Supplementary Material (ESI) for Organic & Biomolecular Chemistry

Organocatalytic Peroxy-Asymmetric Allylic Alkylation

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Supplementary Information

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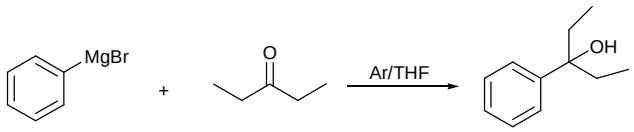
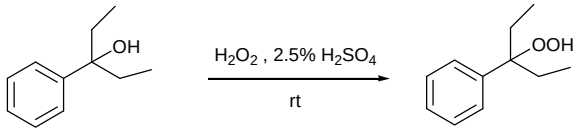
1. General methods

NMR data were obtained for ^{1}H at 400 MHz and for ^{13}C at 50 or 100 MHz. Chemical shifts were given in parts per million (δ) from tetramethylsilane with the solvent resonance as the internal standard in $CDCl_3$ solution. ESI HRMS was recorded on a Bruker Apex-2. In each case, enantiomeric ratio was determined by HPLC analysis on chiral column in comparison with authentic racemates, using a Daicel Chiralpak IC Column (250 x 4.6 mm), Chiralpak OD Column (250 x 4.6 mm) or Chiralpak AD Column (250 x 4.6 mm). UV detection was monitored at 220 nm or 254 nm. Optical rotation data were examined in $CHCl_3$ or MeOH solution at 20 °C. Column chromatography was performed on silica gel (200-300 mesh) eluting with ethyl acetate and petroleum ether. TLC was performed on glass-backed silica plates. UV light and I_2 were used to visualize products. All chemicals were used without purification as commercially available unless otherwise noted. THF, ethyl acetate, petroleum ether, methylene chloride (CH_2Cl_2) and carbon tetrachloride (CCl_4) were distilled before use.

2 Synthesis of hydroperoxyalkane 1b



To a mixture of Mg (3.4 g, 142 mmol) in 20 mL dry THF was added dropwise bromobenzene (15 mL, 142 mmol) in dry THF (60 mL) under argon atmosphere. The mixture was heated at 50 ^{o}C for 3 h. Then a solution of 3-pentanone (5 mL, 47 mmol) in dry THF (30 mL) was added dropwise at 0 ^{o}C. After 30 min the mixture was warmed to rt and stirred overnight. The solution was quenched with aq NH_4Cl and extracted three times with ethyl acetate. The organic layers were combined and washed with brine, dried (Na_2SO_4), filtered and concentrated. Flash chromatography on silica gel (ethyl acetate/petroleum ether) gave the 3-phenylpentan-3-ol as colorless oil (4.7 g, 61% yield).

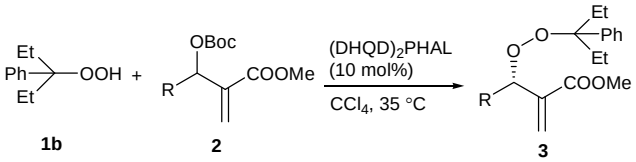


1b

A solution of 3-phenylpentan-3-ol (328 mg, 2 mmol) was added dropwise to the stirred mixture of 30% hydrogen peroxide (150 mL) and 2.5% (w/v) sulfuric acid (15 mL). After stirring for 4 h at room temperature, the reaction mixture was extracted with DCM (3 × 50 mL), washed with water,

dried over sodium sulfate, filtered, and concentrated. Flash chromatography on silica gel (ethyl acetate/petroleum ether) gave the product **1b** as yellow oil (300 mg, 83% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.48-7.42 (m, 4H), 7.37-7.32 (m, 1H), 2.04-1.90 (m, 4H), 0.88 (t, *J* = 7.2 Hz, 6H).

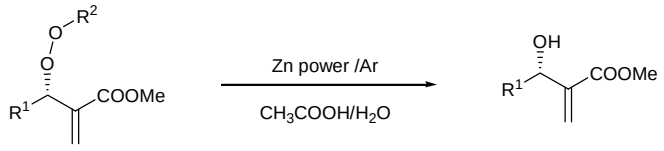
3. General procedure for the peroxy-AAA reaction



1b **2** **3**

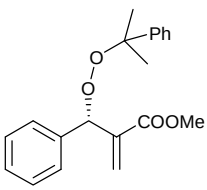
Hydroperoxyalkane **1b** (22 mg, 0.12 mmol)**,** MBH carbonate **2** (0.1 mmol), catalyst $(DHQD)_2PHAL$ (7.8 mg, 0.01 mmol) in CCl_4 (0.4 mL) were stirred at 35 °C. After completion the solvent was removed and flash chromatography on silica gel (EtOAc/petroleum ether) gave the peroxide **3**.

4. General procedure for the reduction of compound 3

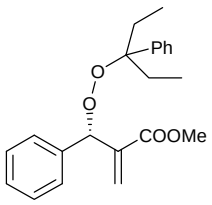


3 **4**

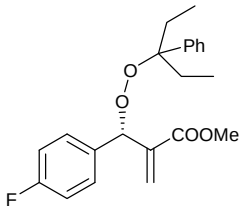
Compound **3** (0.1 mmol) and zinc powder (260 mg, 4 mmol) were stirred in a mixture solvents of $AcOH/H_2O$ (1:1) at 25 ^oC for 3 h under argon atmosphere. Then the solid was filtered and the filtrate was extracted with DCM three times. The organic layers were combined and washed with brine, dried over Na_2SO_4, filtered. Concentration and flash chromatography on silica gel (ethyl acetate/petroleum ether) gave α-methylene-β-hydroxy ester **4**.



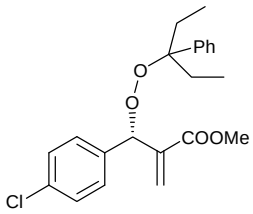
3a, 76% yield; $[\alpha]_D^{20}$ = +31.0 (*c* = 0.40 in $CHCl_3$); 91% ee, determined by HPLC analysis [Daicel chiralpak OD, *n*-hexane/*i*-PrOH = 99/1, 1.0 mL/min, λ = 254 nm, t (major) = 5.69 min, t (minor) = 6.60 min]; ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.46-7.44 (m, 2H), 7.35-7.31 (m, 2H), 7.28-7.24 (m, 4H), 7.18-7.15 (m, 2H), 6.47 (t, *J* = 0.8 Hz, 1H), 6.02 (t, *J* = 1.2 Hz, 1H), 5.84 (s, 1H), 3.67 (s, 3H), 1.59-1.57 (m, 6H); ^{13}C NMR (50 MHz, $CDCl_3$): δ (ppm) 166.1, 145.3, 138.6, 137.2, 128.3, 128.2, 127.9, 127.0, 126.8, 125.6, 83.3, 51.8, 26.7, 26.5; ESI-HRMS: calcd. for $C_{22}H_{26}O_4$+Na 349.1416, found 349.1386.



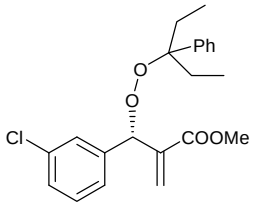
3b, 79% yield; $[\alpha]_D^{20}$ = +64.8 (*c* = 0.25 in $CHCl_3$); 93% ee, determined by HPLC analysis [Daicel chiralpak OD, *n*-hexane/*i*-PrOH = 98/2, 1.0 mL/min, λ = 254 nm, t (major) = 4.78 min, t (minor) = 6.80 min]; ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.31-7.28 (m, 4H), 7.26-7.22 (m, 3H), 7.20-7.17 (m, 3H), 6.50 (t, *J* = 0.8 Hz, 1H), 6.04 (t, *J* = 1.2 Hz, 1H), 5.91 (s, 1H), 3.69 (s, 3H), 2.01-1.91 (m, 3H), 1.89-1.82 (m, 1H), 0.77 (t, *J* = 7.2 Hz, 3H), 0.72 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (50 MHz, $CDCl_3$): δ (ppm) 166.2, 142.9, 138.8, 137.3, 128.3, 128.2, 128.2, 127.7, 126.9, 126.6, 126.2, 88.3, 82.9, 51.8, 28.5, 28.1, 7.9; ESI-HRMS: calcd. for $C_{22}H_{26}O_4$+Na 377.1729, found 377.1729.



3c, 69% yield; $[\alpha]_D^{20}$ = +33.2 (*c* = 0.75 in MeOH)[1]; 90% ee, determined by HPLC analysis after converted to the corresponding α-methylene-β-hydroxy ester [Daicel chiralpak OD, *n*-hexane/*i*-PrOH = 98/2, 1.0 mL/min, λ = 254 nm, t (major) = 30.65 min, t (minor) = 27.27 min]; ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.33-7.30 (m, 4H), 7.25-7.22 (m, 1H), 7.14-7.10 (m, 2H), 6.95 (t, *J* = 8.4 Hz, 2H), 6.49 (s, 1H), 6.03 (s, 1H), 5.85 (s, 1H), 3.68 (s, 3H), 1.97-1.81 (m, 4H), 0.76 (t, *J* = 7.2 Hz, 3H), 0.71 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (50 MHz, $CDCl_3$): δ (ppm) 166.0, 165.1, 160.2, 142.9, 138.6, 133.2, 130.0, 129.8, 127.7, 126.7, 126.6, 126.1, 115.3, 114.9, 88.4, 82.2, 51.8, 28.4, 27.8, 7.8, 7.7; ESI-HRMS: calcd. for $C_{22}H_{25}FO_4$+Na 395.1635, found 395.1624.

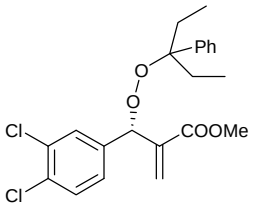


3d, 71% yield; $[\alpha]_D^{20}$ = +25.4 (*c* = 1.00 in MeOH)[1]; 89% ee, determined by HPLC analysis after converted to the corresponding α-methylene-β-hydroxy ester [Daicel chiralpak OD, *n*-hexane/*i*-PrOH = 98/2, 1.0 mL/min, λ = 254 nm, t (major) = 30.41 min, t (minor) = 27.05 min]; ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.33-7.27 (m, 4H), 7.25-7.21 (m, 3H), 7.09 (d, *J* = 8.4 Hz, 2H), 6.47 (s, 1H), 6.00 (s, 1H), 5.83 (s, 1H), 3.68 (s, 3H), 1.96-1.80 (m, 4H), 0.76 (t, *J* = 7.6 Hz, 3H), 0.70 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 166.0, 142.8, 138.5, 136.0, 134.1, 129.5, 128.4, 127.8, 127.0, 126.7, 126.1, 88.5, 82.2, 51.9, 28.3, 27.8, 7.9, 7.8; ESI-HRMS: calcd. for $C_{22}H_{25}ClO_4$+Na 411.1339, found 411.1387.

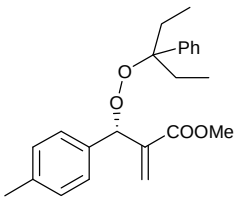


3e, 71% yield; $[\alpha]_D^{20}$ = +26.9 (*c* = 0.70 in MeOH)[1]; 83% ee, determined by HPLC analysis after converted to the corresponding α-methylene-β-hydroxy ester [Daicel chiralcel OD, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t (major) = 9.62 min, t (minor) = 8.92 min]; ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.34-7.29 (m, 4H), 7.26-7.20 (m, 3H), 7.13 (s, 1H), 7.07 (d, *J* = 6.8 Hz, 1H), 6.49

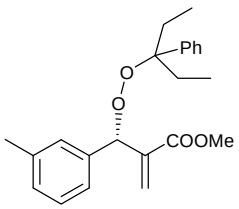
(s, 1H), 5.98 (s, 1H), 5.85 (s, 1H), 3.71 (s, 3H), 1.95 (q, *J* = 7.6 Hz, 2H), 1.86 (q, *J* = 7.2 Hz, 2H), 0.77 (t, *J* = 7.6 Hz, 3H), 0.71 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR (50 MHz, $CDCl_3$): δ (ppm) 165.9, 142.8, 139.6, 138.2, 134.1, 129.4, 128.4, 128.1, 127.8, 127.4, 126.7, 126.3, 126.1, 88.5, 82.1, 51.9, 28.3, 27.7, 7.8, 7.7; ESI-HRMS: calcd. for $C_{22}H_{25}ClO_4$+Na 411.1339, found 411.1312.



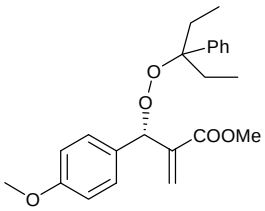
3f, 73% yield; $[\alpha]_D^{20}$ = +32.4 (*c* = 0.90 in MeOH)[1]; 88% ee, determined by HPLC analysis after converted to the corresponding α-methylene-β-hydroxy ester [Daicel chiralpak IC, *n*-hexane/*i*-PrOH = 95/5, 1.0 mL/min, λ = 254 nm, t (major) = 11.84 min, t (minor) = 16.80 min]; ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.36-7.26 (m, 6H), 7.20 (d, *J* = 2.0 Hz, 1H), 7.00 (dd, *J* = 2.0 Hz, 8.4 Hz, 1H), 6.48 (s, 1H), 5.97 (t, *J* = 1.2 Hz, 1H), 5.79 (s, 1H), 3.71 (s, 3H), 1.96 (q, *J* = 7.2 Hz, 2H), 1.87 (q, *J* = 6.8 Hz, 2H), 0.78 (t, *J* = 7.2 Hz, 3H), 0.70 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR (50 MHz, $CDCl_3$): δ (ppm) 165.7, 142.7, 137.9, 132.3, 130.2, 129.9, 127.8, 127.4, 127.3, 126.8, 126.1, 88.6, 81.6, 52.0, 28.2, 27.4, 7.8, 7.7; ESI-HRMS: calcd. for $C_{22}H_{24}Cl_2O_4$+Na 445.0949, found 445.0922.



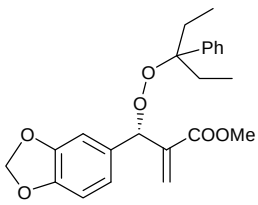
3g, 65% yield; $[\alpha]_D^{20}$ = +18.3 (*c* = 0.40 in MeOH)[1]; 86% ee, determined by HPLC analysis after converted to the corresponding α-methylene-β-hydroxy ester [Daicel chiralpak AD, *n*-hexane/*i*-PrOH = 95/5, 1.0 mL/min, λ = 254 nm, t (major) = 19.65 min, t (minor) = 20.87 min]; ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.31-7.30 (m, 4H), 7.25-7.22 (m, 1H), 7.09-7.04 (m, 4H), 6.50 (s, 1H), 6.07 (s, 1H), 5.87 (s, 1H), 3.68 (s, 3H), 2.30 (s, 3H), 1.99-1.89 (m, 3H), 1.89-1.81 (m, 1H), 0.77 (t, *J* = 7.2 Hz, 3H), 0.72 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (50 MHz, $CDCl_3$): δ (ppm) 166.3, 142.9, 138.8, 138.1, 134.2, 128.9, 128.2, 127.7, 126.5, 126.2, 88.3, 82.8, 51.8, 28.5, 28.0, 21.2, 7.9; ESI-HRMS: calcd. for $C_{23}H_{28}O_4$+Na 391.1885, found 391.1894.



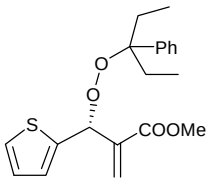
3h, 67% yield; $[\alpha]_D^{20}$ = +16.0 (*c* = 0.25 in $CHCl_3$); 92% ee, determined by HPLC analysis [Daicel chiralcel OD, *n*-hexane/*i*-PrOH = 99/1, 1.0 mL/min, λ = 254 nm, t (major) = 4.99 min, t (minor) = 5.79 min]; ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.31-7.29 (m, 4H), 7.25-7.22 (m, 1H), 7.16 (t, *J* = 7.6 Hz, 1H), 7.07 (d, *J* = 7.6 Hz, 1H), 6.97 (d, *J* = 7.6 Hz, 1H), 6.91 (s, 1H), 6.50 (s, 1H), 6.05 (s, 1H), 5.86 (s, 1H), 3.69 (s, 3H), 2.28 (s, 3H), 1.99-1.90 (m, 3H), 1.88-1.81 (m, 1H), 0.77 (t, *J* = 7.2 Hz, 3H), 0.71 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 166.3, 143.0, 138.8, 137.8, 137.1, 129.1, 128.9, 128.1, 127.7, 126.8, 126.6, 126.2, 125.3, 88.3, 83.0, 51.8, 28.5, 27.9, 21.4, 7.9, 7.8; ESI-HRMS: calcd. for $C_{23}H_{28}O_4$+Na 391.1885, found 391.1894.



