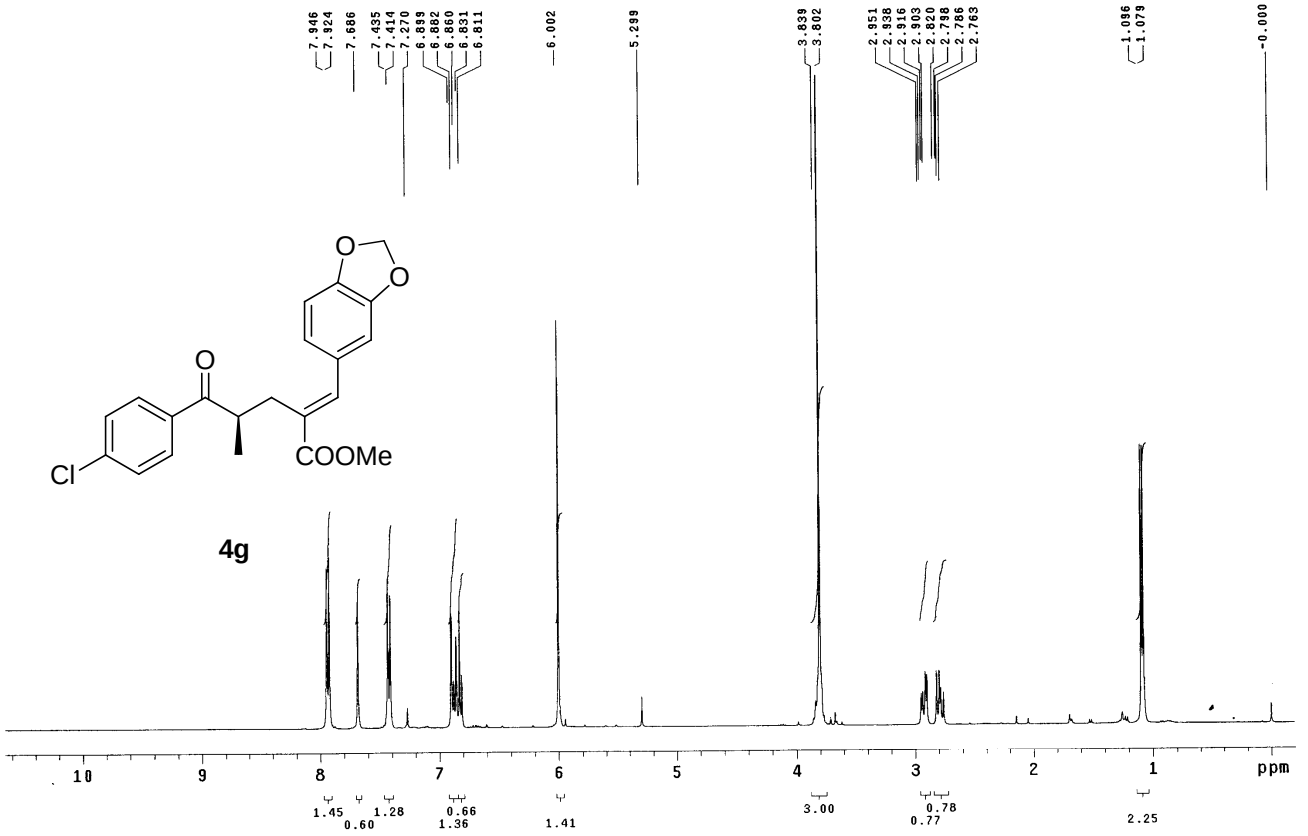
rac-4f

	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	15.353	16446007	50.04	716665	51.88
2	16.573	16421483	49.96	664619	48.12



4f

	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	14.496	855334	6.16	37219	7.30
2	15.707	13031976	93.84	472561	92.70


