

Supporting Information

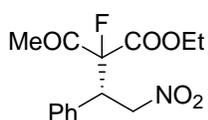
Synthesis of chiral fluorinated quaternary carbon containing β -ketoesters by direct organocatalytic asymmetric conjugate addition reactions of fluoroketoesters with nitroolefins

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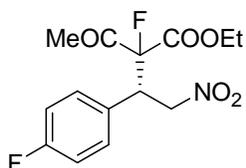
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General Information: Commercial reagents were used as received, unless otherwise stated. Merck 60 silica gel was used for chromatography, and Whatman silica gel plates with fluorescence F₂₅₄ were used for thin-layer chromatography (TLC) analysis. ¹H and ¹³C NMR spectra were recorded on Broker Avance 500, and tetramethylsilane (TMS) was used as a reference. Data for ¹H are reported as follows: chemical shift (ppm), and multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet). Data for ¹³C NMR are reported as ppm. Mass Spectra were obtained from Ohio State University Mass Spectral facility.

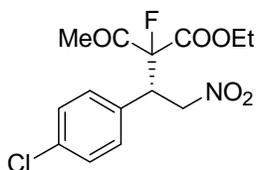
General Procedure for addition of ethyl 2-fluoro-3-oxobutanoate to *trans*-β-Nitrostyrenes: 0.105 mmol *trans*-β-Nitrostyrenes in the presence of 1 mol % catalyst in 0.5 ml 1,2-dichloethane was added 0.1 mmol ethyl 2-fluoro-3-oxobutanoate and the resulting solution stirred for 24-48 h at rt. The solution was purified by silica gel chromatography without work-up and fractions were collected and concentrated *in vacuo* to provide the desired products.



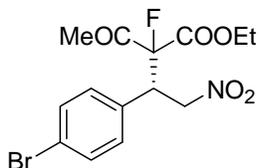
(2S, 3S) Ethyl 2-acetyl-2-fluoro-4-nitro-3-phenylbutanoate (3a) (Table 2, entry 1): Yield: 97%; ¹H NMR (500 MHz, CDCl₃): δ 7.31 (m, 8H), 4.83-4.89 (m, 2H), 4.53-4.76 (m, 2.5H), 4.33 (dq, 2H, *J*₁ = 1.5 Hz, *J*₂ = 7.0 Hz) (major), 3.98 (q, 1H, *J* = 7.0 Hz) (minor), 2.34 (d, 1.5H, *J* = 4.5 Hz) (minor), 1.87 (d, 3H, *J* = 5.5 Hz) (major), 1.34 (t, 3H, *J* = 7.0 Hz) (major), 0.99 (t, 1.5H, *J* = 7.0 Hz) (minor); ¹³C NMR (125 MHz, CDCl₃): δ 201.3, 201.1, 199.6, 199.3, 164.5, 164.3, 163.8, 163.7, 133.0, 132.4, 129.4, 129.1, 129.0, 128.9, 128.8, 101.7, 101.3, 100.0, 99.6, 75.21, 75.18, 63.5, 63.0, 47.2, 47.0, 46.4, 46.3, 29.6, 26.3, 25.7, 13.8, 13.5; [α]_D²³ = + 33.0 (*c* = 3.0, CHCl₃); HPLC (Daicel CHIRALPAK AS-H, hexane/2-PrOH = 90:10, flow rate 0.5 mL/min, λ = 220 nm); *t*_{Rmajor} = 19.09 (major), 22.13 (minor) min; *t*_{Rminor} = 21.33 (major), 27.55 (minor) min, ee = 97% (major) and 96% (minor).



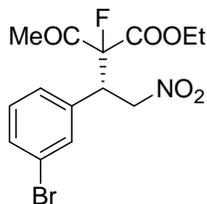
(2S, 3S) Ethyl 2-acetyl-2-fluoro-3-(4-fluorophenyl)-4-nitrobutanoate (3b) (Table 2, entry 2): Yield: 95%; ¹H NMR (500 MHz, CDCl₃): δ 7.29 (m, 2.3H), 7.03 (m, 2.4H), 4.81 (m, 2H), 4.63 (m, 2H), 4.33 (q, 2H, *J* = 7.5 Hz) (major), 4.01 (q, 0.5H, *J* = 7.0 Hz) (minor), 2.34 (d, 0.8H, *J* = 4.5 Hz) (minor), 1.91 (d, 3H, *J* = 5.5 Hz) (major), 1.34 (t, 3H, *J* = 7.0 Hz) (major), 1.04 (t, 0.7H, *J* = 7.0 Hz) (minor); ¹³C NMR (125 MHz, CDCl₃): δ 201.0, 200.8, 164.4, 164.2, 163.8, 163.5, 161.8, 131.35, 131.28, 131.03, 130.98, 128.9, 128.3, 116.1, 115.9, 115.8, 101.2, 99.6, 75.2, 63.6, 63.1, 46.5, 46.3, 45.7, 45.6, 26.3, 25.7, 13.81, 13.50; [α]_D²³ = + 24.2 (*c* = 4.0, CHCl₃); HPLC (Daicel CHIRALPAK AS-H, hexane/2-PrOH = 85:15, flow rate 0.5 mL/min, λ = 220 nm); *t*_{Rmajor} = 17.69 (major), 23.44 (minor) min; *t*_{Rminor} = 19.70 (major), 28.16 (minor) min, ee = 98% (major) and 97% (minor).



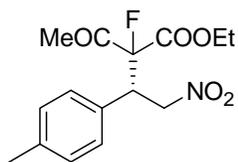
(2S, 3S) Ethyl 2-acetyl-3-(4-chlorophenyl)-2-fluoro-4-nitrobutanoate (3c) (Table 2, entry 3): Yield: 98%; ¹H NMR (500 MHz, CDCl₃): δ 7.29 (m, 5.6H), 4.82 (m, 2H), 4.61 (m, 2H), 4.33 (q, 2H, *J* = 7.0 Hz) (major), 4.02 (q, 0.5H, *J* = 7.0 Hz) (minor), 2.34 (d, 0.7H, *J* = 5.0 Hz) (minor), 1.93 (d, 3H, *J* = 5.5 Hz) (major), 1.34 (t, 3H, *J* = 7.0 Hz) (major), 1.05 (t, 0.6H, *J* = 7.0 Hz) (minor); ¹³C NMR (125 MHz, CDCl₃): δ 200.9, 200.6, 199.3, 199.1, 164.3, 164.1, 135.0, 131.6, 131.0, 130.9, 130.5, 129.2, 129.1, 101.2, 99.5, 75.09, 75.06, 63.7, 63.2, 46.5, 46.3, 45.8, 45.6, 26.3, 25.7, 13.8, 13.6; [α]_D²³ = + 38.2 (*c* = 3.0, CHCl₃); HPLC (Daicel CHIRALCEL OD-H, hexane/2-PrOH = 90:10, flow rate 0.5 mL/min, λ = 220 nm); *t*_{Rmajor} = 27.36 (major), 69.24 (minor) min; *t*_{Rminor} = 33.27 (major), 44.83 (minor) min, ee = 98% (major) and 97% (minor).