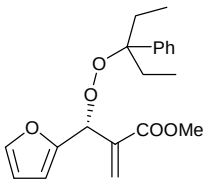
3i, 73% yield; $[\alpha]_D^{20}$ = +25.8 (*c* = 0.40 in $CHCl_3$); 93% ee, determined by HPLC analysis [Daicel chiralpak IC, *n*-hexane/*i*-PrOH = 99/1, 1.0 mL/min, λ = 254 nm, t (major) = 7.28 min, t (minor) = 6.77 min]; ^{1}H NMR (400 Hz, $CDCl_3$): δ (ppm) 7.32-7.31 (m, 4H), 7.25-7.24 (m, 1H), 7.08 (d *J* = 6.8 Hz, 2H), 6.80 (d, *J* = 6.4 Hz, 2H), 6.50 (s, 1H), 6.09 (t, *J* = 1.2 Hz, 1H), 5.84 (s, 1H), 3.77 (s, 3H), 3.68 (s, 3H), 1.99-1.90 (m, 3H), 1.88-1.81 (m, 1H), 0.78 (t, *J* = 7.2 Hz, 3H), 0.72 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 166.3, 159.7, 143.0, 138.9, 129.6, 127.7, 126.6, 126.3, 126.2, 125.5, 113.6, 88.2, 82.6, 55.2, 51.8, 28.5, 27.9, 7.9, 7.8; ESI-HRMS: calcd. for $C_{23}H_{28}O_5$+Na 407.1834, found 407.1835.



3j, 50% yield; $[\alpha]_D^{20}$ = +13 (*c* = 0.90 in $CHCl_3$); 89% ee, determined by HPLC analysis [Daicel chiralpak IC, *n*-hexane/*i*-PrOH = 99/1, 1.0 mL/min, λ = 254 nm, t (major) = 8.90 min, t (minor) = 7.79 min]; ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.39-7.37 (m, 1H), 7.31-7.30 (m, 4H), 7.27-7.23 (m, 1H), 6.71 (d, *J* = 8.0 Hz, 1H), 6.65 (dd, *J* = 8.0 Hz, 1.6 Hz, 1H), 6.60 (d, *J* = 1.6 Hz, 1H), 6.49 (s, 1H), 6.07 (t, *J* = 1.2 Hz, 1H), 5.92 (s, 2H), 5.79 (s,1H), 3.70 (s, 3H), 1.99-1.98 (m, 4H), 0.78 (t, *J* = 7.2 Hz, 3H), 0.72 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (50 MHz, $CDCl_3$): δ (ppm) 166.2, 147.5, 142.9, 138.7, 130.9, 128.3, 127.7, 126.6, 126.4, 126.2, 122.1, 108.7, 108.0, 101.1, 88.3, 82.7, 51.8, 28.4, 27.8, 7.9, 7.8; ESI-HRMS: calcd. for $C_{23}H_{26}O_6$+Na 421.1627, found 421.1632.

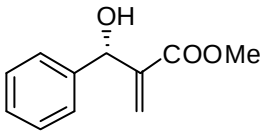


3k, 53% yield; $[\alpha]_D^{20}$ = +35.8 (*c* = 0.50 in MeOH)[1]; 92% ee, determined by HPLC analysis after converted to the corresponding α-methylene-β-hydroxy ester [Daicel chiralpak IC, *n*-hexane/*i*-PrOH = 95/5, 1.0 mL/min, λ = 254 nm, t (major) = 22.18 min, t (minor) = 32.73 min]; ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.35-7.30 (m, 4H), 7.27-7.23 (m, 2H), 6.92 (dd, *J* = 3.6 Hz, 4.8 Hz, 1H), 6.86 (d, *J* = 3.2 Hz, 1H), 6.54 (s, 1H), 6.17 (s, 1H), 6.10 (s, 1H), 3.74 (s, 3H), 2.01-1.85 (m, 4H), 0.80-0.73 (m, 6H); ^{13}C NMR (50 MHz, $CDCl_3$): δ (ppm) 166.0, 142.8, 140.1, 138.8, 127.8, 127.1, 126.8, 126.6, 126.5, 126.1, 88.5, 77.8, 51.9, 28.4, 28.1, 7.8; ESI-HRMS: calcd. for $C_{20}H_{24}O_4S$+Na 383.1293, found 383.1310.



3l, 62% yield; $[\alpha]_D^{20}$ = +29.3 (*c* = 0.30 in $CHCl_3$); 91% ee, determined by HPLC analysis [Daicel chiralpak IC, *n*-hexane/*i*-PrOH = 98/2, 1.0 mL/min, λ = 254 nm, t (major) = 7.28 min, t (minor) = 9.09 min]; ^{1}H NMR (400 MHz, $CDCl_3$): δ

(ppm) 7.38-7.37 (m, 1H), 7.31-7.28 (m, 4H), 7.26-7.21 (m, 1H), 6.53 (s, 1H), 6.30 (dd, *J* = 2.0 Hz, 3.2 Hz, 1H), 6.22 (d, *J* = 3.6 Hz, 1H), 6.15 (t, *J* = 2.8 Hz, 1H), 6.00 (s, 1H), 3.74 (s, 3H), 1.96 (q, *J* = 7.2 Hz, 2H), 1.90 (q, *J* = 7.2 Hz, 2H), 0.78-0.72 (m, 6H); ^{13}C NMR (50 MHz, $CDCl_3$): δ (ppm) 165.9, 150.5, 143.1, 142.7, 136.6, 127.7, 127.5, 126.5, 126.0, 110.4, 110.1, 88.6, 76.0, 51.9, 28.7, 28.4, 7.8; ESI-HRMS: calcd. for $C_{20}H_{24}O_5$+Na 367.1521, found 367.1539.



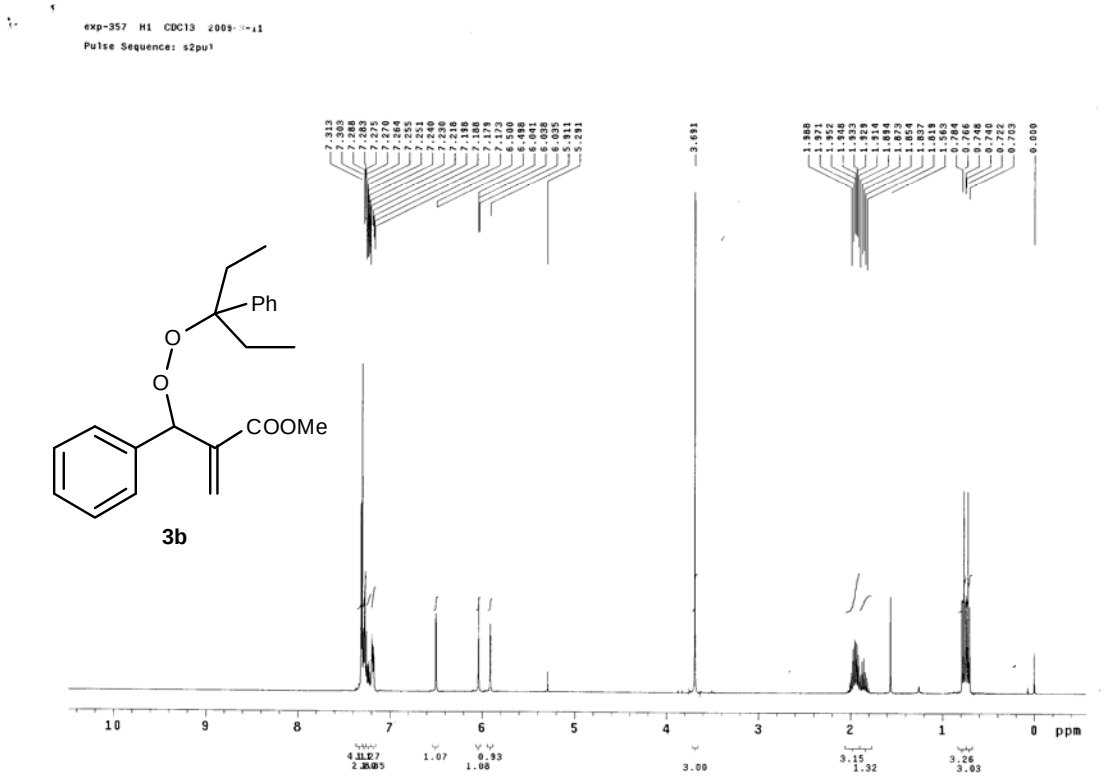
4b, 93% yield; $[\alpha]_D^{20}$ = +82.9 (*c* = 0.6 in MeOH) {lit.:[2] $[\alpha]_D^{22}$ = +85.5 (c = 1.11 in MeOH, 84% ee)}; 93% ee, determined by HPLC analysis [Daicel chiralpak IC, *n*-hexane/*i*-PrOH = 95/5, 1.0 mL/min, λ = 220 nm, t (major) = 19.96 min, t (minor) = 35.15 min]; ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.38-7.32 (m, 4H), 7.29-7.25 (m, 1H), 6.33 (s, 1H), 5.82 (s, 1H), 5.56 (d, *J* = 5.2 Hz, 1H), 3.71 (s, 3H), 3.03 (d, *J* = 5.6 Hz, 1H); ^{13}C NMR (50 MHz, $CDCl_3$): δ (ppm) 166.8, 142.0, 141.3, 128.4, 127.8, 126.6, 126.1, 73.2, 51.9.

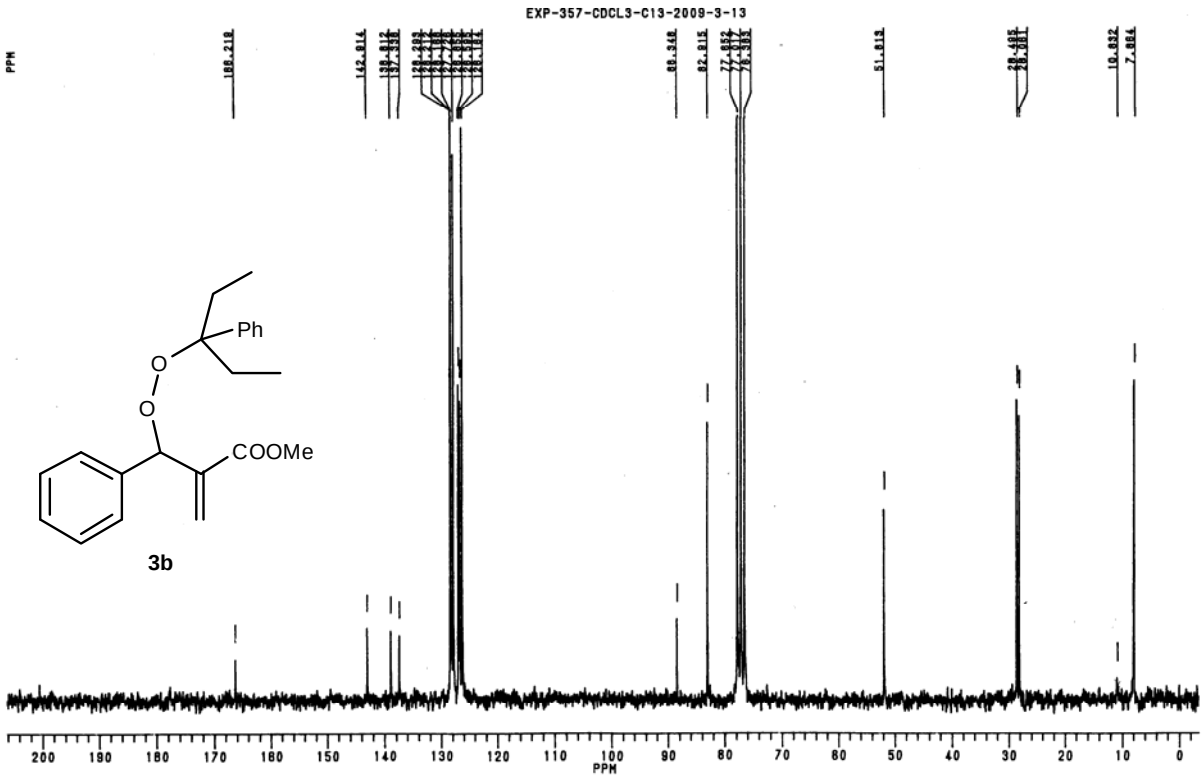
Notes and references

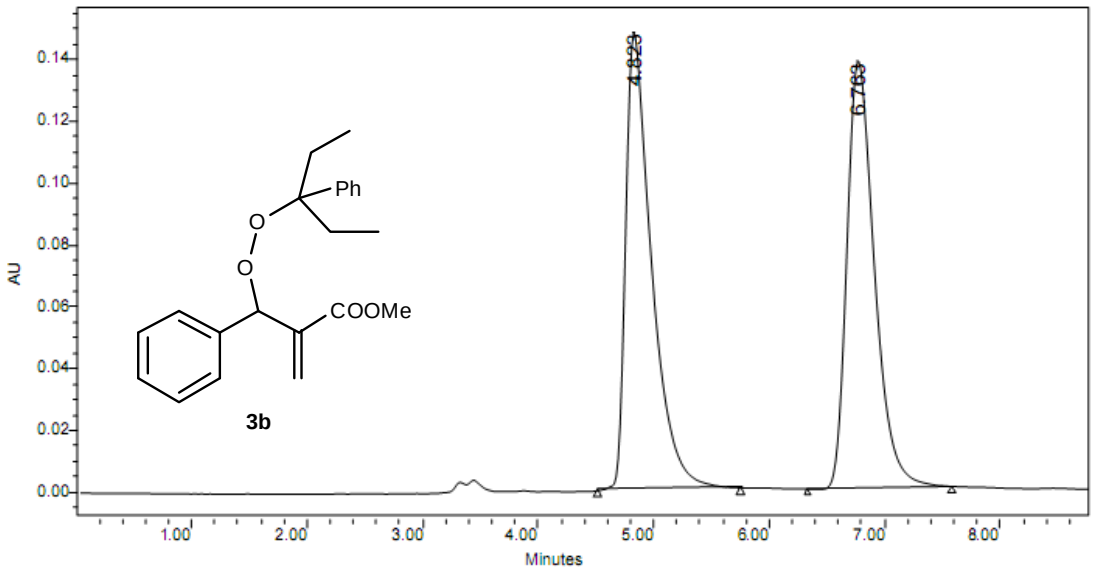
(1) The optical rotation was related to the corresponding α-methylene-β-hydroxy ester.

(2) J.-N. Kim, H.-J. Lee, J.-H. Gong, *Tetrahedron Lett.,* 2002, **43**, 9141.

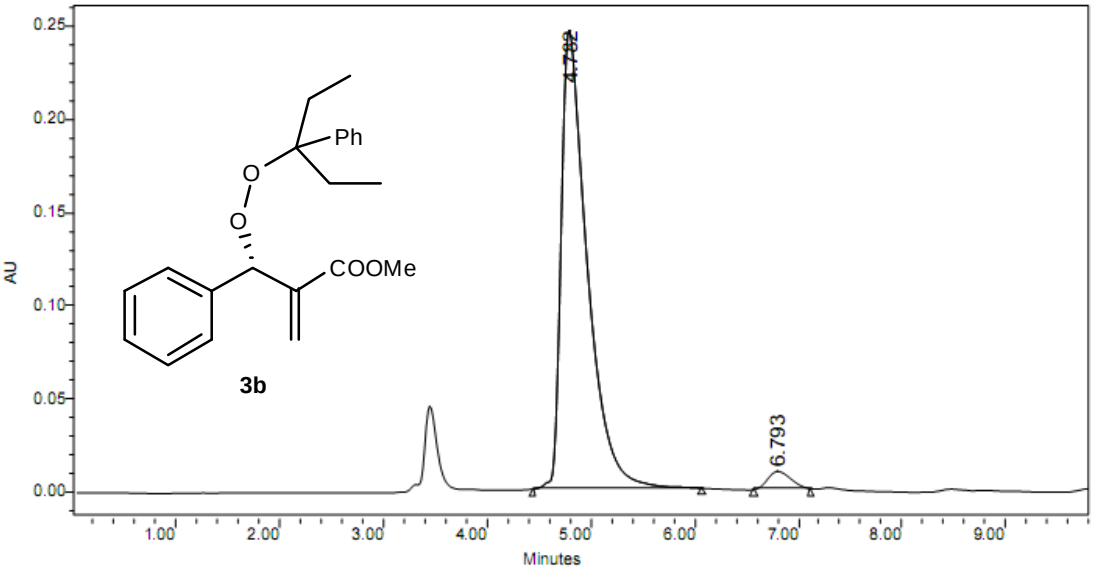
5. NMR and HPLC spectra



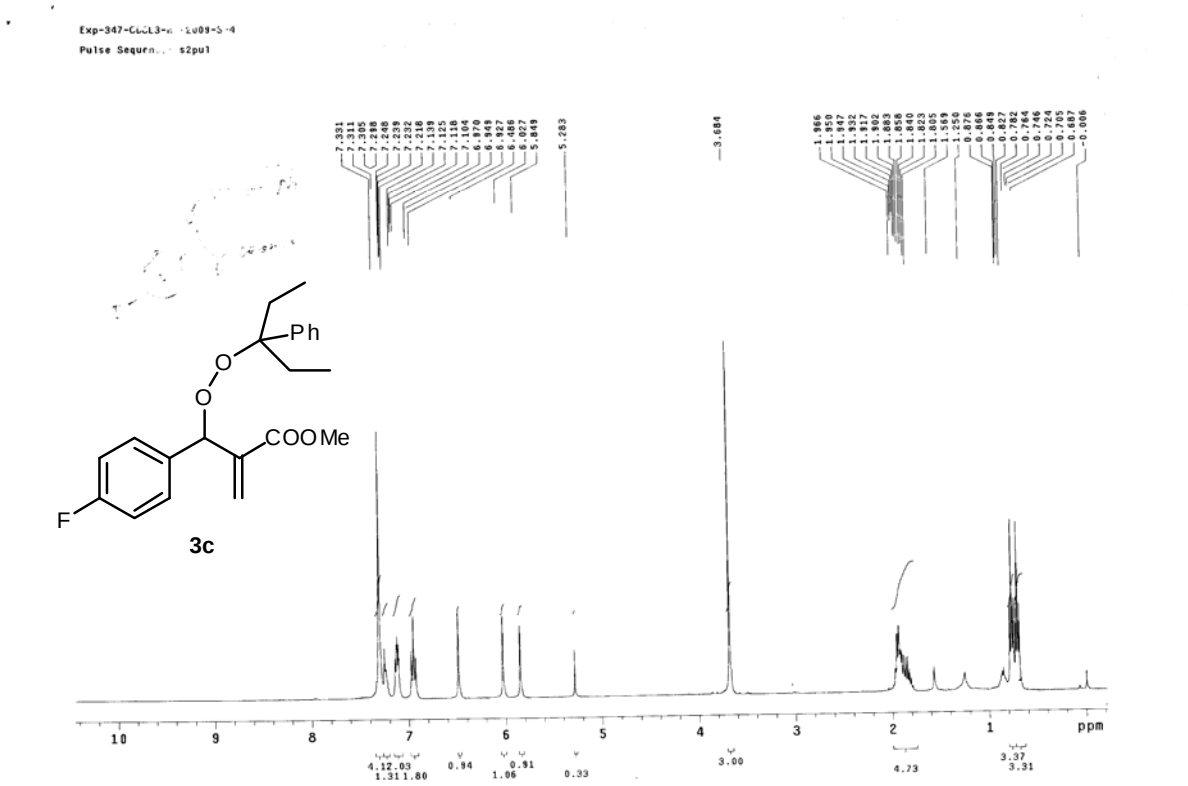




	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	4.823	2312065	50.24	148412	51.80
2	6.763	2290118	49.76	138082	48.20