7.946
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7.686
7.435
7.414
7.270
6.885
6.882
6.860
6.831
6.811
6.002
5.299
3.839
3.802
2.951
2.938
2.916
2.903
2.820
2.798
2.786
2.763
1.096
1.079
0.000
O
O
O
Cl
COOMe
4g
10
9
8
7
6
5
4
3
2
1
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1.36
1.41
3.00
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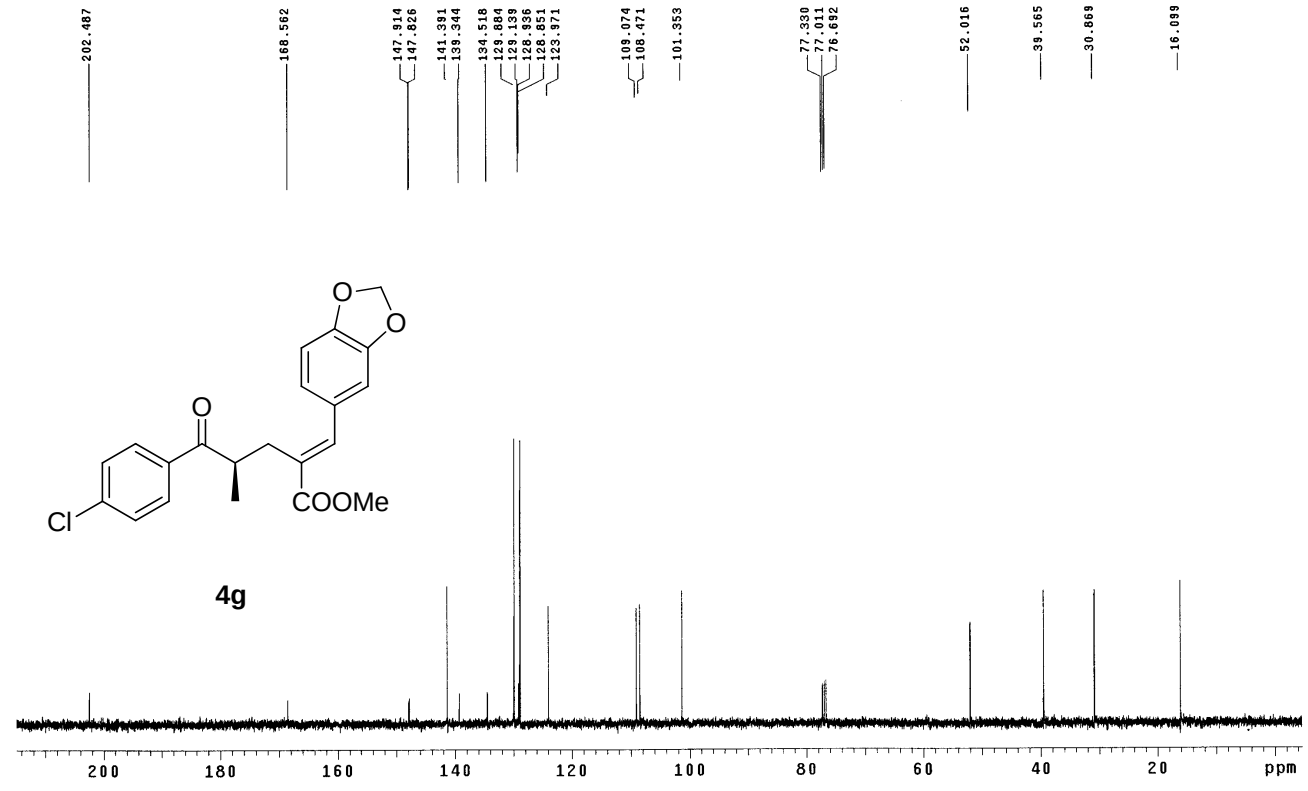
0.77
2.25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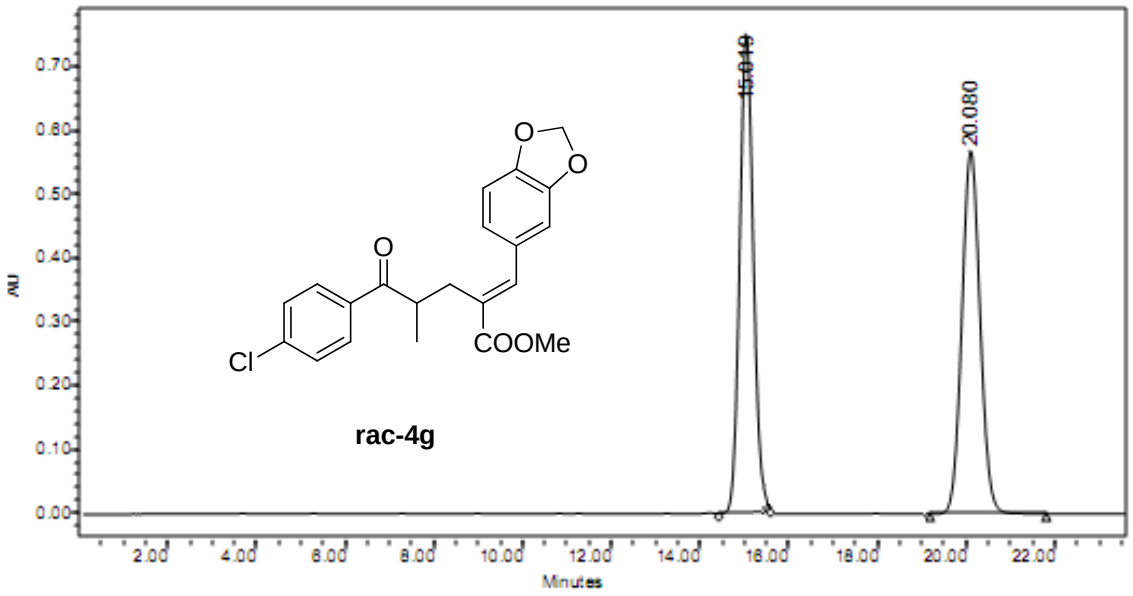
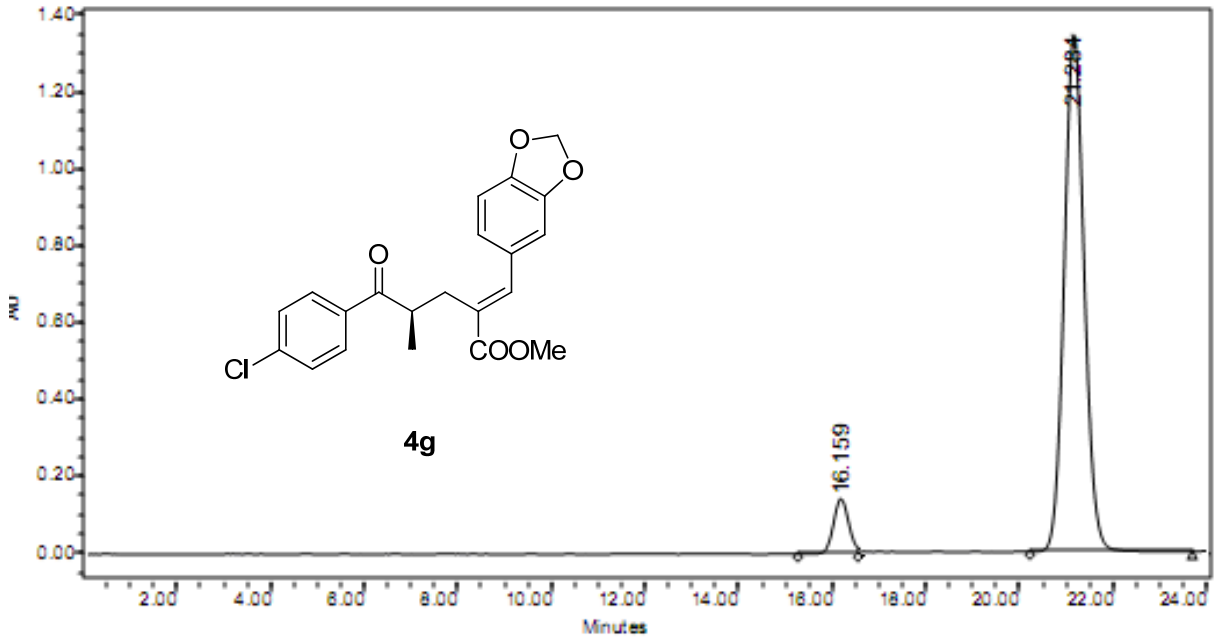
128.936
128.851
123.971
109.074
108.471
101.353
77.330
77.011
76.692
52.016
39.565
30.869
16.089
O
O
O
Cl
COOMe
4g
200
180
160
140
120
100
80
60
40
20
ppm