(2S, 3S) Ethyl 2-acetyl-3-(4-bromophenyl)-2-fluoro-4-nitrobutanoate (3d) (Table 2, entry 4): Yield: 97%; ¹H NMR (500 MHz, CDCl₃): δ 7.47 (m, 2.5H), 7.20 (m, 2.5H), 4.82 (m, 2H), 4.60 (m, 2H), 4.33 (m, 2H) (major), 4.03 (m, 0.5H) (minor), 2.34 (d, 0.8H, *J* = 4.5 Hz) (minor), 1.94 (d, 3H, *J* = 5.5 Hz) (major), 1.34 (t, 3H, *J* = 7.5 Hz) (major), 1.05 (t, 0.8H, *J* = 7.0 Hz) (minor); ¹³C NMR (125 MHz, CDCl₃): δ 200.9, 200.6, 199.3, 199.1, 164.3, 164.1, 163.6, 163.4, 132.2, 132.1, 131.6, 131.2, 130.8, 123.1, 101.1, 99.4, 75.03, 75.00, 63.7, 63.2, 46.6, 46.4, 45.9, 45.7, 26.3, 25.7, 13.8, 13.6; [α]_D²³ = + 19.2 (*c* = 5.0, CHCl₃); HPLC (Daicel CHIRALCEL OD-H, hexane/2-PrOH = 90:10, flow rate 0.5 mL/min, λ = 220 nm); *t*_{Rmajor} = 35.15 (major), 46.91 (minor) min; *t*_{Rminor} = 39.98 (major), 75.02 (minor) min, ee = 99% (major) and 94% (minor).

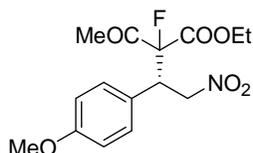


(2S, 3S) Ethyl 2-acetyl-3-(4-bromophenyl)-2-fluoro-4-nitrobutanoate (3e) (Table 2, entry 5): Yield: 97%; ¹H NMR (500 MHz, CDCl₃): δ 7.47 (m, 2.5H), 7.20 (m, 2.5H), 4.82 (m, 2H), 4.60 (m, 2H), 4.33 (m, 2H) (major), 4.03 (m, 0.5H) (minor), 2.34 (d, 0.8H, *J* = 4.5 Hz) (minor), 1.94 (d, 3H, *J* = 5.5 Hz) (major), 1.34 (t, 3H, *J* = 7.5 Hz) (major), 1.06 (t, 0.8H, *J* = 7.0 Hz) (minor); ¹³C NMR (125 MHz, CDCl₃): δ 200.9, 200.6, 199.3, 199.1, 164.3, 164.1, 163.6, 163.4, 132.2, 132.1, 131.6, 131.2, 130.8, 123.1, 101.1, 99.4, 75.03, 75.00, 63.7, 63.2, 46.6, 46.4, 45.9, 45.7, 26.3, 25.7, 13.8, 13.6; [α]_D²³ = + 18.6 (*c* = 5.0, CHCl₃); HPLC (Daicel CHIRALCEL OD-H, hexane/2-PrOH = 90:10, flow rate 0.5 mL/min, λ = 220 nm); *t*_{Rmajor} = 35.15 (major), 46.91 (minor) min; *t*_{Rminor} = 39.98 (major), 75.02 (minor) min, ee = 98% (major) and 97% (minor).

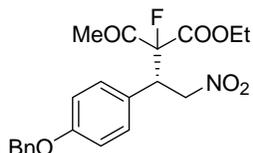


(2S, 3S) Ethyl 2-acetyl-2-fluoro-4-nitro-3-p-tolylbutanoate (3f) (Table 2, entry 6): Yield: 92%; ¹H NMR (500 MHz, CDCl₃): δ 7.16 (m, 5.0H), 4.82 (d, 2.4H, *J* = 7.5 Hz), 4.61 (m, 2.4H), 4.32 (q, 2H, *J* =

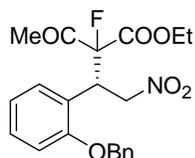
7.0 Hz) (major), 4.00 (m, 0.5H) (minor), 2.32 (d, 1.7H, $J = 5.0$ Hz) (minor), 2.30 (s, 3H), 1.88 (d, 3H, $J = 5.5$ Hz) (major), 1.33 (t, 3H, $J = 7.0$ Hz) (major), 1.04 (t, 0.7H, $J = 7.0$ Hz) (minor); ^{13}C NMR (125 MHz, CDCl_3): δ 201.2, 201.0, 199.7, 199.4, 164.6, 164.4, 163.9, 163.7, 138.7, 129.9, 129.7, 129.5, 129.3, 129.0, 101.4, 99.7, 75.36, 75.33, 63.5, 63.0, 46.9, 46.8, 46.1, 46.0, 26.3, 25.7, 21.0, 13.8, 13.6; $[\alpha]_{\text{D}}^{23} = +31.5$ ($c = 4.0$, CHCl_3); HPLC (Daicel CHIRALCEL OD-H, hexane/2-PrOH = 95:5, flow rate 0.5 mL/min, $\lambda = 220$ nm); $t_{\text{Rmajor}} = 32.05$ (major), 48.27 (minor) min; $t_{\text{Rminor}} = 43.83$ (major), 40.72 (minor) min, ee = 99% (major) and 98% (minor).