	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	4.782	4302909	96.64	246153	96.24
2	6.793	149467	3.36	9610	3.76


Ph
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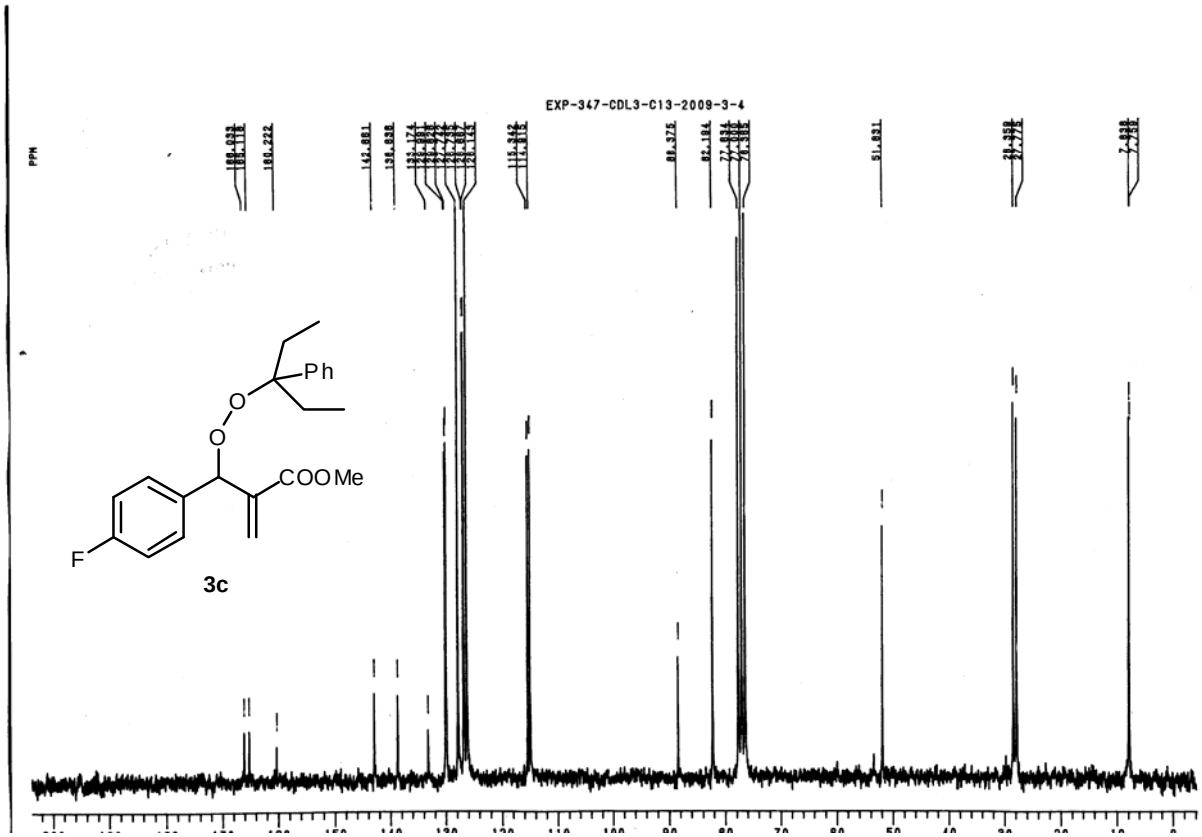
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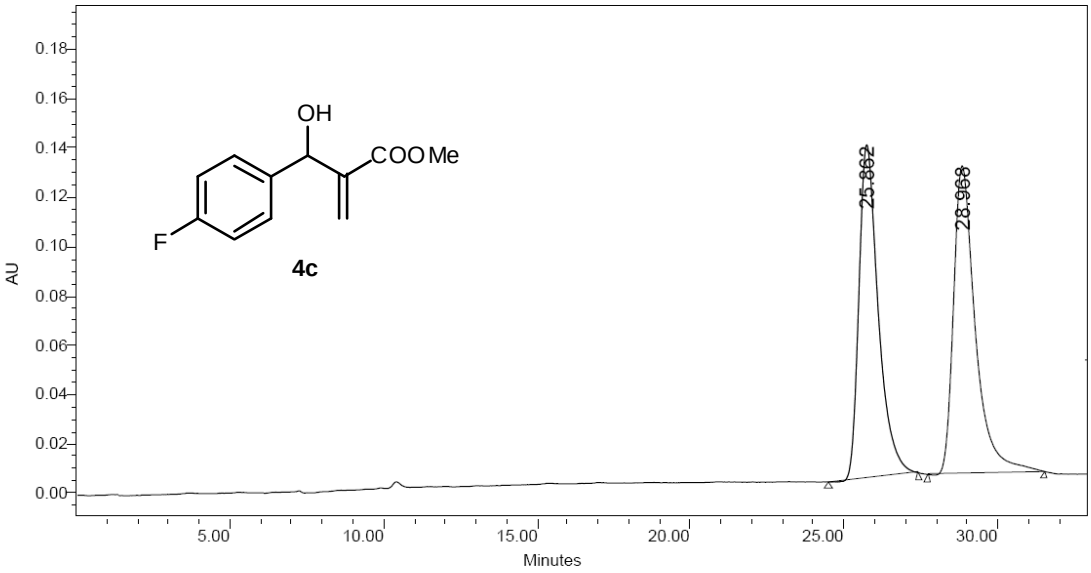
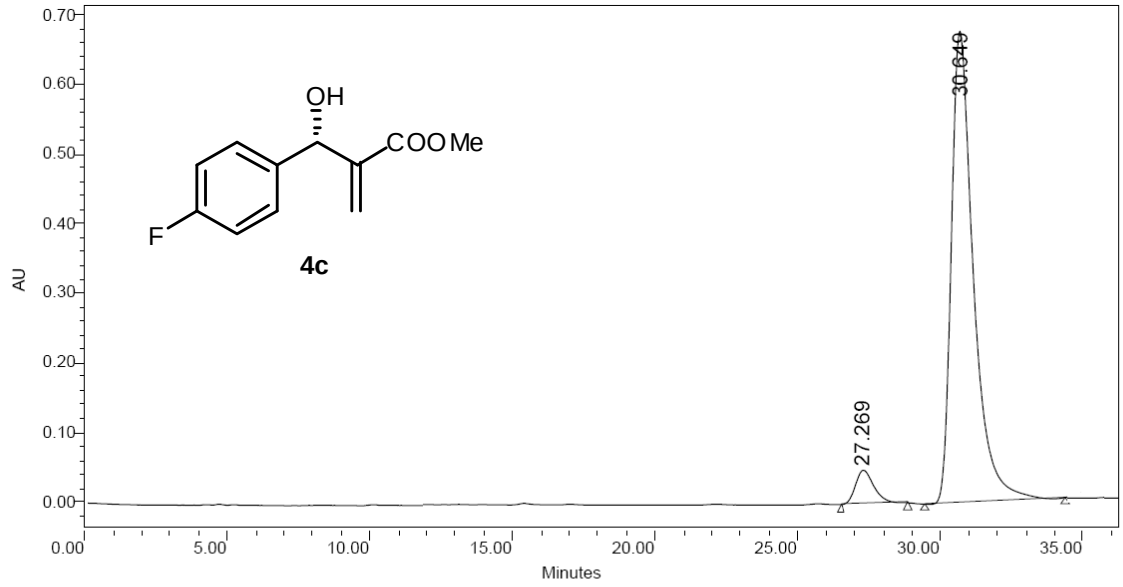
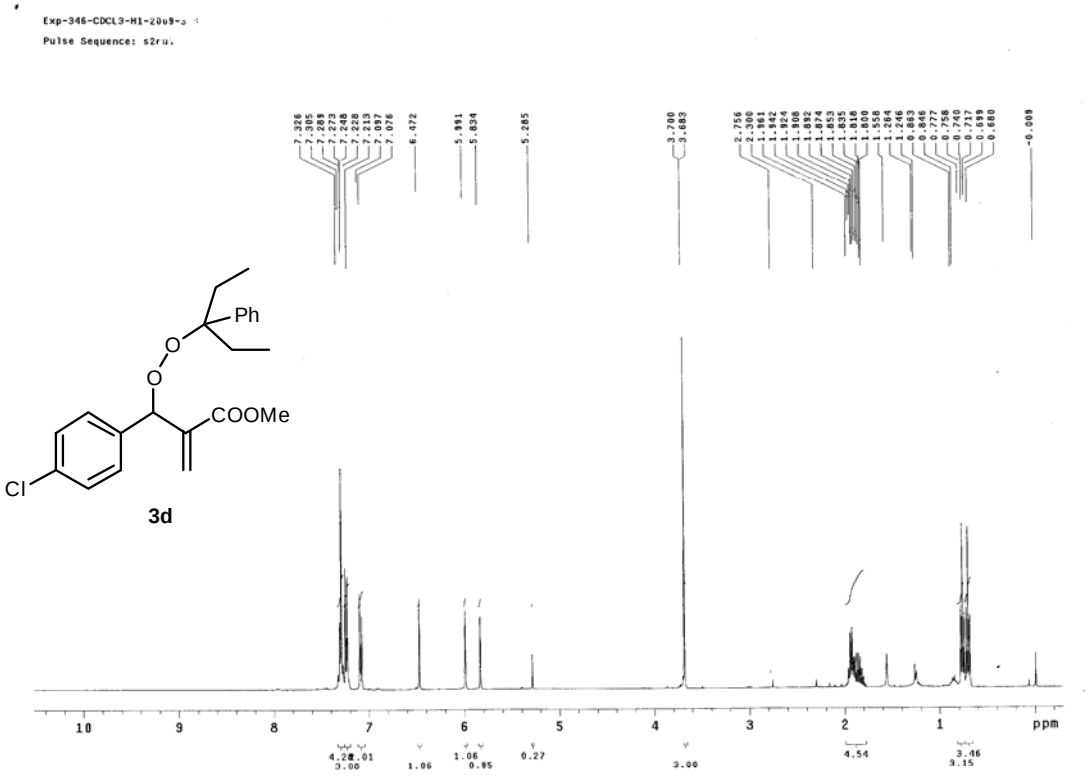


	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	25.862	5947863	49.04	135301	51.99
2	28.968	6180173	50.96	124935	48.01



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	27.269	2197050	5.70	48433	6.68
2	30.649	36362805	94.30	676620	93.32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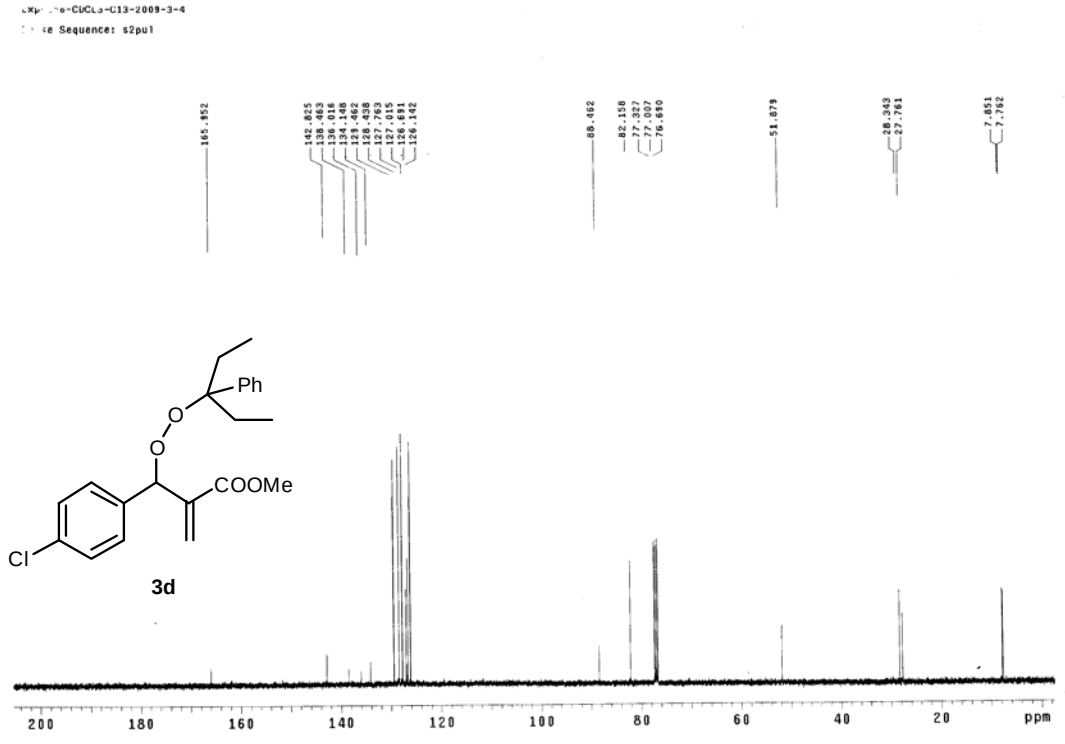
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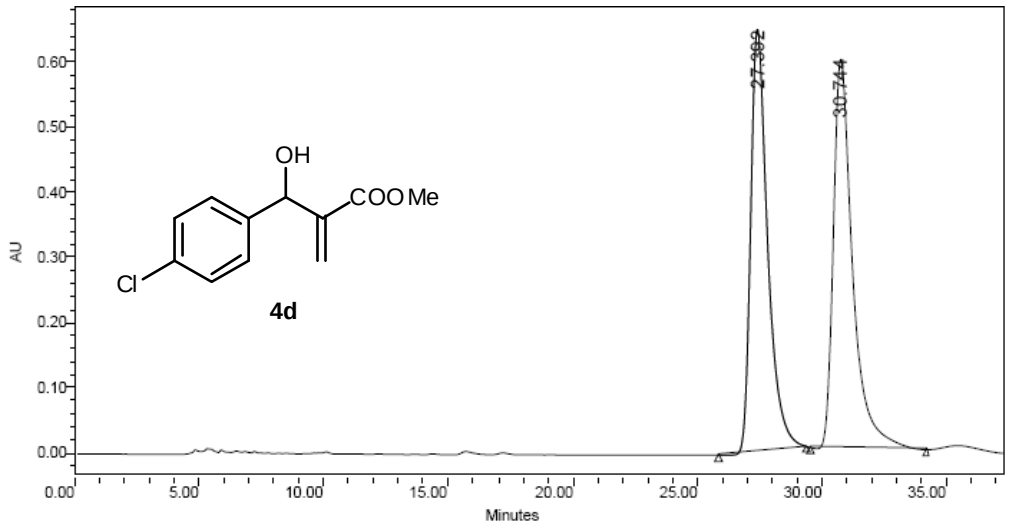
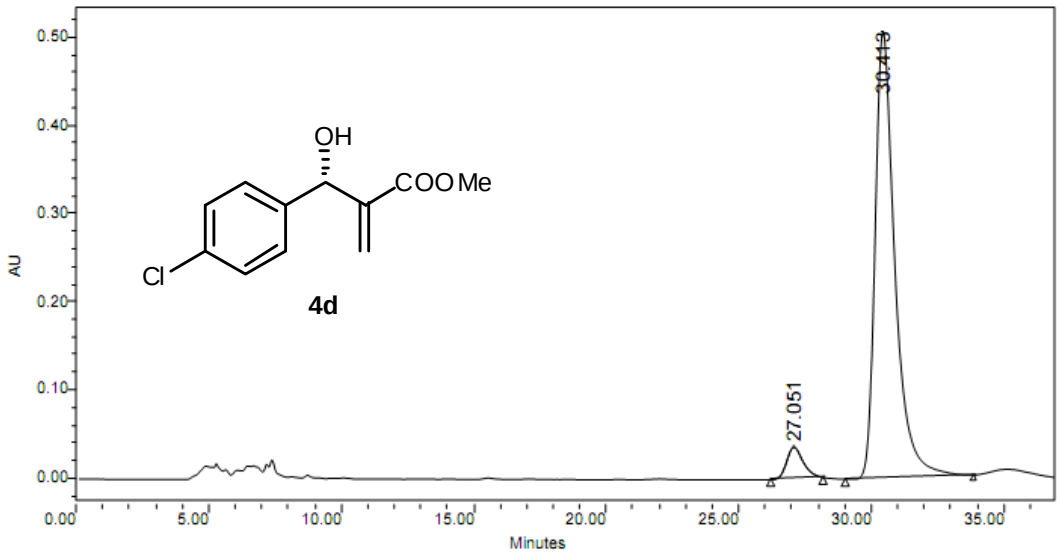
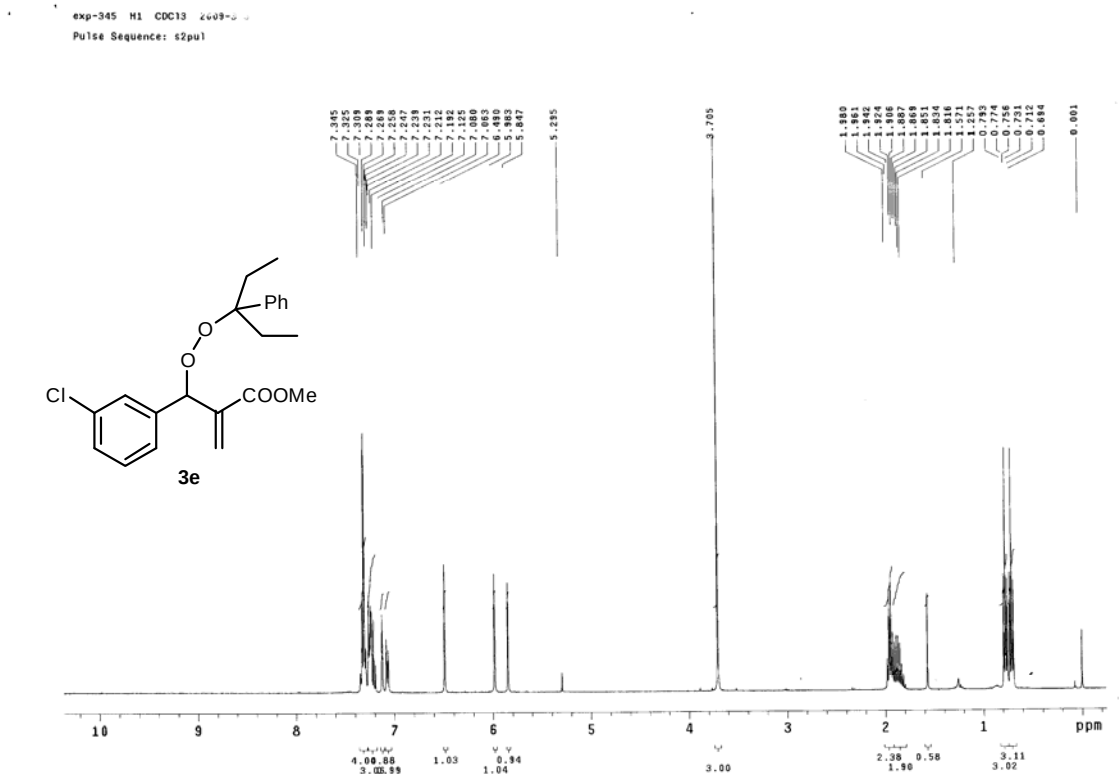