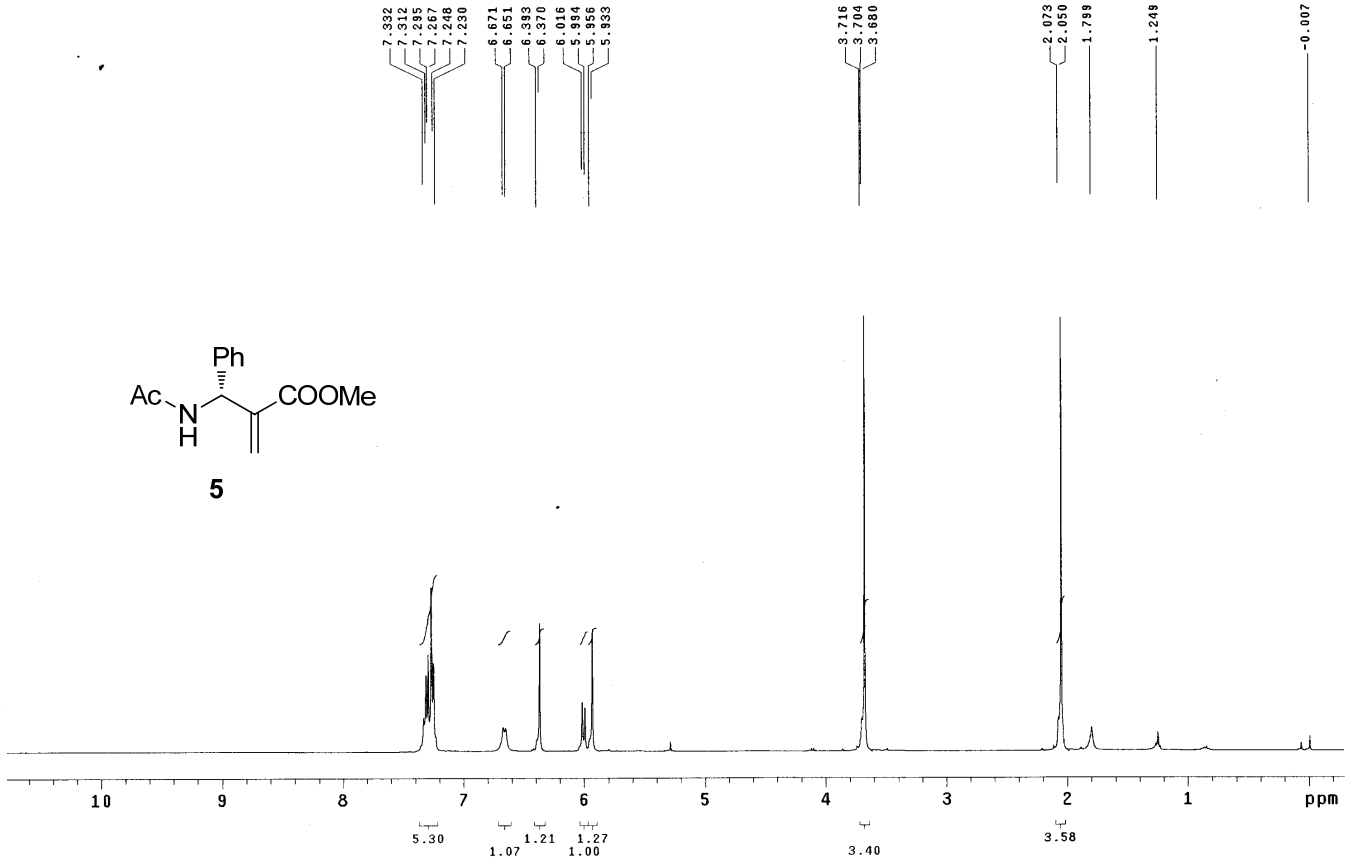
rac-4g

	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	15.019	16130745	49.87	751785	56.93
2	20.080	16214630	50.13	568743	43.07



4g

	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	16.159	3316567	7.66	143369	9.64
2	21.284	39982744	92.34	1344606	90.36