(2S, 3S) Ethyl 2-acetyl-2-fluoro-3-(4-methoxyphenyl)-4-nitrobutanoate (3g) (Table 2, entry 7): Yield: 95%; ^1H NMR (500 MHz, CDCl_3): δ 7.23 (m, 2.5H), 6.84 (d, 2H, $J = 8.5$ Hz), 4.81 (d, 2H, $J = 7.5$ Hz), 4.61 (m, 2H), 4.32 (q, 2H, $J = 6.0$ Hz) (major), 4.01 (q, 0.7H, $J = 7.0$ Hz) (minor), 3.77 (s, 3H), 2.33 (d, 0.7H, $J = 4.5$ Hz) (minor), 1.89 (d, 3H, $J = 5.5$ Hz) (major), 1.33 (t, 3H, $J = 7.0$ Hz) (major), 1.04 (t, 0.9H, $J = 7.0$ Hz) (minor); ^{13}C NMR (125 MHz, CDCl_3): δ 201.3, 201.1, 199.7, 199.4, 164.6, 164.4, 159.8, 130.6, 130.3, 124.8, 124.1, 114.3, 114.2, 101.4, 99.8, 75.4, 63.5, 63.0, 55.1, 46.6, 45.5, 45.8, 45.7, 26.3, 25.7, 13.8, 13.6; $[\alpha]_{\text{D}}^{23} = +31.0$ ($c = 4.0$, CHCl_3); HPLC (Daicel CHIRALCEL OD-H, hexane/2-PrOH = 80:20, flow rate 0.5 mL/min, $\lambda = 220$ nm); $t_{\text{Rmajor}} = 23.47$ (major), 34.58 (minor) min; $t_{\text{Rminor}} = 25.91$ (major), 29.88 (minor) min, ee = 98% (major) and 95% (minor).

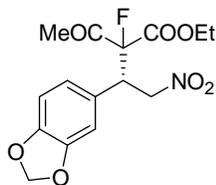


(2S, 3S) Ethyl 2-acetyl-3-(4-(benzyloxy)phenyl)-2-fluoro-4-nitrobutanoate (3h) (Table 2, entry 8): Yield: 96%; ^1H NMR (500 MHz, CDCl_3): δ 7.32-7.40 (m, 6.5H), 7.22 (m, 2.7H), 6.91 (m, 2.7H), 5.01 (s, 2.7H), 4.80 (d, 2H, $J = 7.5$ Hz), 4.56 (m, 2H), 4.32 (m, 2H) (major), 3.99 (q, 0.6H, $J = 7.0$ Hz) (minor), 2.32 (d, 0.9H, $J = 4.5$ Hz) (minor), 1.88 (d, 3H, $J = 5.5$ Hz) (major), 1.33 (t, 3H, $J = 7.0$ Hz) (major), 1.01 (t, 1H, $J = 7.0$ Hz) (minor); ^{13}C NMR (125 MHz, CDCl_3): δ 201.3, 201.1, 199.6, 199.4, 164.6, 164.4, 163.9, 163.7, 159.1, 136.6, 136.5, 130.7, 130.3, 128.5, 128.0, 127.5, 125.1, 124.4, 115.3, 115.1, 101.4, 99.8, 75.4, 70.0, 63.5, 63.0, 46.6, 46.5, 45.8, 45.7, 26.3, 25.7, 13.8, 13.6; $[\alpha]_{\text{D}}^{23} = +22.7$ ($c = 5.0$, CHCl_3); HPLC (Daicel CHIRALCEL OD-H, hexane/2-PrOH = 80:20, flow rate 0.5 mL/min, $\lambda = 220$ nm); $t_{\text{Rmajor}} = 28.20$ (major), 57.72 (minor) min; $t_{\text{Rminor}} = 33.23$ (major), 42.24 (minor) min, ee = 98% (major) and 97% (minor).

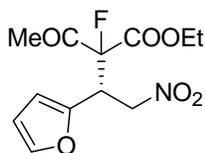


(2S, 3S) Ethyl 2-acetyl-3-(2-(benzyloxy)phenyl)-2-fluoro-4-nitrobutanoate (3i) (Table 2, entry 9): Yield: 90%; ^1H NMR (500 MHz, CDCl_3): δ 7.50 (m, 3.5H), 7.36 (m, 6H), 7.24 (m, 1.5H), 6.94 (m, 3H), 5.33 (m, 1.5H), 5.11 (s, 3H), 4.71-4.88 (m, 3H), 4.29 (q, 2H, $J = 7.0$ Hz) (major), 3.95 (m, 1.2H) (minor), 2.32 (d, 1.8H, $J = 4.5$ Hz) (minor), 1.96 (d, 3H, $J = 5.0$ Hz) (major), 1.31 (t, 3H, $J = 7.0$ Hz) (major), 0.90 (t, 1.9H, $J = 7.0$ Hz) (minor); ^{13}C NMR (125 MHz, CDCl_3): δ 200.1, 199.9, 199.7, 199.5, 164.9, 164.7,

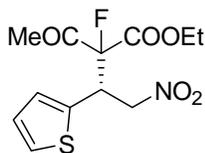
164.3, 164.1, 156.4, 136.7, 136.6, 130.1, 129.9, 129.3, 128.6, 128.5, 127.9, 127.8, 127.0, 122.4, 121.7, 121.2, 112.9, 112.5, 101.7, 101.3, 100.0, 99.6, 75.22, 75.18, 75.0, 70.5, 70.4, 63.4, 62.8, 39.4, 26.1, 25.7, 13.8, 13.4; $[\alpha]_D^{23} = +18.1$ ($c = 2.0$, CHCl_3); HPLC (Daicel CHIRALCEL OD-H, hexane/2-PrOH = 95:5, flow rate 0.5 mL/min, $\lambda = 220$ nm); $t_{R\text{major}} = 30.39$ (major), 41.43 (minor) min; $t_{R\text{minor}} = 26.50$ (major), 32.96 (minor) min, ee = 97% (major) and 95% (minor).



(2S, 3S) Ethyl 2-acetyl-3-(benzo[d][1,3]dioxol-5-yl)-2-fluoro-4-nitrobutanoate (3j) (Table 2, entry 10): Yield: 95%; $^1\text{H NMR}$ (500 MHz, CDCl_3): δ 6.76 (m, 3.6H), 7.02 (s, 1H), 5.93 (s, 2.5H), 4.76 (s, 2H, $J = 7.0$ Hz), 4.43-4.67 (m, 2.6H), 4.30 (q, 2H, $J = 6.5$ Hz) (major), 4.03 (q, 0.6H, $J = 7.0$ Hz) (minor), 2.31 (d, 1H, $J = 4.5$ Hz) (minor), 1.94 (d, 3H, $J = 5.5$ Hz) (major), 1.31 (t, 3H, $J = 7.0$ Hz) (major), 1.06 (t, 0.8H, $J = 7.0$ Hz) (minor); $^{13}\text{C NMR}$ (125 MHz, CDCl_3): δ 201.1, 200.9, 164.5, 164.3, 148.0, 126.2, 125.6, 123.3, 123.0, 109.4, 109.2, 108.6, 108.4, 101.3, 99.7, 75.4, 63.5, 63.1, 46.9, 46.8, 46.1, 46.0, 26.4, 25.7, 13.8, 13.7; $[\alpha]_D^{23} = +28.7$ ($c = 5.0$, CHCl_3); HPLC (Daicel CHIRALCEL OD-H, hexane/2-PrOH = 90:10, flow rate 0.5 mL/min, $\lambda = 220$ nm); $t_{R\text{major}} = 39.20$ (major), 59.40 (minor) min; $t_{R\text{minor}} = 46.80$ (major) min, ee = 97% (major) and 99% (minor).