	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	27.392	30803596	48.82	648975	52.05
2	30.744	32286911	51.18	597867	47.95



	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	27.051	1554152	5.60	35677	6.58
2	30.413	26199785	94.40	506235	93.42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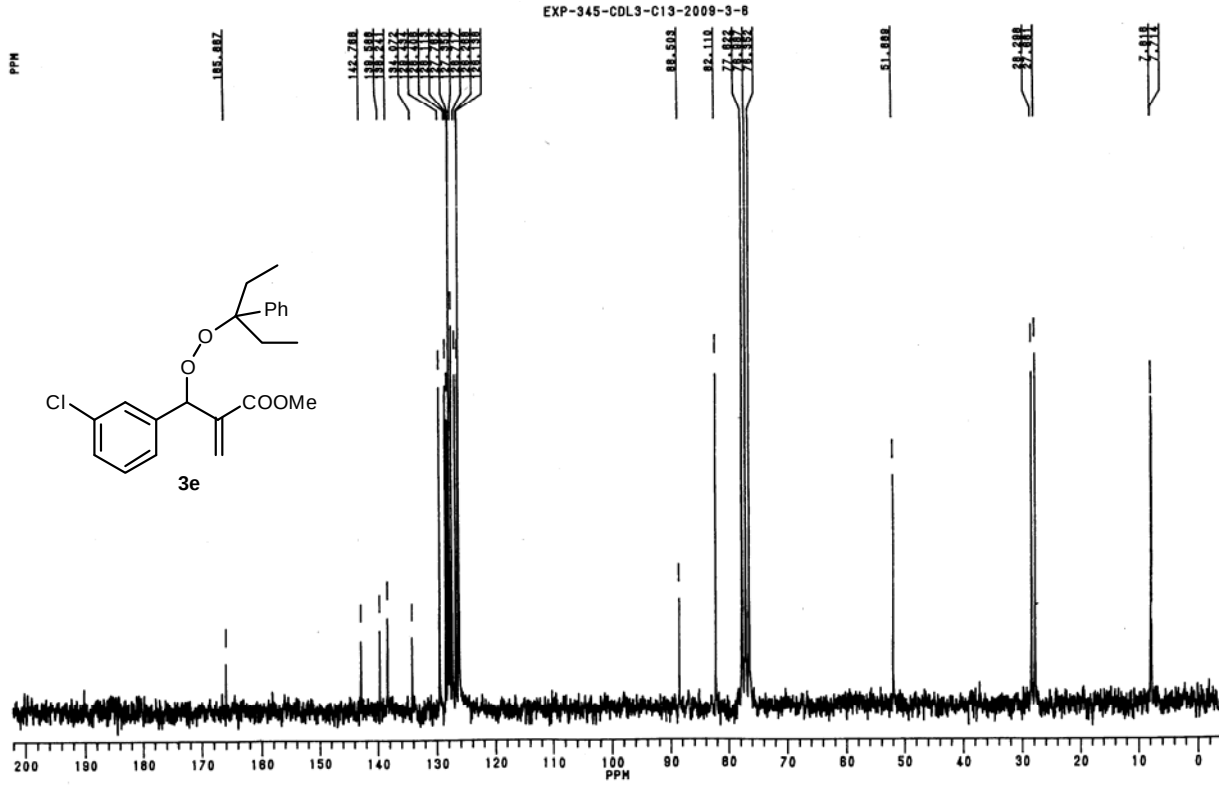
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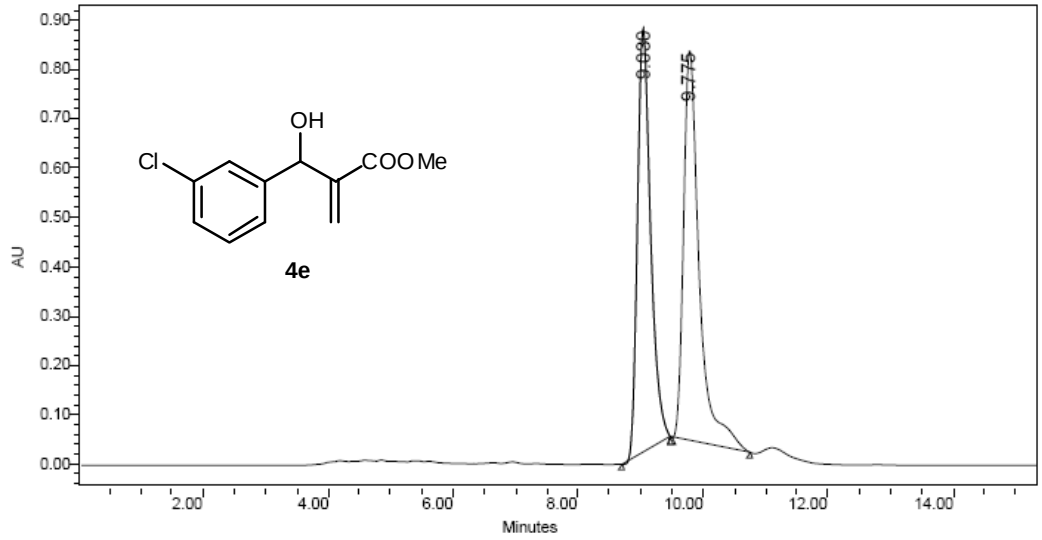
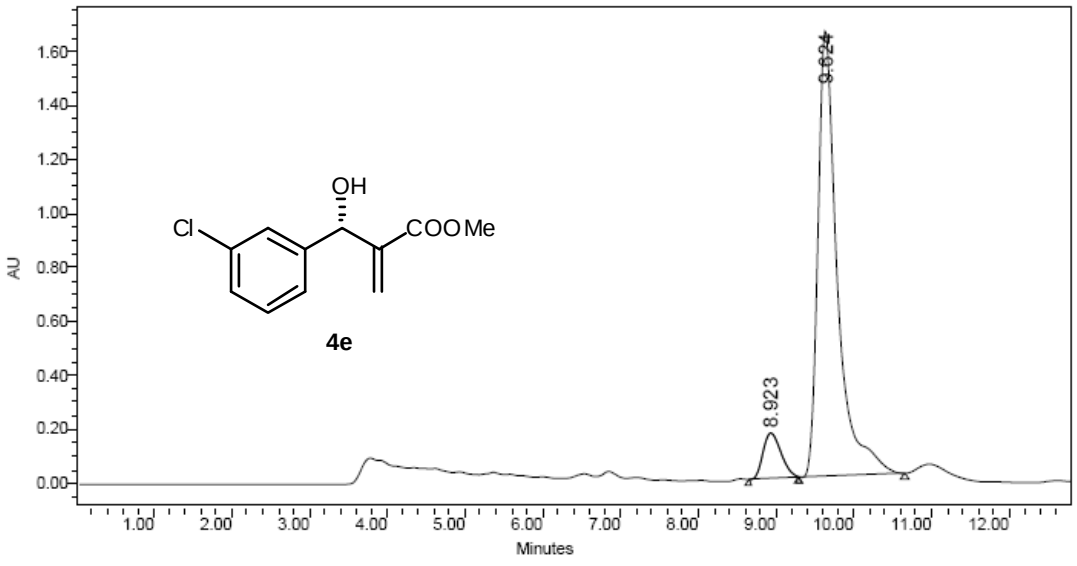
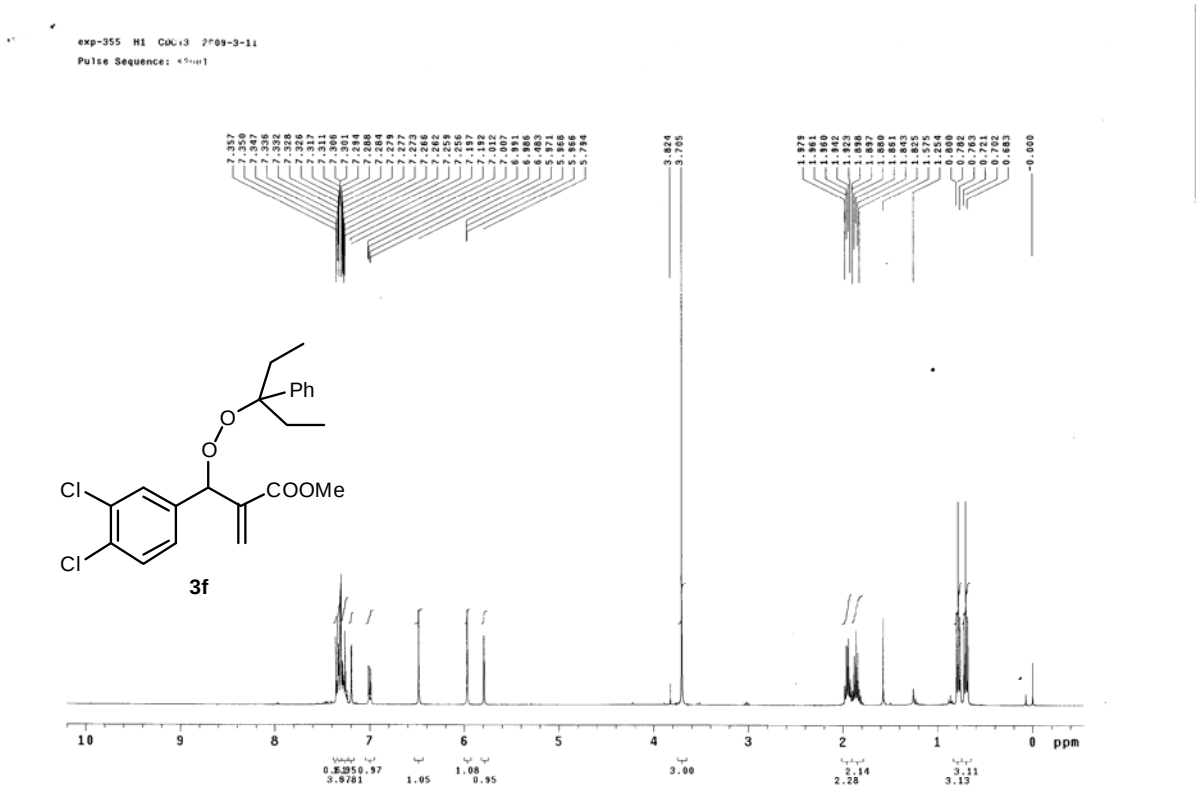


	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	9.030	12671435	47.93	859985	52.04
2	9.775	13768403	52.07	792570	47.96



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	8.923	2706691	8.59	168623	9.25
2	9.624	28808307	91.41	1654973	90.75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4
3
2
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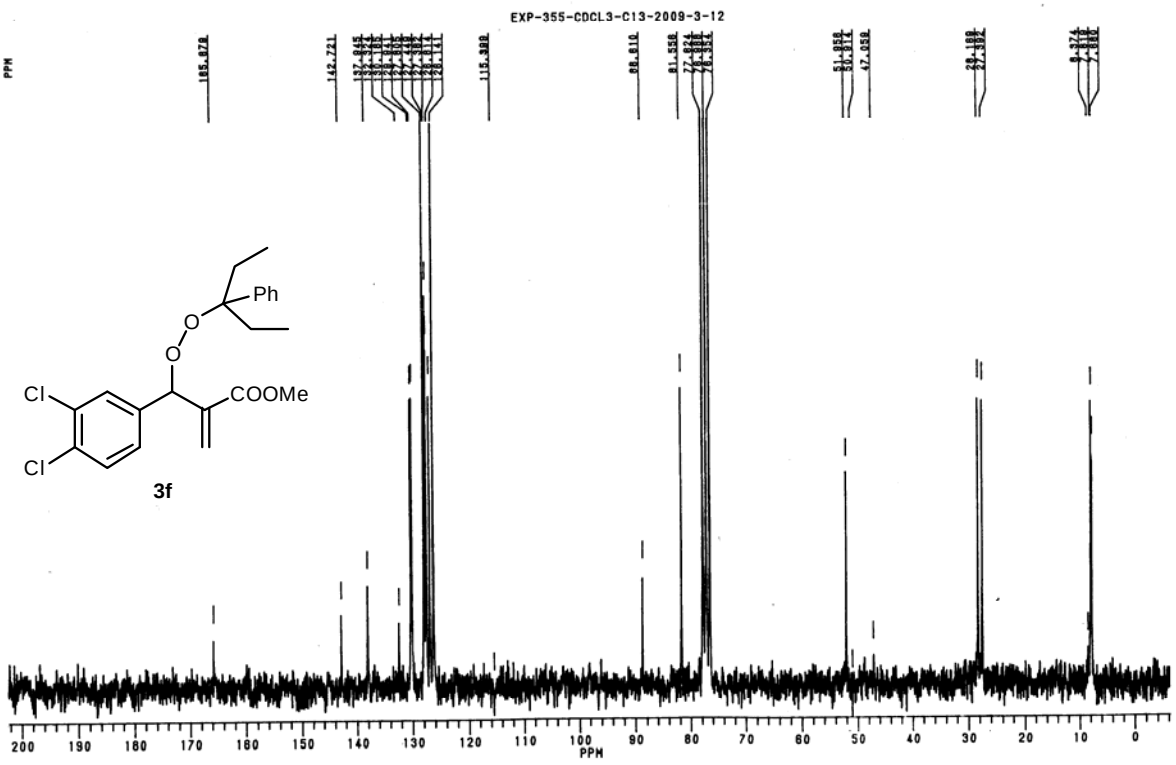
0
ppm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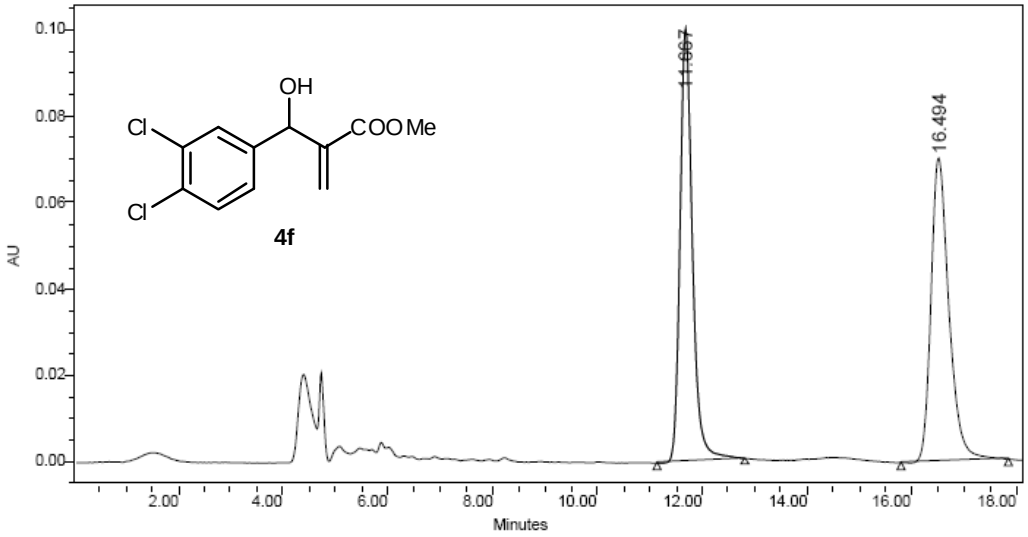
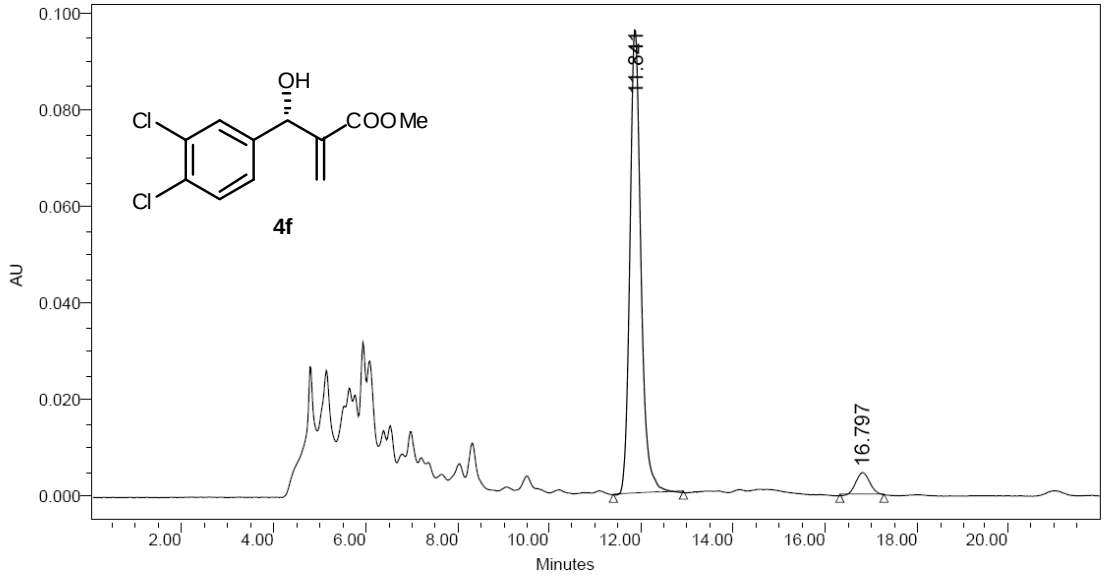