7.332
7.312
7.295
7.267
7.248
7.230
6.671
6.651
6.393
6.370
6.016
5.984
5.956
5.933
3.716
3.704
3.680
2.073
2.050
1.799
1.249
-0.007
Ph
Ac
N
H
COOMe
5
10
9
8
7
6
5
4
3
2
1
ppm
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1.21
1.27
1.00
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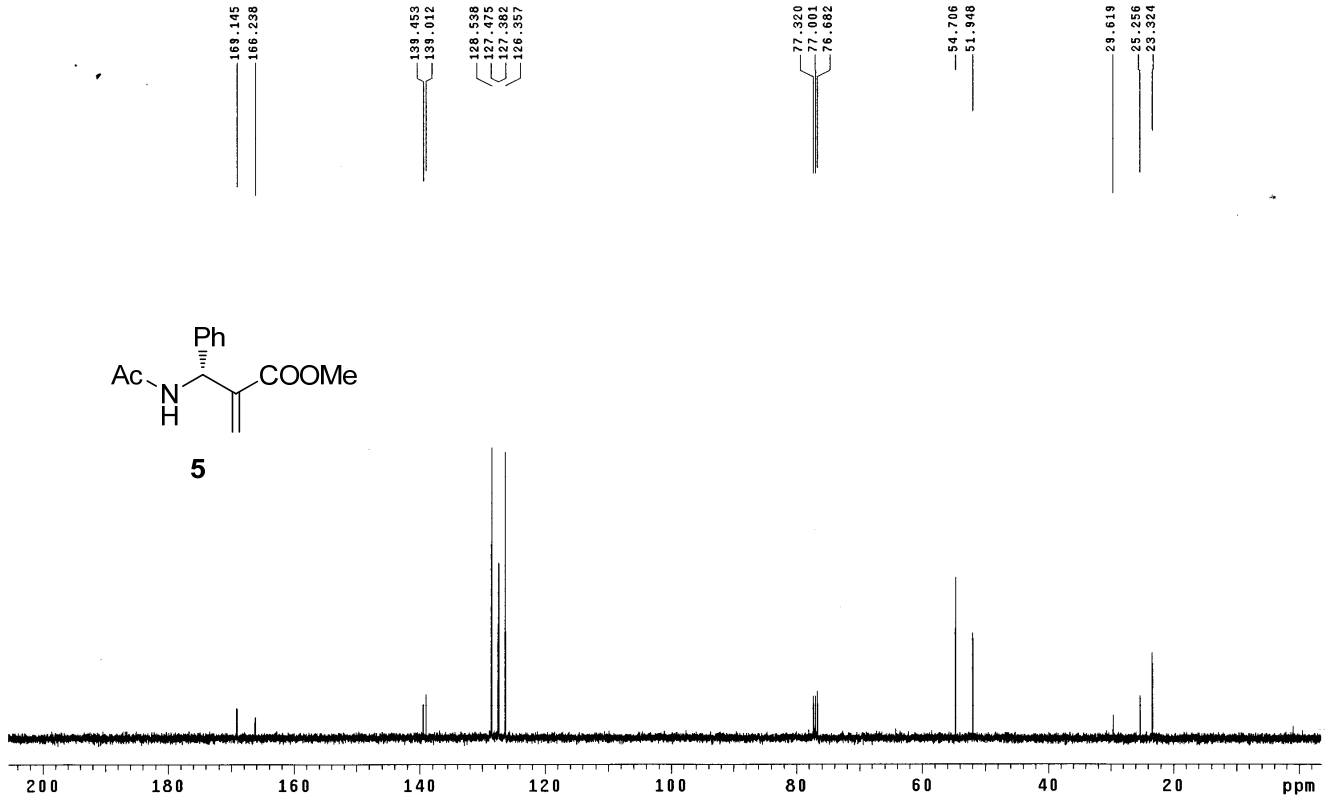
3.58