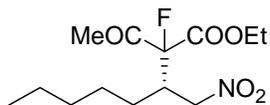


(2S, 3R) Ethyl 2-acetyl-2-fluoro-3-(furan-2-yl)-4-nitrobutanoate (3k) (Table 2, entry 11): Yield: 94%; $^1\text{H NMR}$ (500 MHz, CDCl_3): δ 7.37 (s, 1H), 6.31 (s, 1H), 6.27 (d, 1H, $J = 8.0$ Hz), 4.87 (m, 1.2H), 4.73 (m, 2.6H), 4.31 (q, 2H, $J = 7.0$ Hz) (major), 4.16 (m, 0.5H) (minor), 2.30 (d, 0.8H, $J = 4.5$ Hz) (minor), 2.04 (d, 3H, $J = 5.5$ Hz) (major), 1.31 (t, 3H, $J = 7.0$ Hz) (major), 1.17 (t, 0.8H, $J = 7.0$ Hz) (minor); $^{13}\text{C NMR}$ (125 MHz, CDCl_3): δ 200.9, 200.7, 164.2, 164.0, 146.7, 146.1, 143.4, 143.3, 110.9, 110.7, 110.3, 100.1, 98.4, 73.2, 63.6, 63.3, 41.4, 41.2, 40.9, 40.7, 29.6, 25.73, 25.66, 13.8; $[\alpha]_D^{23} = +19.8$ ($c = 4.0$, CHCl_3); HPLC (Daicel CHIRALCEL OD-H, hexane/2-PrOH = 90:10, flow rate 0.5 mL/min, $\lambda = 220$ nm); $t_{R\text{major}} = 15.77$ (major), 39.61 (minor) min; $t_{R\text{minor}} = 19.31$ (major), 28.09 (minor) min, ee = 99% (major) and 99% (minor).

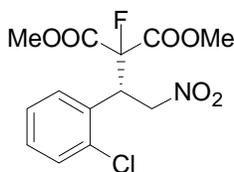


(2S, 3S) Ethyl 2-acetyl-2-fluoro-4-nitro-3-(thiophen-2-yl)butanoate (3l) (Table 2, entry 12): Yield: 97%; $^1\text{H NMR}$ (500 MHz, CDCl_3): δ 7.29 (d, 1H, $J = 5.0$ Hz), 7.02 (s, 1H), 6.96 (s, 1H), 4.67-4.99 (m, 4H), 4.33 (q, 2H, $J = 6.5$ Hz) (major), 4.10 (q, 0.6H, $J = 7.0$ Hz) (minor), 2.36 (d, 0.8H, $J = 4.5$ Hz) (minor), 2.04 (d, 3H, $J = 5.0$ Hz) (major), 1.34 (t, 3H, $J = 7.0$ Hz) (major), 1.12 (t, 0.8H, $J = 7.0$ Hz) (minor); $^{13}\text{C NMR}$ (125 MHz, CDCl_3): δ 200.9, 200.6, 164.2, 164.0, 134.0, 133.5, 129.2, 128.5, 127.1, 127.0, 126.9, 126.6, 100.8, 99.1, 76.2, 75.9, 63.7, 63.3, 43.1, 42.9, 42.2, 42.0, 29.6, 26.3, 25.9, 13.8; $[\alpha]_D^{23} = +16.4$ ($c = 3.0$, CHCl_3); HPLC (Daicel CHIRALCEL OD-H, hexane/2-PrOH = 90:10, flow rate

0.5 mL/min, $\lambda = 220$ nm); $t_{R\text{major}} = 19.44$ (major), 44.81 (minor) min; $t_{R\text{minor}} = 24.47$ (major), 36.31 (minor) min, ee = 99% (major) and 98% (minor).

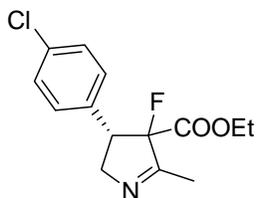


(2S, 3S) Ethyl 2-acetyl-2-fluoro-3-(nitromethyl)octanoate (3m) (Table 2, entry 13): Yield: 75%; ^1H NMR (500 MHz, CDCl_3): δ 4.58 (q, 1H, $J_1 = 5.5$ Hz, $J_2 = 14.0$ Hz), 4.36 (q, 1H, $J_1 = 5.5$ Hz, $J_2 = 14.0$ Hz), 4.24 (q, 2H, $J = 6.5$ Hz), 3.28 (m, 1H), 2.33 (d, 3H, $J = 5.5$ Hz), 1.24-1.41 (m, 11H), 0.86 (t, 3H, $J = 7.0$ Hz); ^{13}C NMR (125 MHz, CDCl_3): δ 201.4, 201.2, 164.8, 164.6, 101.8, 100.2, 74.5, 63.2, 41.4, 41.2, 31.4, 27.9, 26.4, 26.3, 22.2, 13.8, 13.7; $[\alpha]_{\text{D}}^{23} = -1.5$ ($c = 3.0$, CHCl_3); HPLC (Daicel CHIRALCEL OD-H, hexane/2-PrOH = 99.5:0.5, flow rate 0.5 mL/min, $\lambda = 220$ nm); $t_{R\text{major}} = 24.46$ (major), 59.20 (minor) min; $t_{R\text{minor}} = 26.28$ (major), 44.57 (minor) min, ee = 98% (major) and 96% (minor).



(S) Dimethyl 2-(1-(2-chlorophenyl)-2-nitroethyl)-2-fluoromalonate (3n) (Table 2, entry 14): Yield: 98%; ^1H NMR (500 MHz, CDCl_3): δ 7.48 (d, 1H, $J = 7.0$ Hz), 7.41 (d, 1H, $J = 7.0$ Hz), 7.28 (m, 2H), 5.35 (m, 1H), 4.98 (dd, 1H, $J_1 = 5.0$ Hz, $J_2 = 13.5$ Hz), 4.75 (dd, 1H, $J_1 = 9.5$ Hz, $J_2 = 13.0$ Hz), 3.91 (s, 3H), 3.60 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ 164.9, 164.7, 164.0, 163.8, 135.0, 131.3, 130.3, 130.1, 129.1, 127.6, 95.7, 94.0, 74.92, 74.88, 54.1, 53.4, 42.4, 42.2; $[\alpha]_{\text{D}}^{23} = +7.3$ ($c = 4.0$, CHCl_3); HPLC (Daicel CHIRALPAK AS-H, Hexane/2-PrOH = 70:30, flow rate 0.5 mL/min, $\lambda = 235$ nm); $t_{\text{R}} = 18.25$ (major), 21.86 (minor) min, ee = 86%.