	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	11.667	1653973	49.65	100421	58.79
2	16.494	1677567	50.35	70382	41.21



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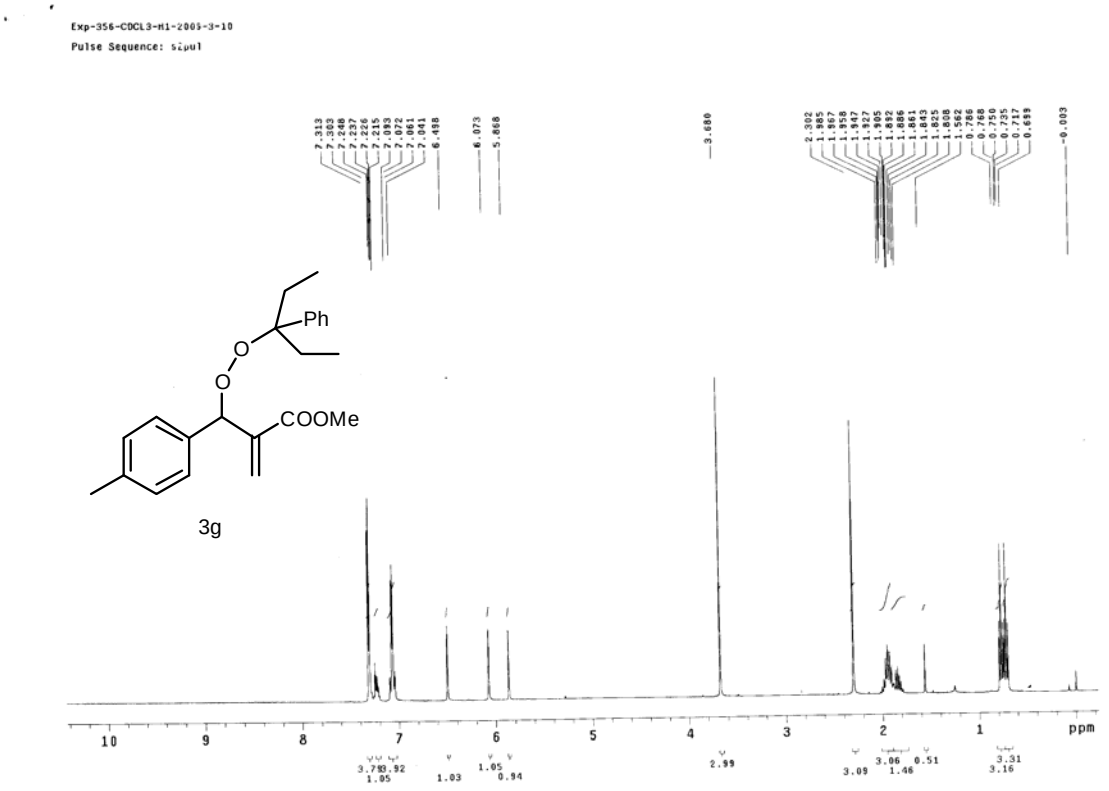
1	11.841	1653673	94.13	96407	95.32
2	16.797	103089	5.87	4730	4.68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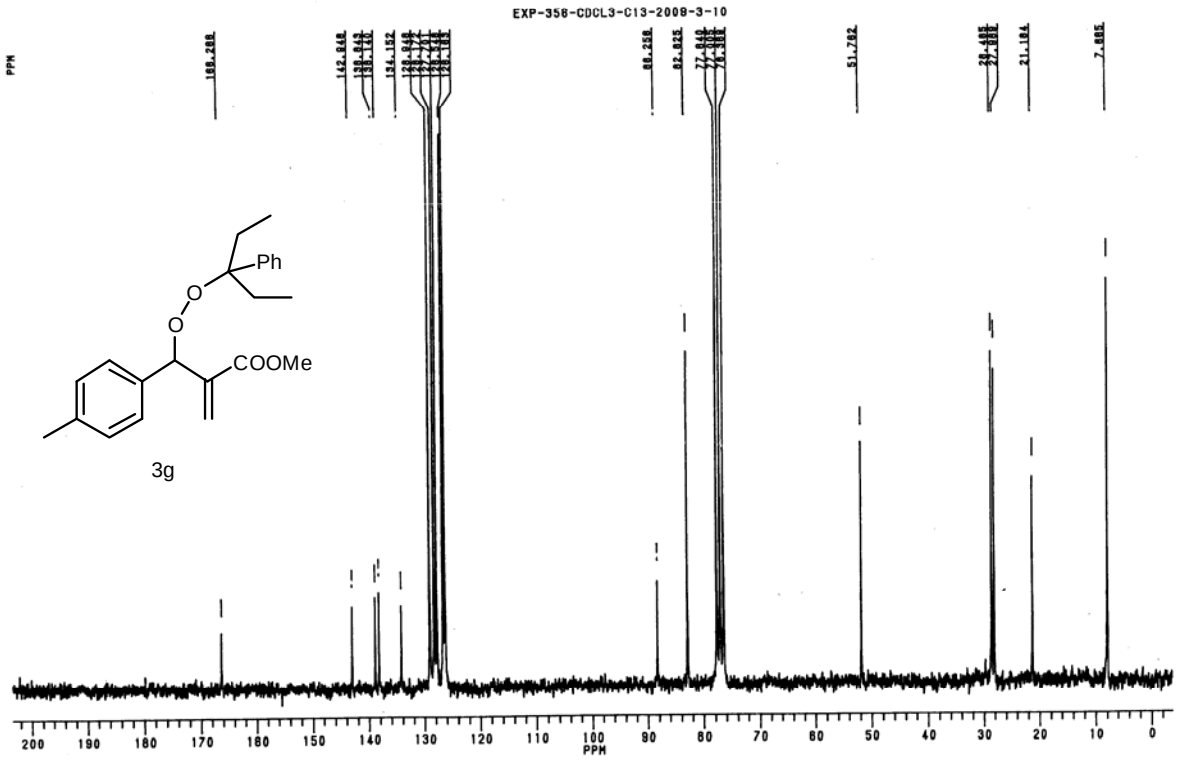
COOMe
3g
ppm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COOMe
3g
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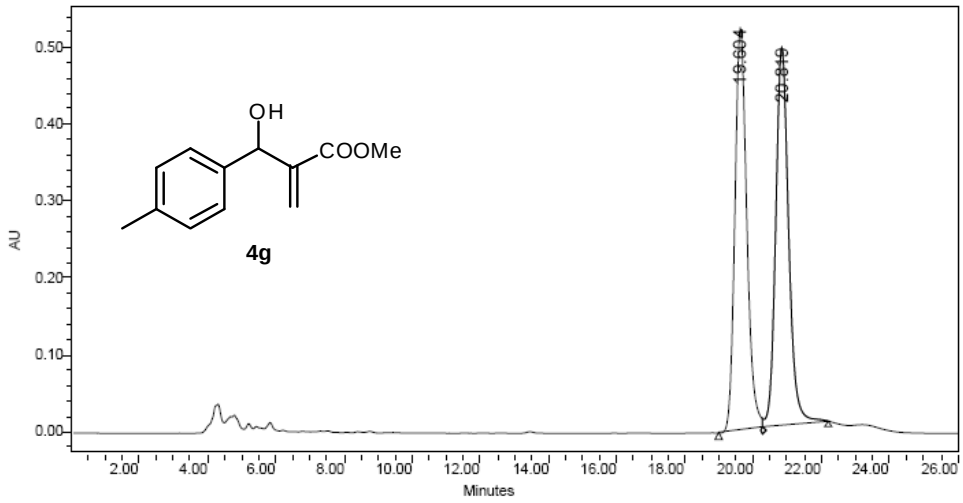

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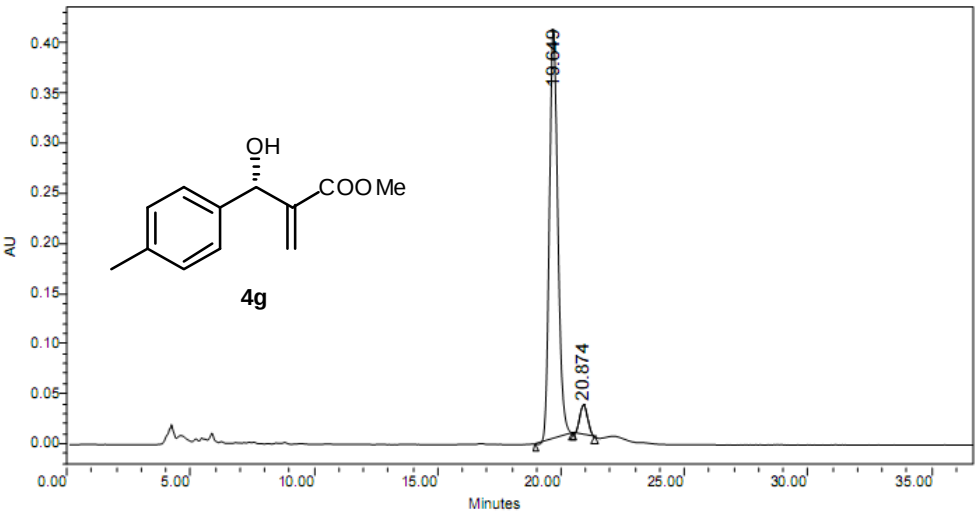
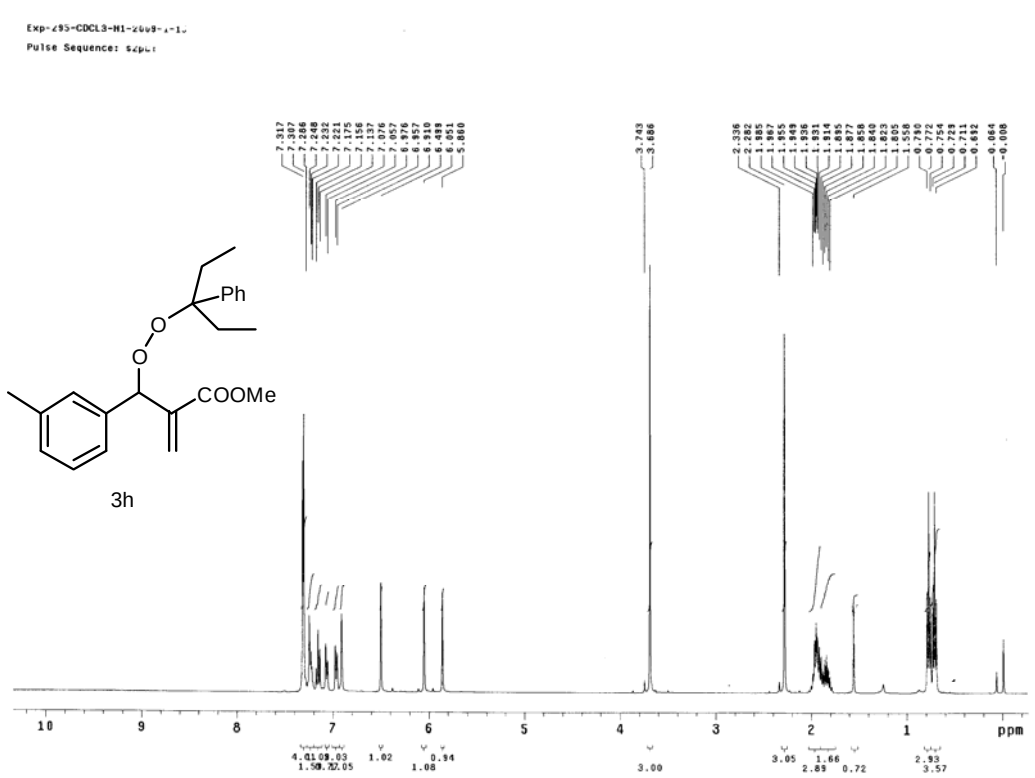
1	19.604	12719341	49.36	523463	51.52
2	20.819	13047153	50.64	492508	48.48



	RT (min)	Area (µV*sec)	% Area	Height (µV)	% Height
1	19.649	9796908	93.39	409485	93.08
2	20.874	693127	6.61	30429	6.92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COOMe
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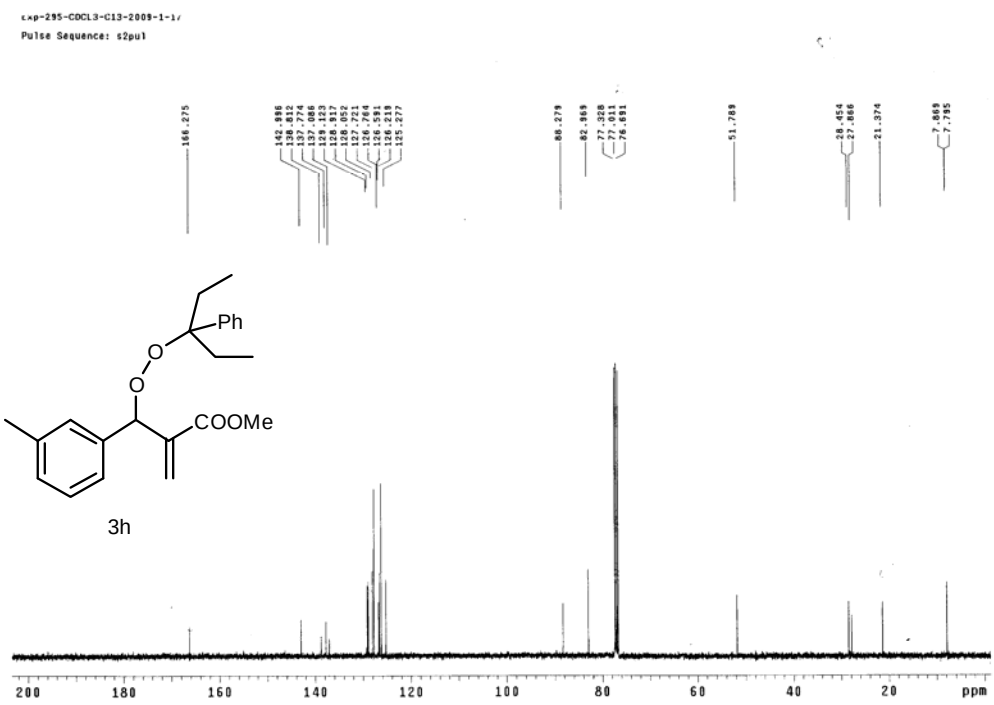
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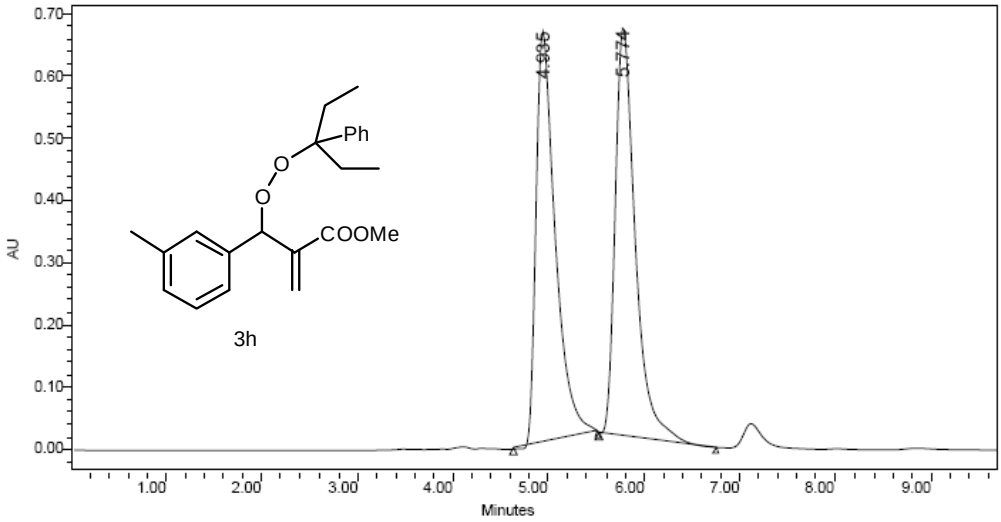