169.145
166.238
139.453
139.012
128.538
127.475
127.382
126.357
77.320
77.001
76.682
54.706
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29.619
25.256
23.324
Ph
Ac
N
H
COOMe
5
200
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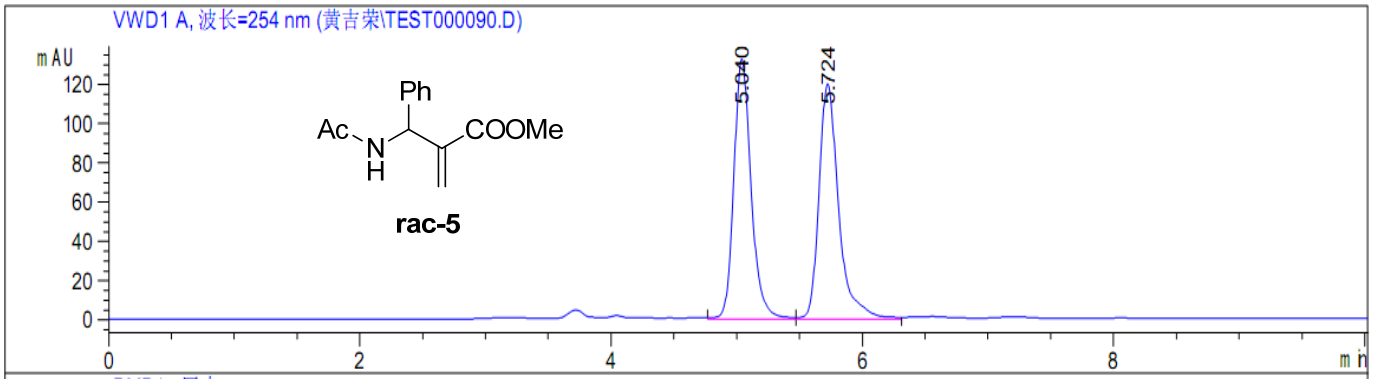
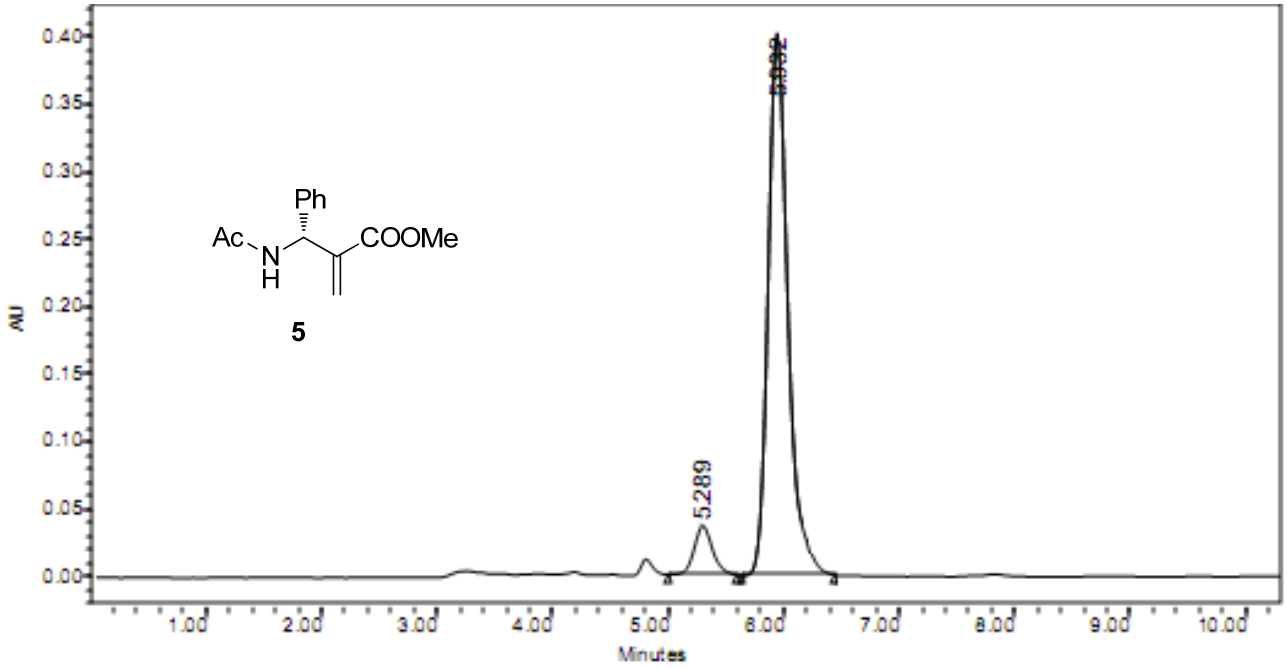
140
120
100
80
60
40
20
ppm



信号 1: VWD1 A, 波长=254 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	5.040	VV	0.1470	1272.63318	132.46182	49.2461
2	5.724	VV	0.1656	1311.59741	119.81437	50.7539



	RT (min)	Area (V*sec)	% Area	Height (V)	% Height
1	5.289	402668	7.59	36321	8.30
2	5.932	4904401	92.41	401160	91.70