General Procedure for synthesis of Δ^1 -pyrrolidines: The fluorine-containing Michael addition product (2S,3S)-ethyl 2-acetyl-3-(4-chlorophenyl)-2-fluoro-4-nitrobutanoate can be converted into the corresponding functional Δ^1 -pyrrolidines under H_2 balloon with catalytic amount Raney Ni in EtOH.



(4S) Ethyl 4-(4-chlorophenyl)-3-fluoro-2-methyl-4,5-dihydro-3H-pyrrole-3-carboxylate (4): Yield: 80%; ^1H NMR (500 MHz, CDCl_3): δ 7.29 (d, 2H, $J = 8.5$ Hz), 7.18 (d, 2H, $J = 8.5$ Hz), 4.36 (dd, 1H, $J_1 = 8.0$ Hz, $J_2 = 15.0$ Hz), 4.19 (m, 1H), 3.93 (t, 2H, $J = 7.0$ Hz), 3.85-3.98 (m, 1H), 2.10 (s, 3H), 0.96 (t, 3H, $J = 7.0$ Hz); ^{13}C NMR (125 MHz, CDCl_3): δ 170.2, 170.1, 166.1, 165.9, 133.7, 129.3, 128.7, 107.2, 105.6, 61.9, 61.7, 61.6, 53.9, 53.7, 15.7, 13.7; $[\alpha]_{\text{D}}^{23} = -75.3$ ($c = 2.0$, CHCl_3); HPLC (Daicel CHIRALCEL OJ-H, hexane/2-PrOH = 93:7, flow rate 0.5 mL/min, $\lambda = 220$ nm); $t_{R\text{major}} = 24.04$ (major), 31.40 (minor) min; $t_{R\text{minor}} = 29.34$ (minor), 80.88 (major) min.