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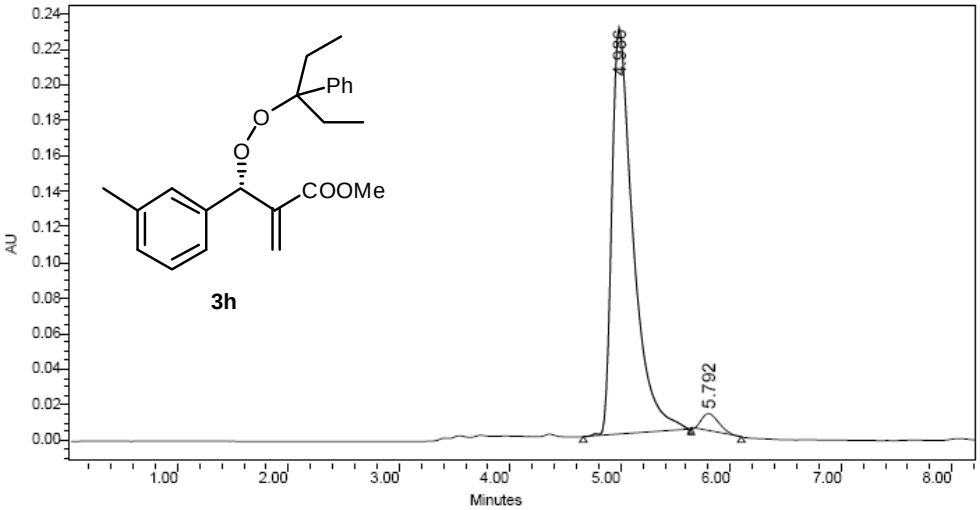
1	4.935	9297882	49.30	662276	50.18
2	5.774	9562636	50.70	657557	49.82



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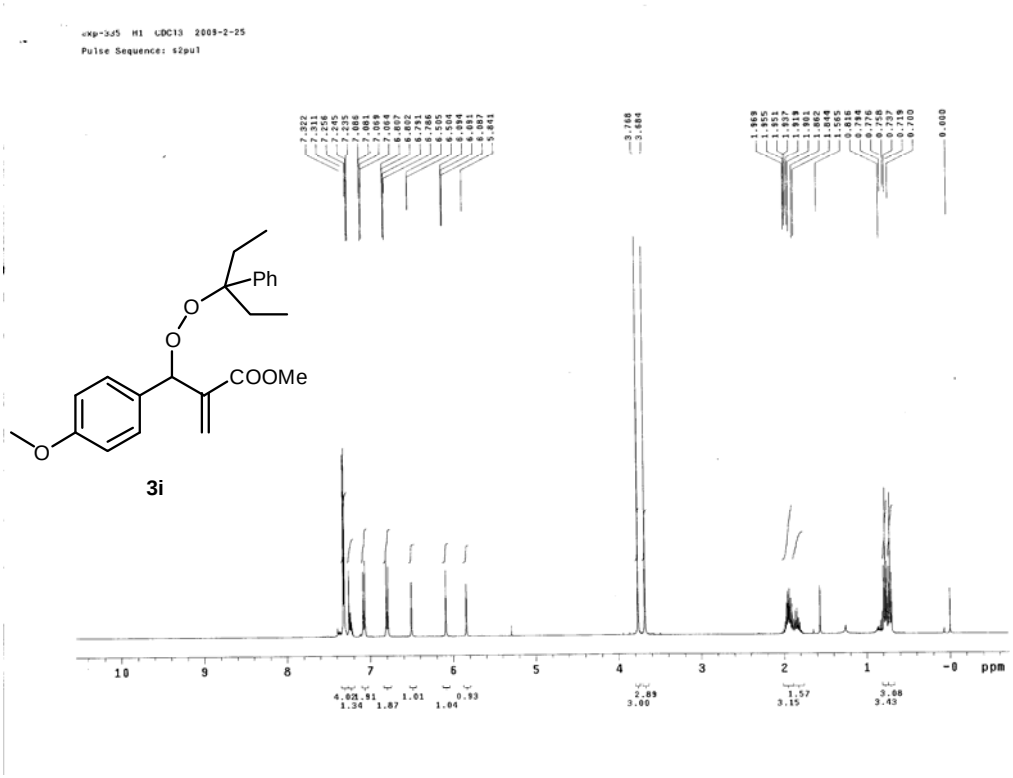
1	4.986	3072903	96.41	228857	95.75
2	5.792	114538	3.59	10149	4.25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COOMe
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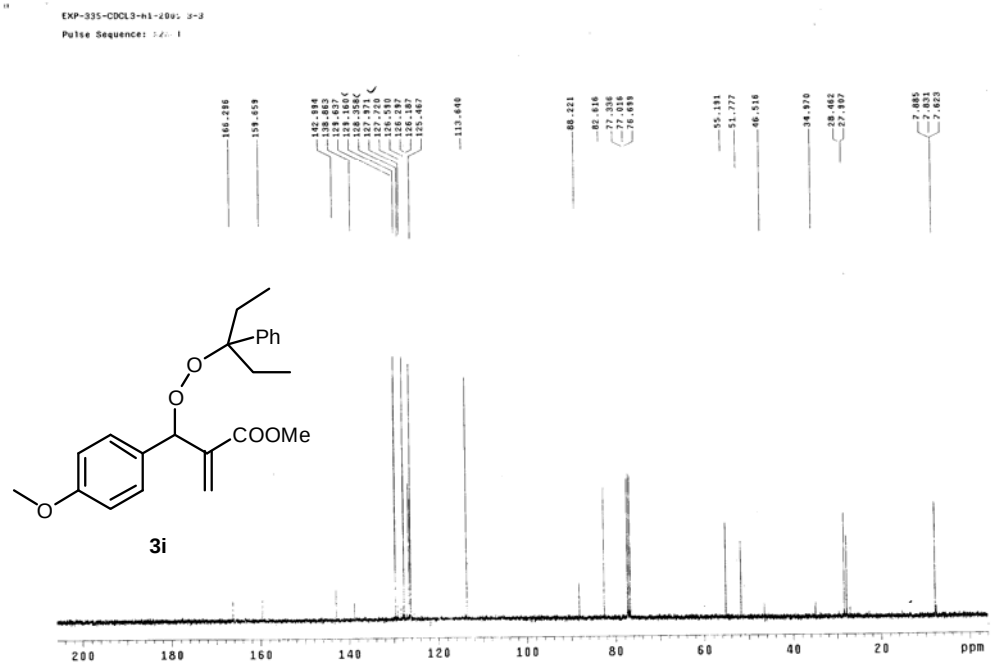
3i
ppm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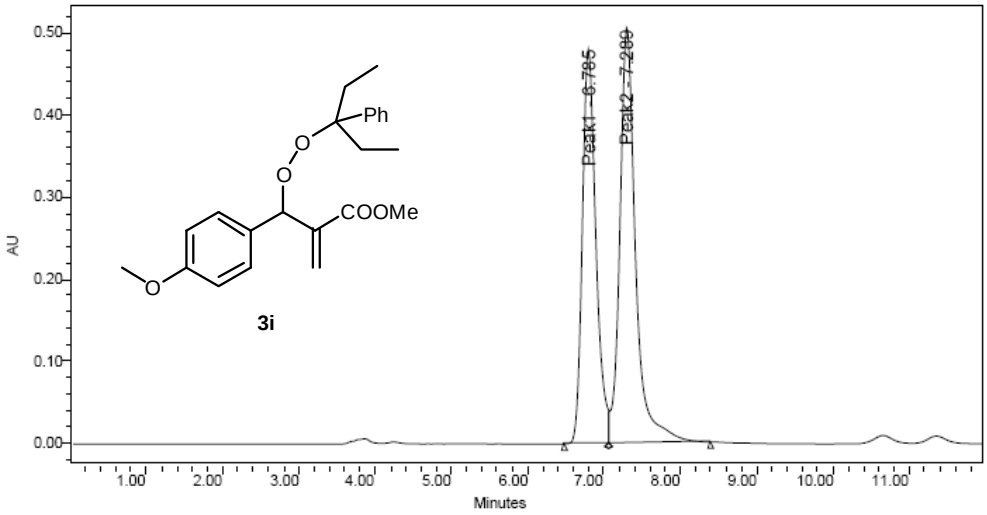

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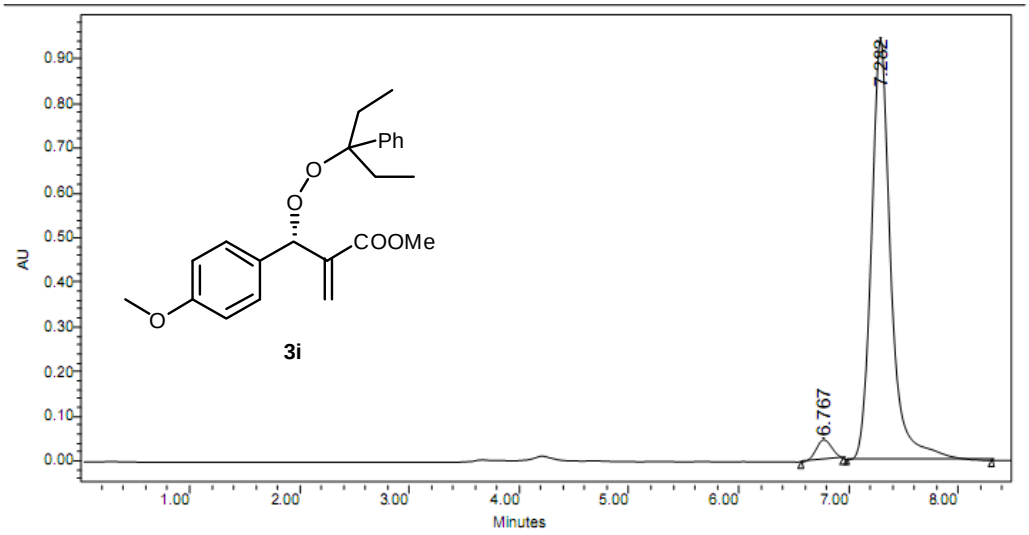
1	Peak1	6.785	5952573	45.08	480851	48.64
2	Peak2	7.289	7250455	54.92	507766	51.36



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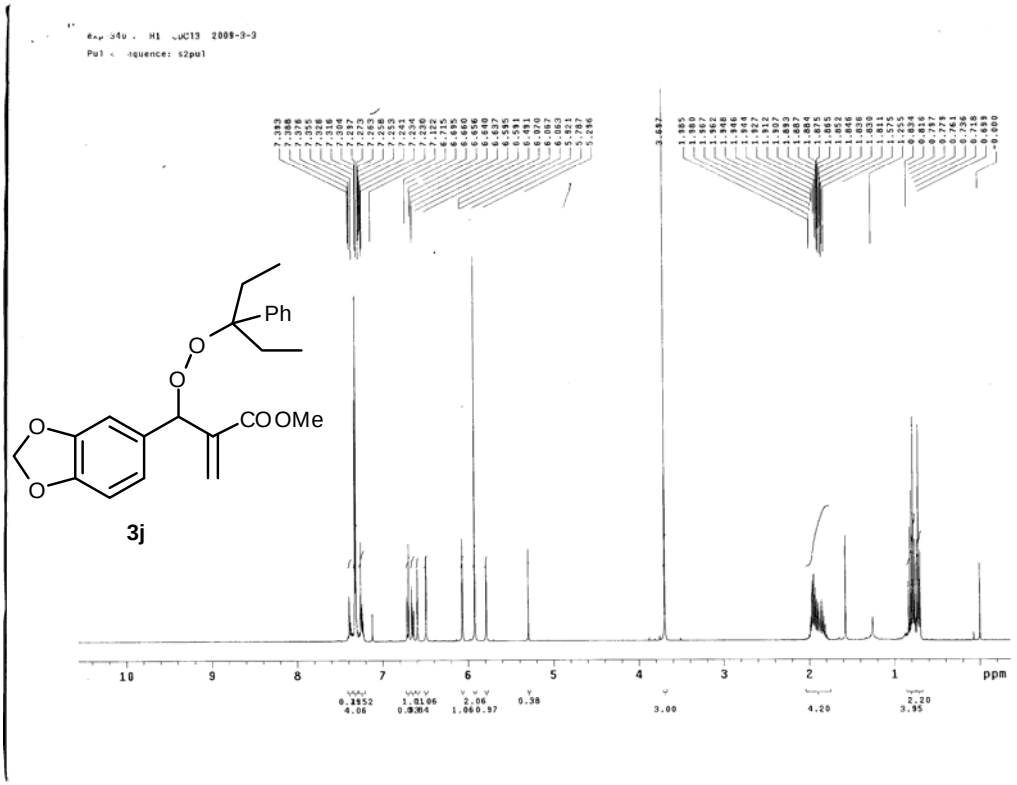
1	6.767	460228	3.81	45689	4.62
2	7.282	11629229	96.19	943574	95.38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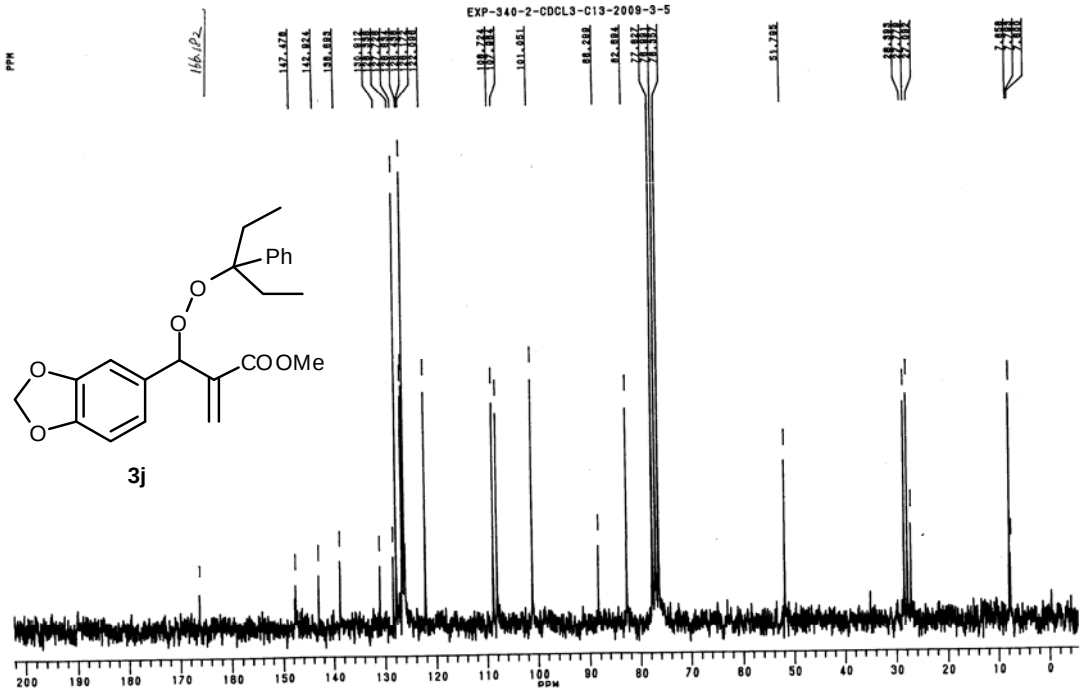
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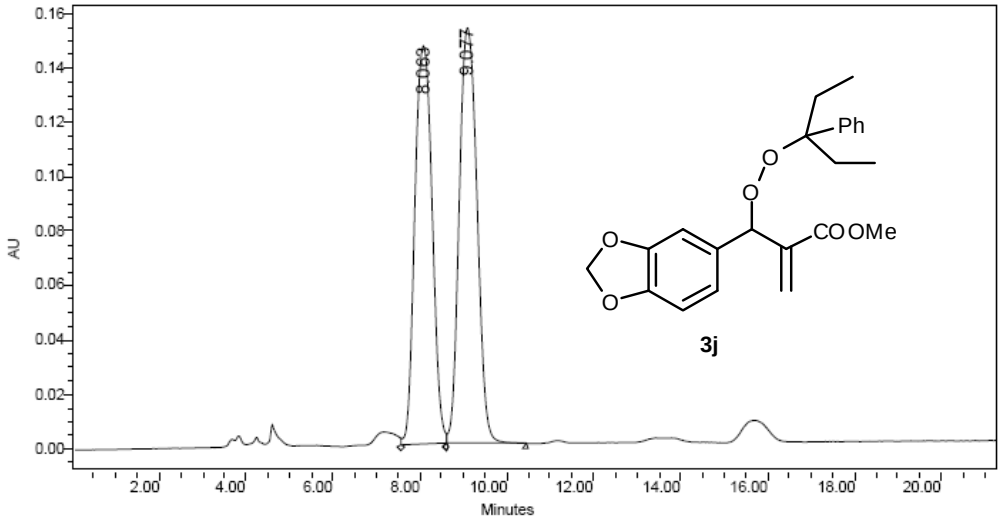