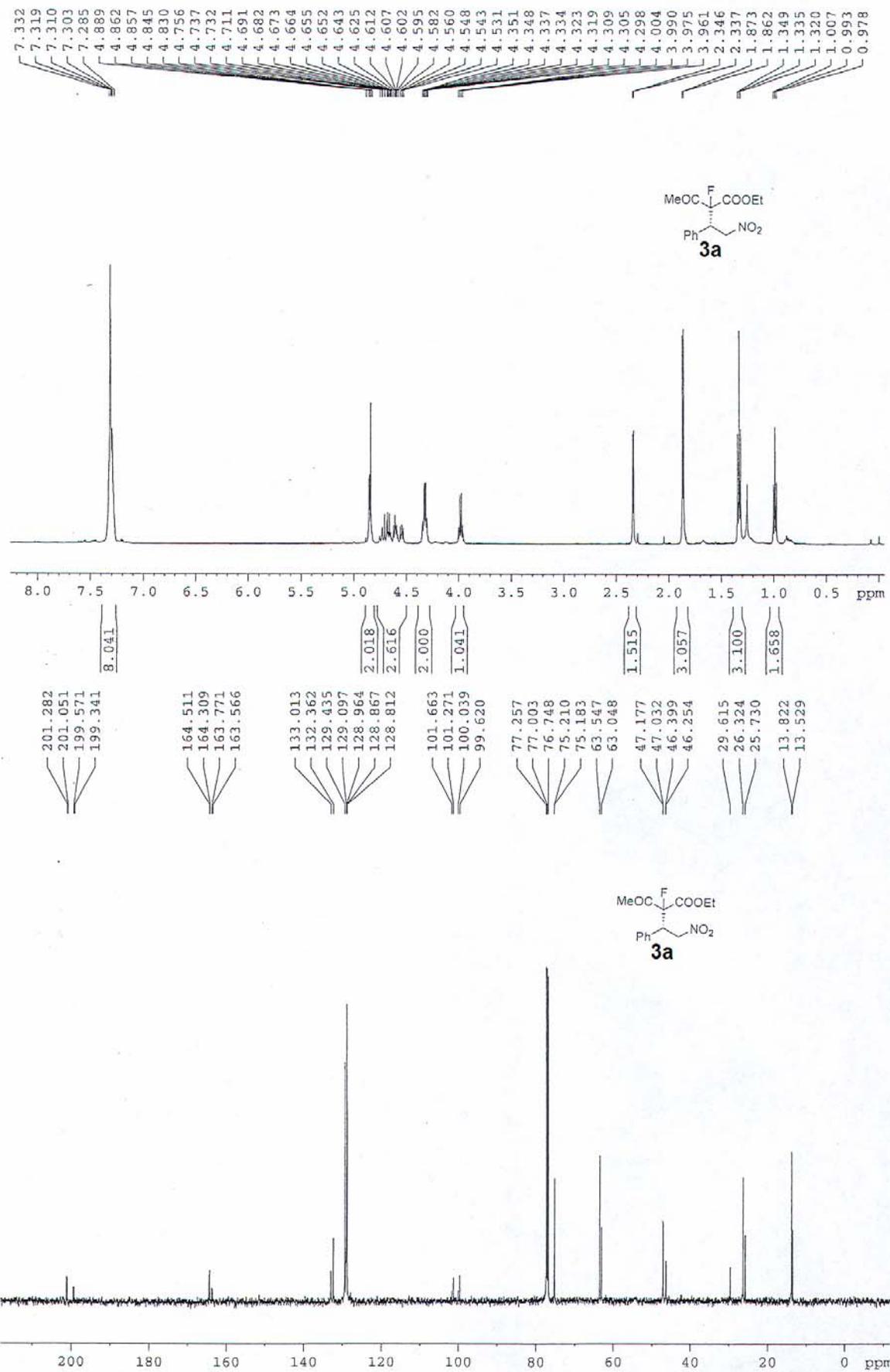
Crystallography Report

Experimental for compound 3n

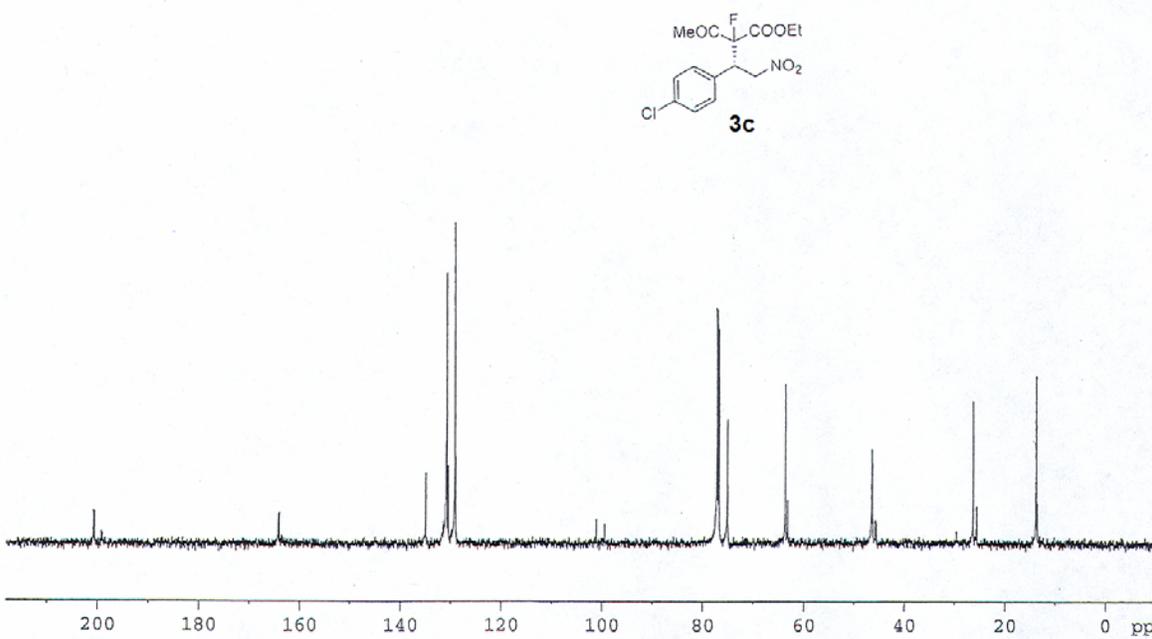
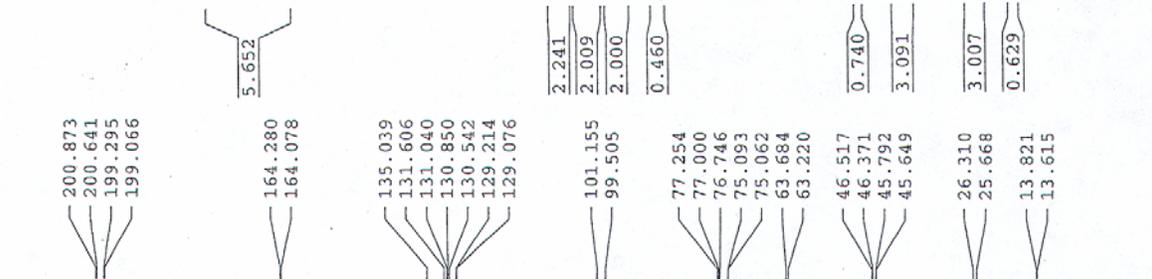
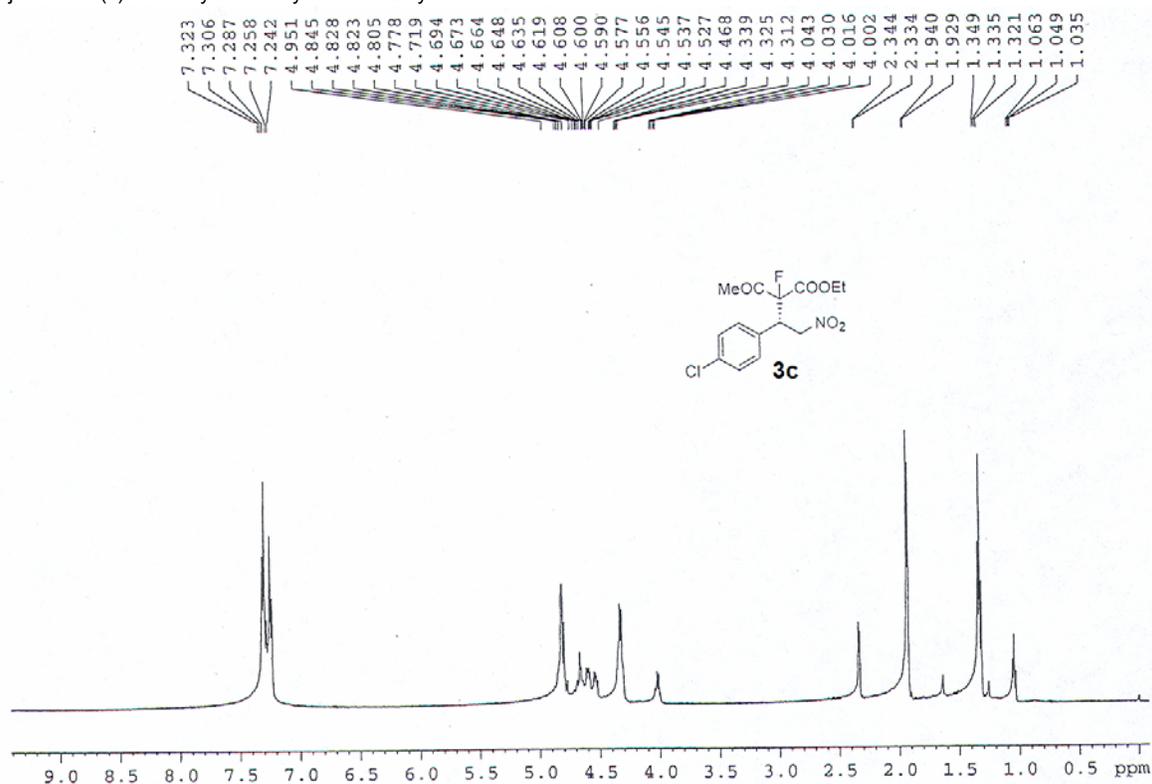
Large colorless crystals were submitted. These were plates, largest about 2mm long, 1mm wide. These are moderate diffractors. The crystals were cut to suitable dimensions. A crystal of dimensions 0.460 x 0.460 x 0.500 mm³ was mounted on a standard Bruker X8 Apex2 CCD-based X-ray diffractometer equipped with an Oxford Cryostream 700 low temperature device, graphite monochromator and normal focus Mo-target X-ray tube ($\lambda = 0.71073 \text{ \AA}$) operated at 1500 W power (50 kV, 30 mA). The X-ray intensities were measured at 225(2) K; the detector was placed at a distance 4.5 cm from the crystal. A full sphere of data consisting of 3138 frames was collected with a scan width of 0.5° in ω and ϕ with an exposure time of 20 s/frame. The data collection time was 25 hrs. The frames were integrated with the Bruker SAINT software package with a narrow frame algorithm. Preliminary cell constants showed a triclinic cell which could be monoclinic [two cell angles close to 90°]. For completeness, the data were collected in the triclinic system. The integration of the data yielded a total 21749 reflections to a maximum 2θ value of 64.40 ° of which 18419 were independent. The final cell constants (Table 1) were based on the xyz centroids of 7017 reflections above $10\sigma(I)$. Analysis of the data showed negligible decay during data collection; the data were processed with SAINT, SADABS and corrected for absorption. The structure was solved and refined with the Bruker SHELXTL (version 6.12) software package, using the chiral monoclinic space group P2(1). The absolute configuration was determined from anomalous dispersion effects of Cl. All non-hydrogen atoms were refined anisotropically. Hydrogen atoms were included in ideal positions with fixed isotropic U's set to 1.2U_{eq} of parent atom. The molecule is chiral with assignment for C7 as S. The structure shows no unusual bond lengths or bond angles. There are several possible hydrogen bond between CH—Cl or CH—O, CH—F. However, hydrogen bonds between C-H and X are considered very weak hydrogen bonds.

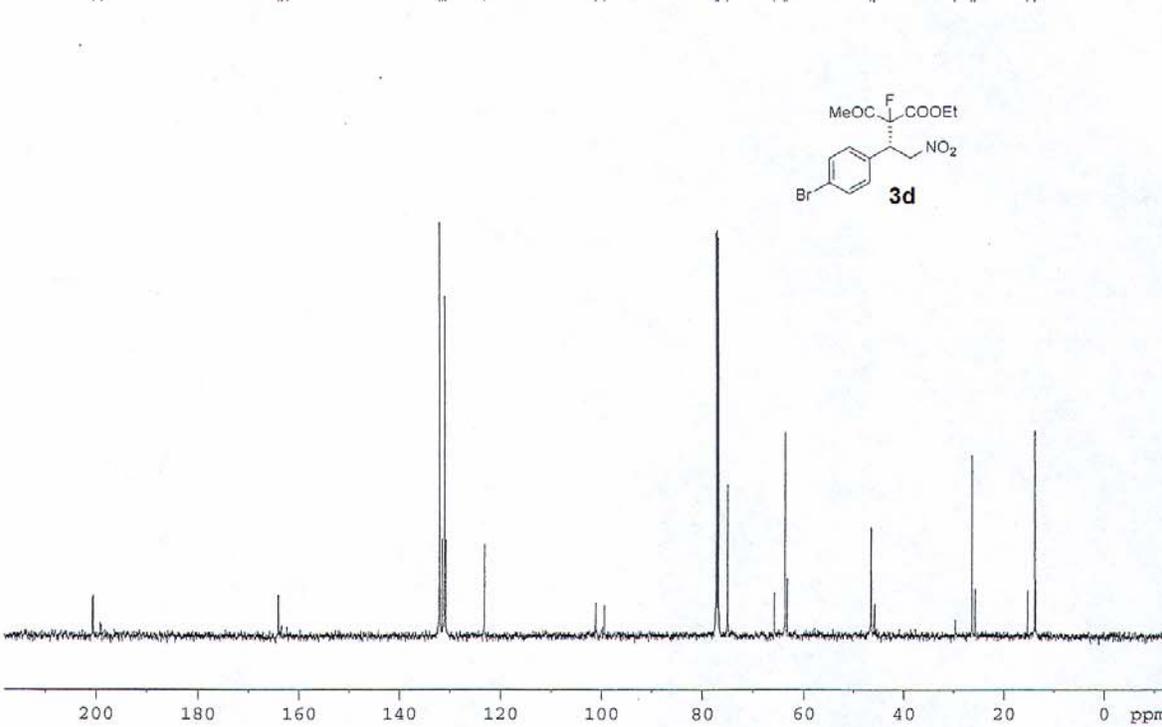
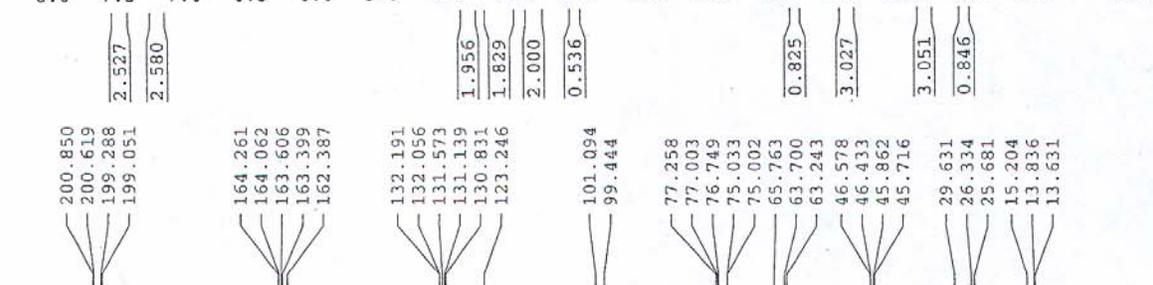
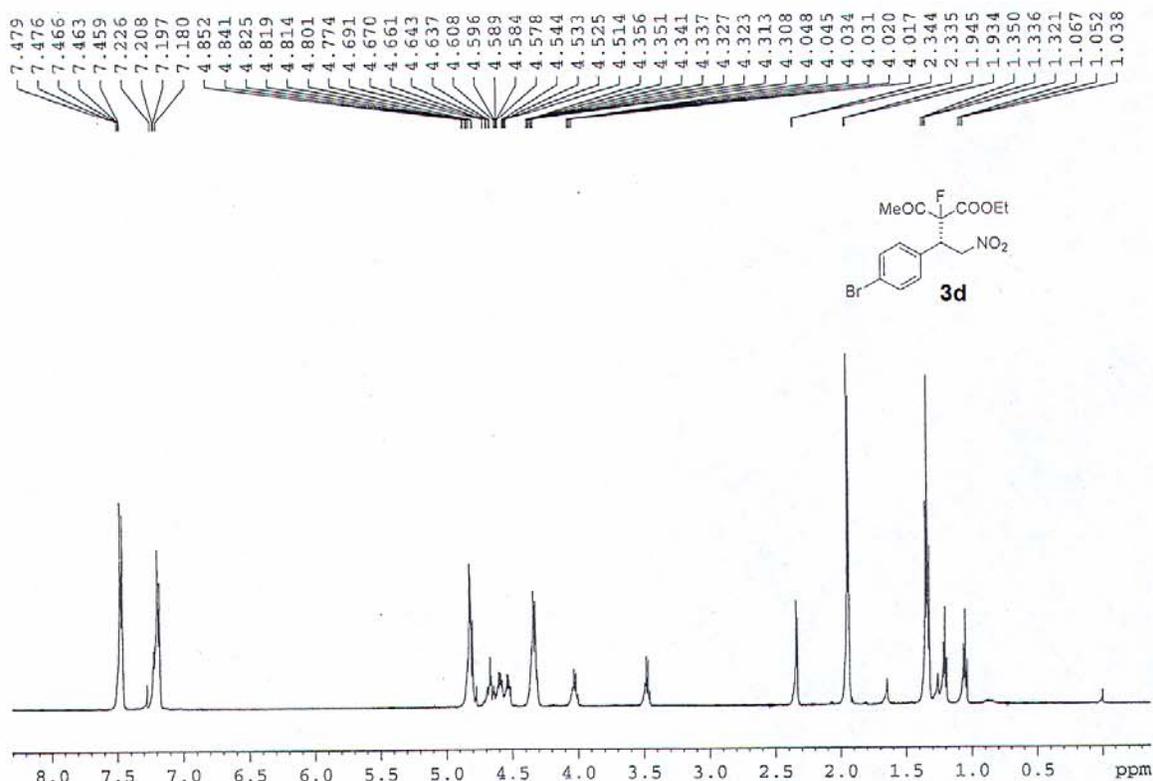
Table S1. Crystal data and structure refinement for **3n**.