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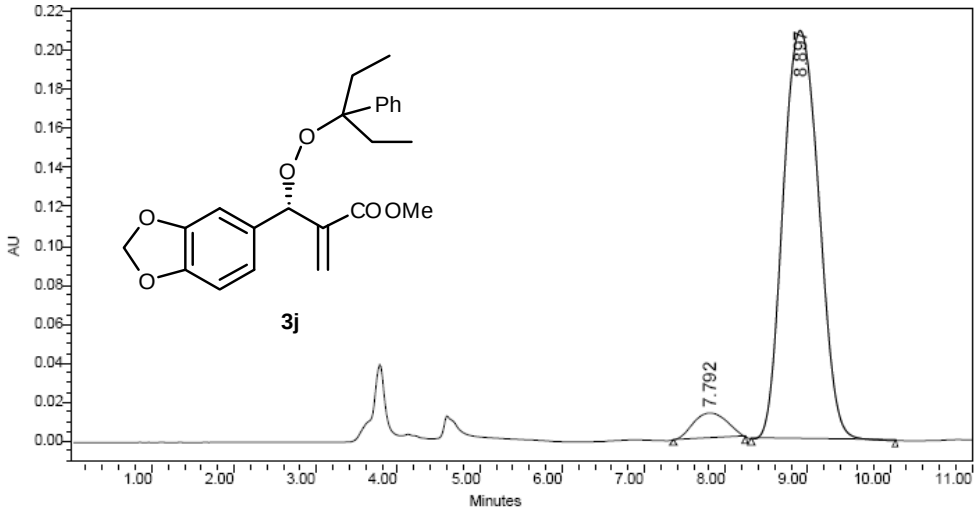
1	8.063	4073506	48.24	146589	48.93
2	9.077	4370645	51.76	153021	51.07



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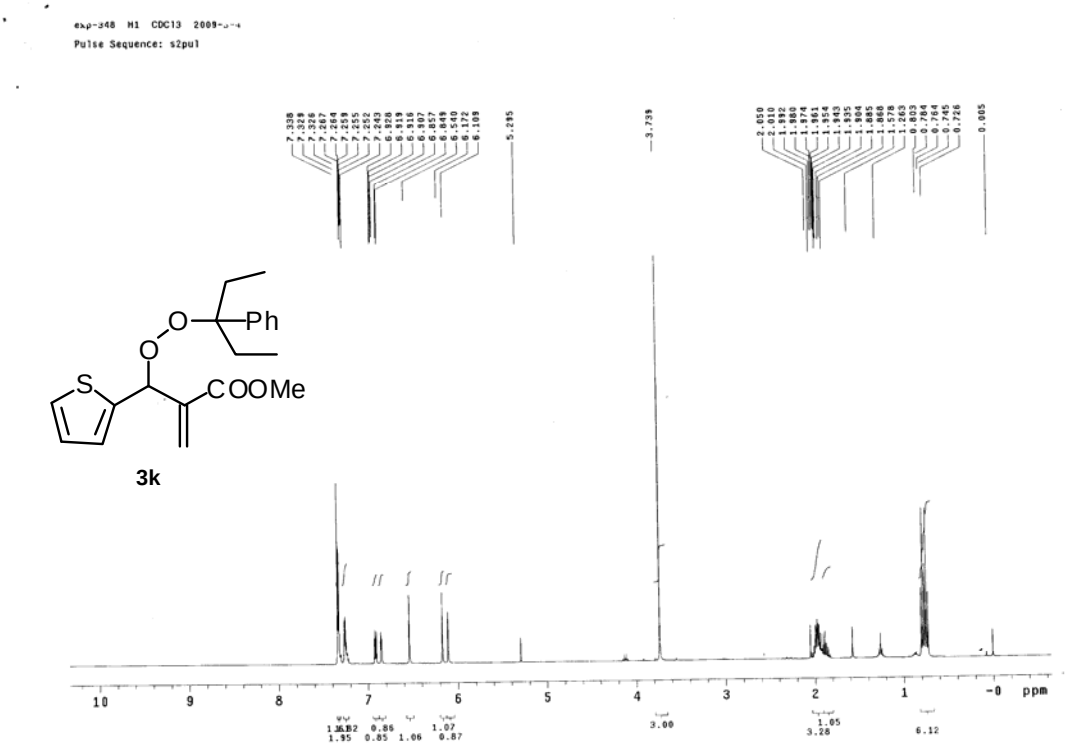
1	7.792	374355	5.58	12963	5.84
2	8.897	6334600	94.42	209194	94.16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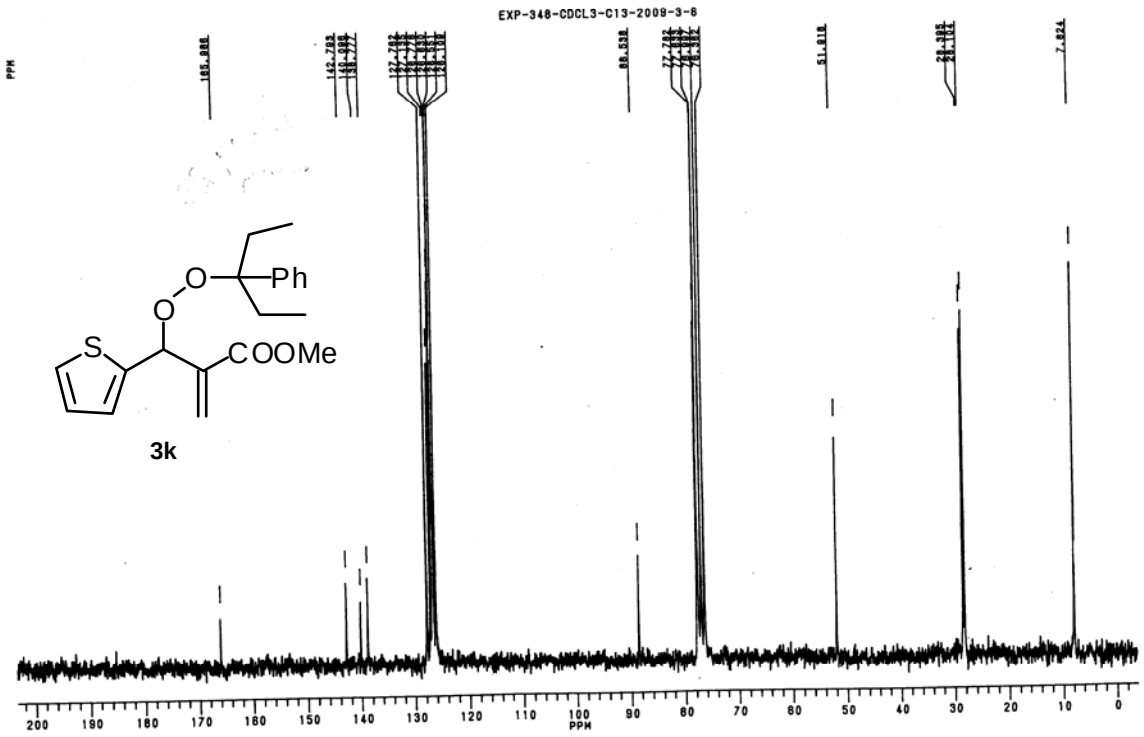
S
COOMe
3k
ppm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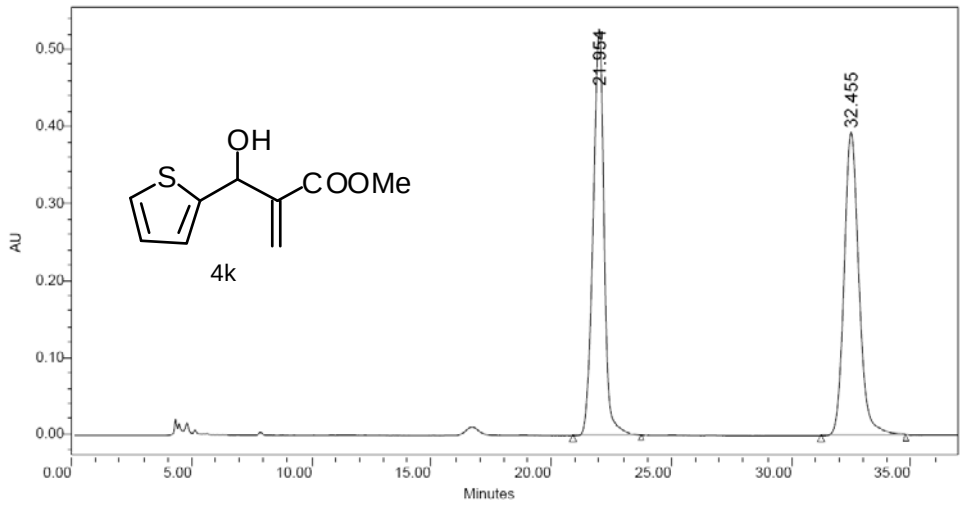

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1	21.954	17801245	49.94	527413	57.24
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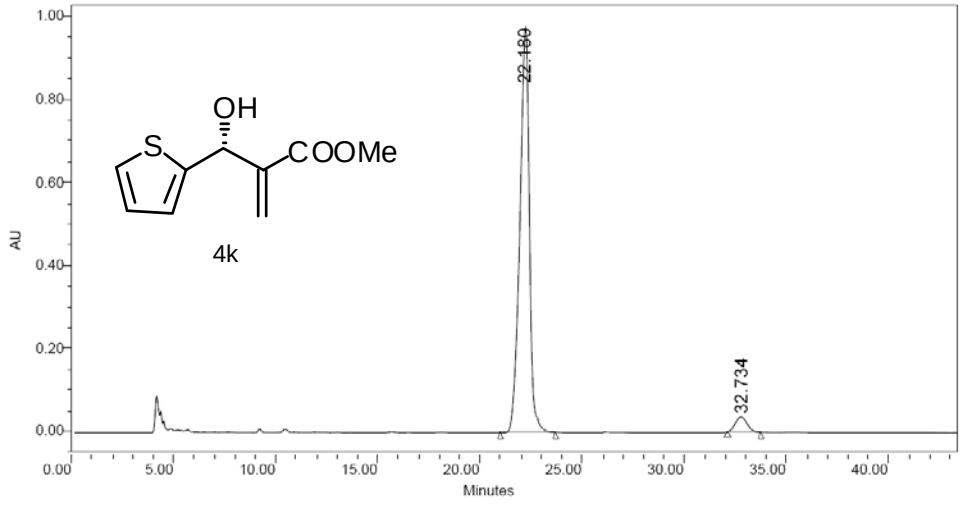
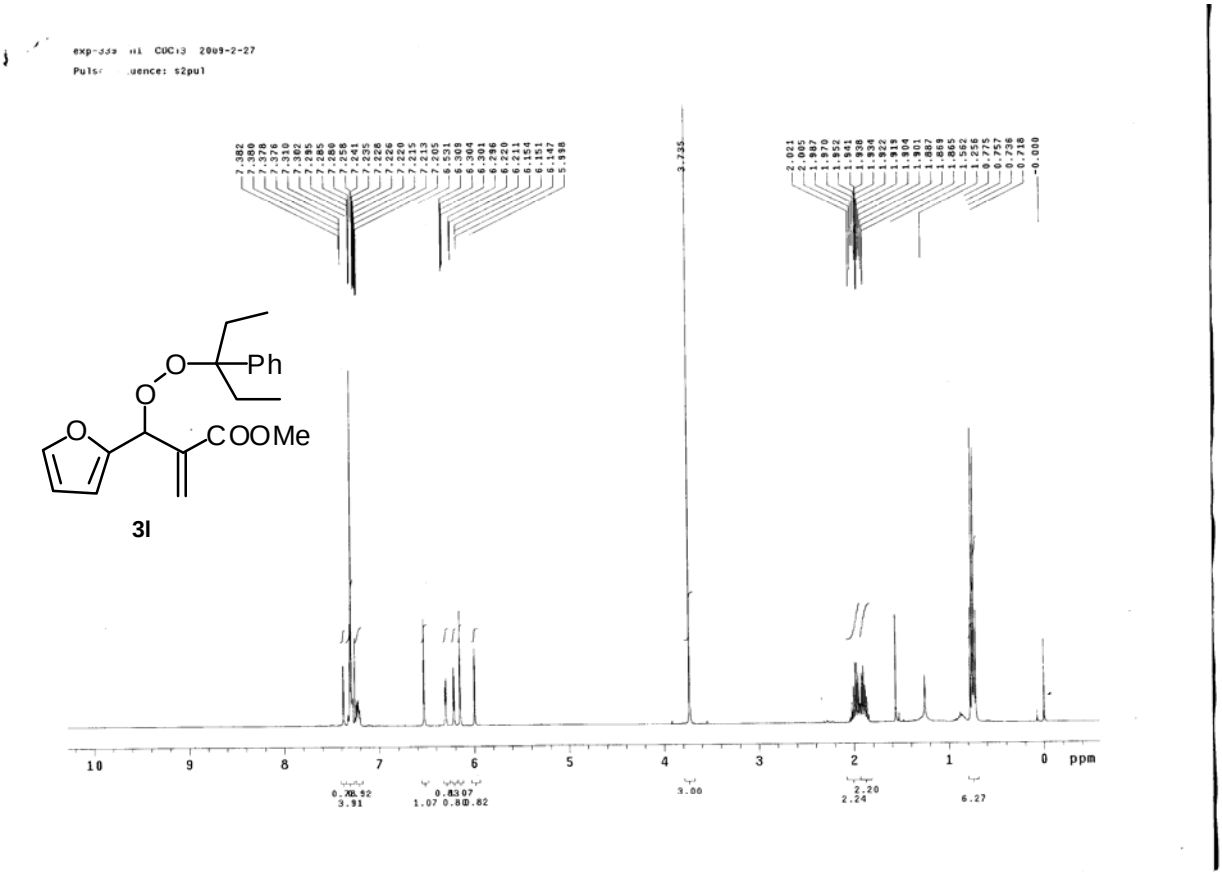
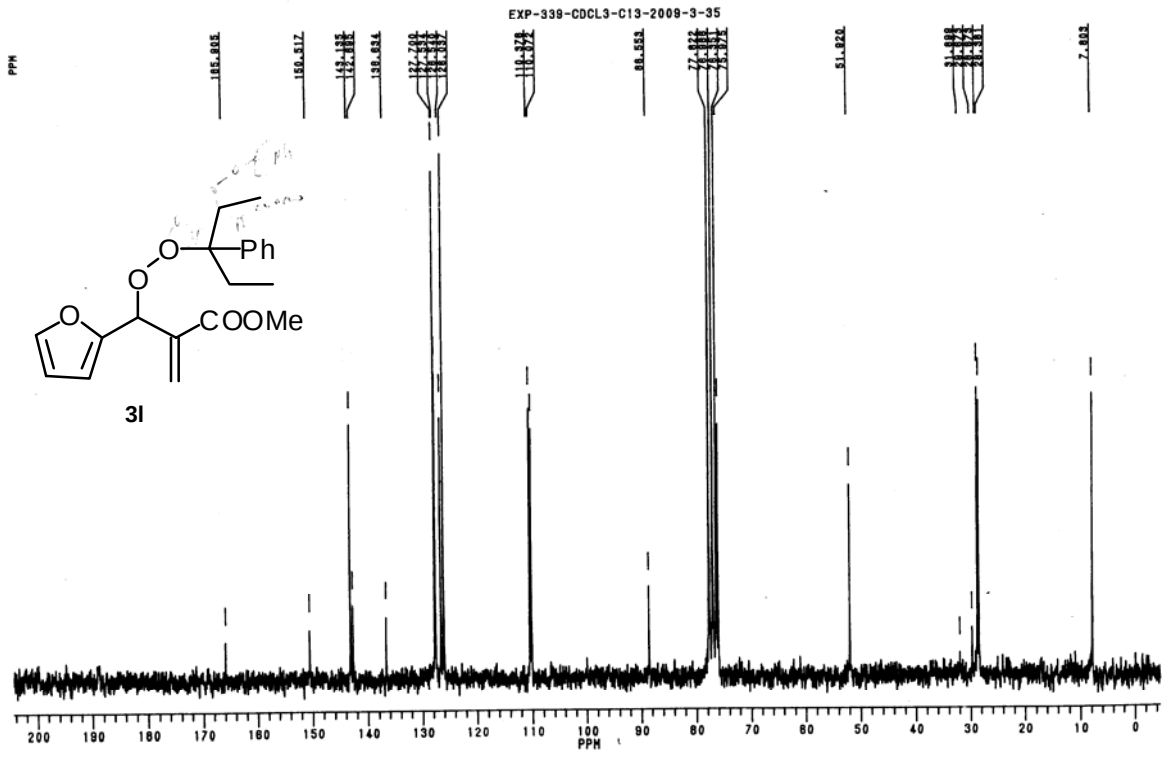
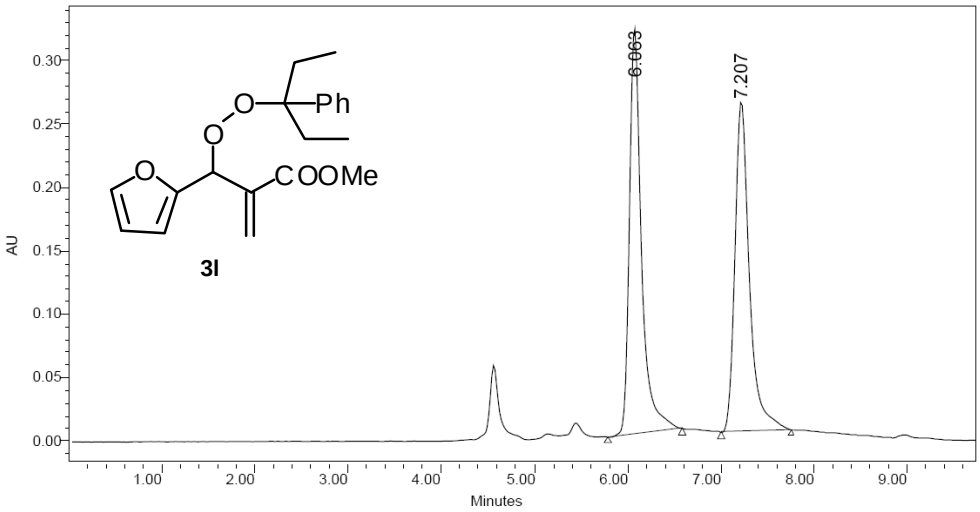


	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	22.180	34046554	95.95	975496	96.43
2	32.734	1436131	4.05	36092	3.57

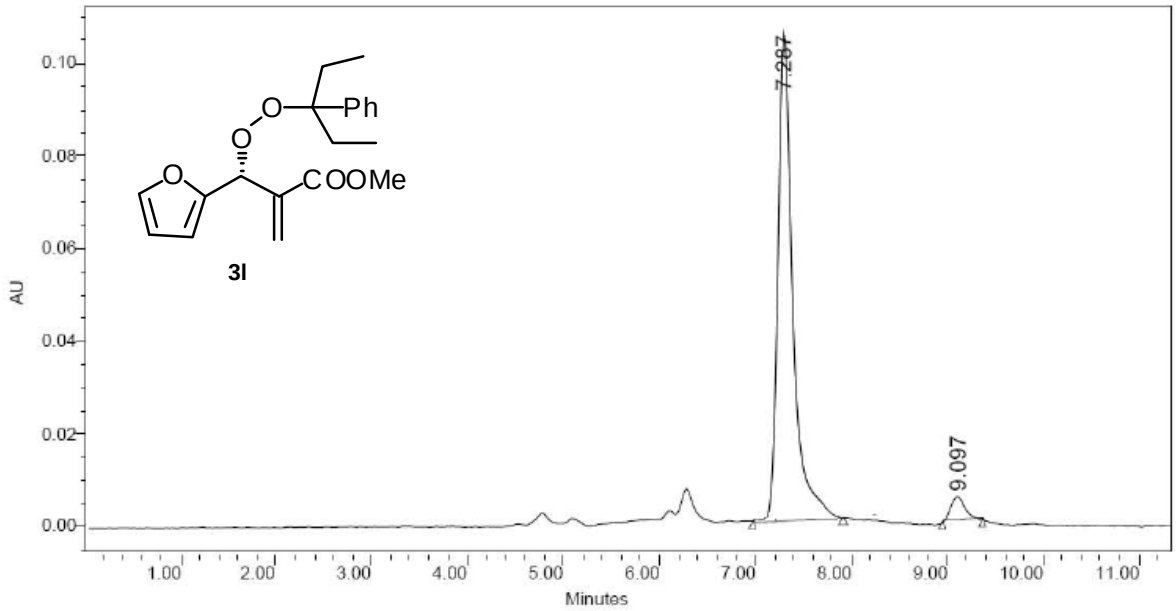

Ph
O
O
O
COOMe
3l
ppm


EXP-339-CDCL3-C13-2009-3-35
PPM
Ph
O
O
O
COOMe
3l



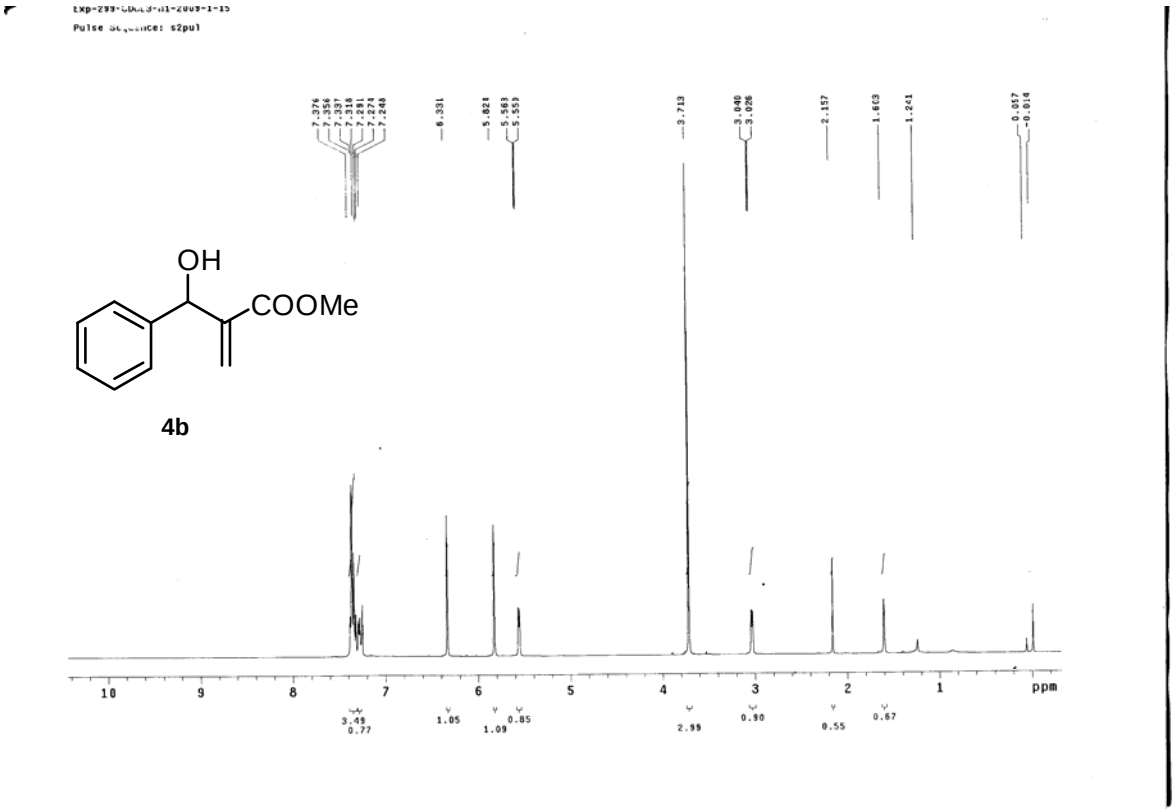
3l

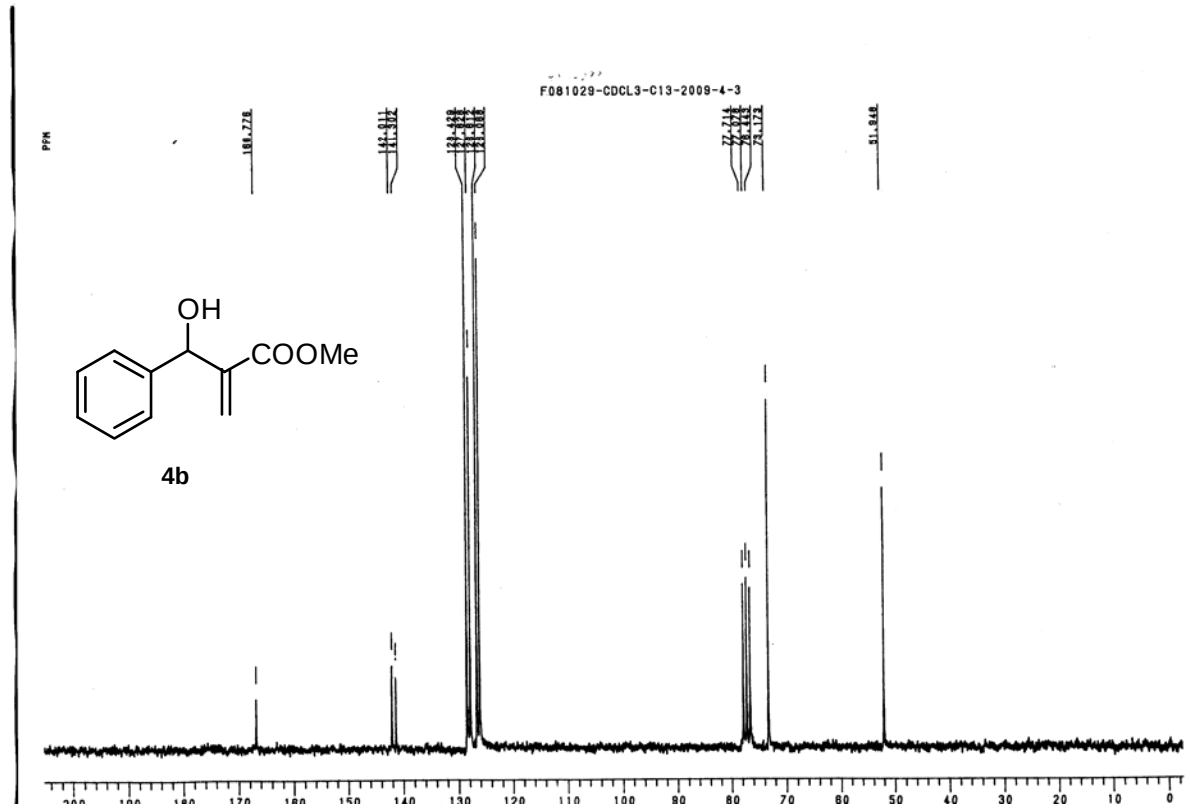
	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	6.063	2895198	50.68	319725	55.09
2	7.207	2817763	49.32	260618	44.91

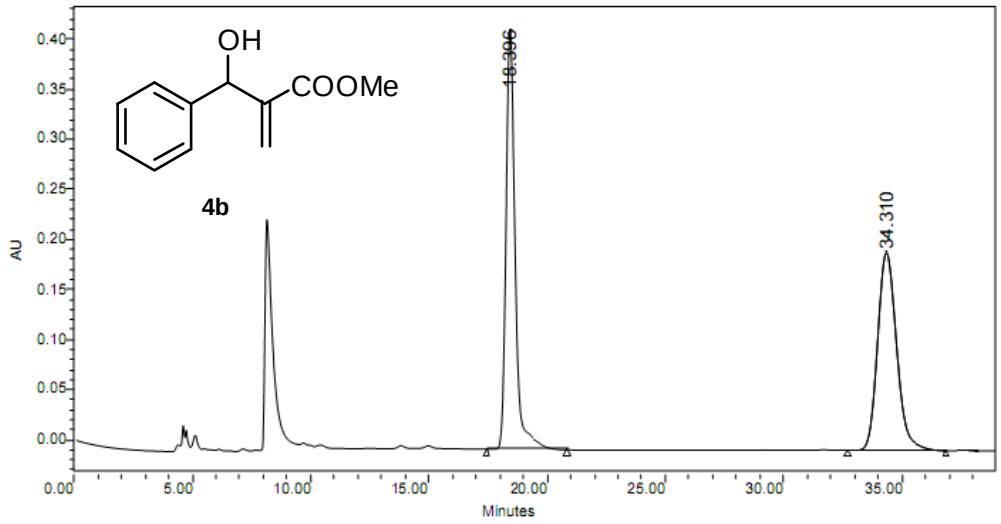


3l

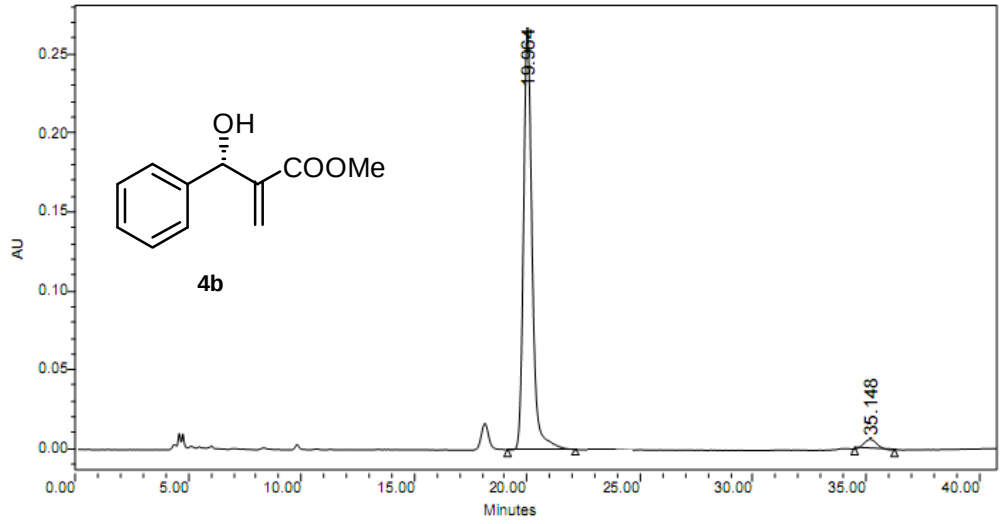
	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	7.287	1101077	95.20	104583	95.18
2	9.097	55474	4.80	5301	4.82


OH
COOMe
4b
ppm

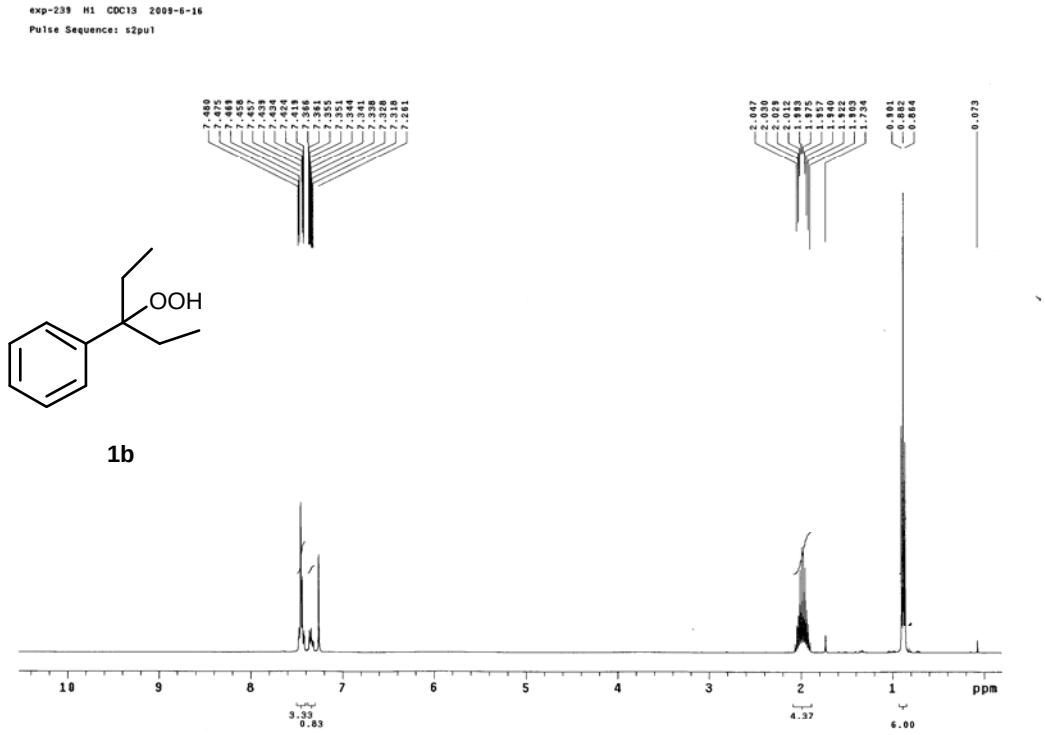

F081029-CDCL3-C13-2009-4-3
PPM
OH
COOMe
4b



	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	18.396	11850416	50.39	419460	68.00
2	34.310	11667636	49.61	197420	32.00



	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	19.964	7524568	96.72	267156	97.69
2	35.148	254857	3.28	6305	2.31


exp-239 H1 CDCl3 2009-6-16
Pulse Sequence: s2pul
OOH
10
9
8
7
6
5
4
3
2
1
ppm
3.33
0.83
4.37
6.00

1b