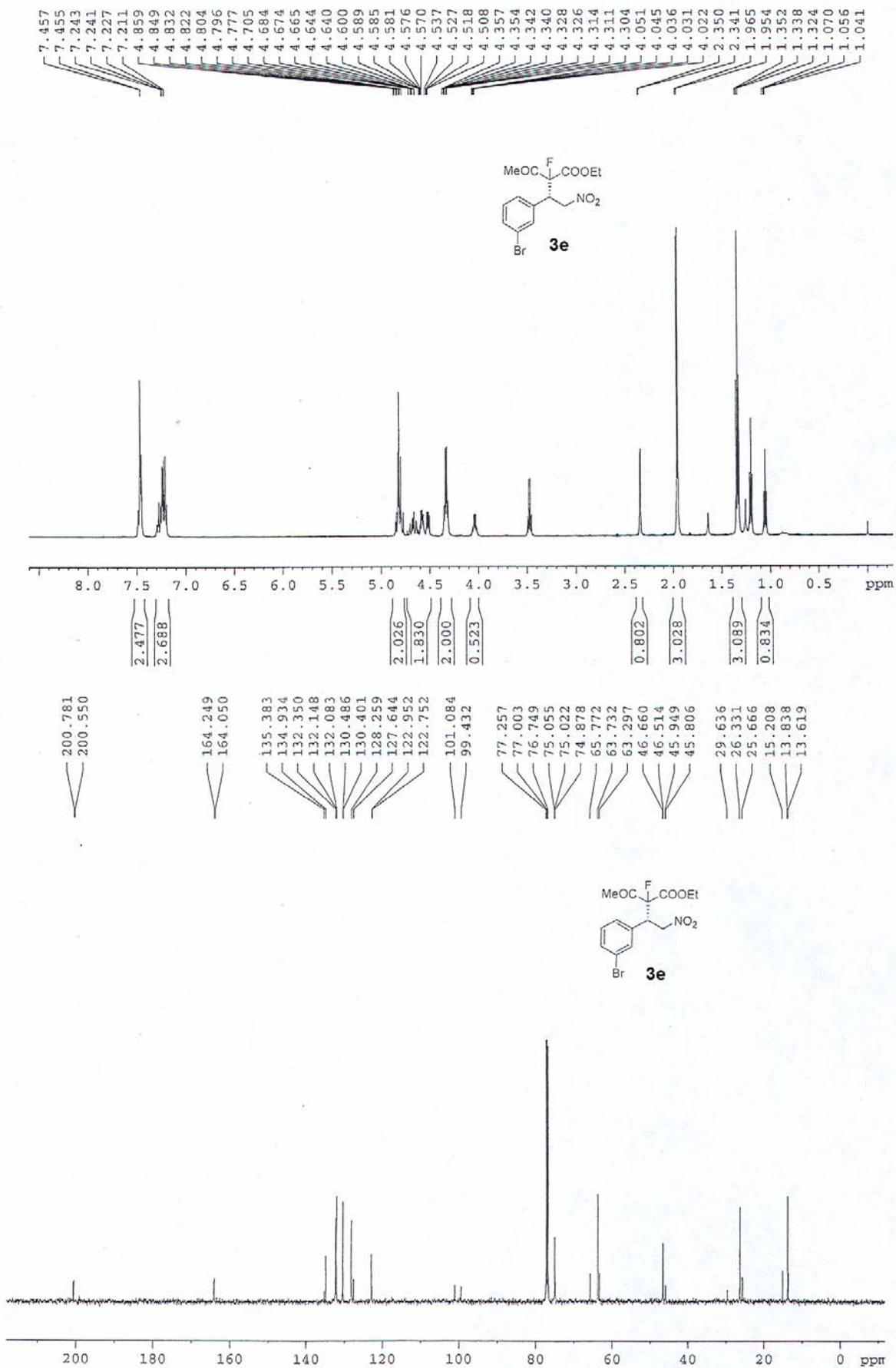
Identification code	ww77	
Empirical formula	C ₁₃ H ₁₃ Cl F N O ₆	
Formula weight	333.69	
Temperature	225(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)	
Unit cell dimensions	a = 9.2128(6) Å	α = 90°.
	b = 7.5548(5) Å	β = 112.449(4)°.
	c = 11.3782(8) Å	γ = 90°.
Volume	731.92(9) Å ³	
Z	2	
Density (calculated)	1.514 Mg/m ³	
Absorption coefficient	0.301 mm ⁻¹	
F(000)	344	
Crystal size	0.50 x 0.46 x 0.46 mm ³	
Theta range for data collection	3.61 to 32.19°.	
Index ranges	-13 ≤ h ≤ 13, -11 ≤ k ≤ 11, -17 ≤ l ≤ 17	
Reflections collected	21749	
Independent reflections	5088 [R(int) = 0.0235]	
Completeness to theta = 32.19°	99.1 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.8739 and 0.8641	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	5088 / 1 / 201	
Goodness-of-fit on F ²	1.025	
Final R indices [I > 2σ(I)]	R1 = 0.0341, wR2 = 0.0834	
R indices (all data)	R1 = 0.0405, wR2 = 0.0875	
Absolute structure parameter	0.07(5)	
Largest diff. peak and hole	0.239 and -0.327 e.Å ⁻³	

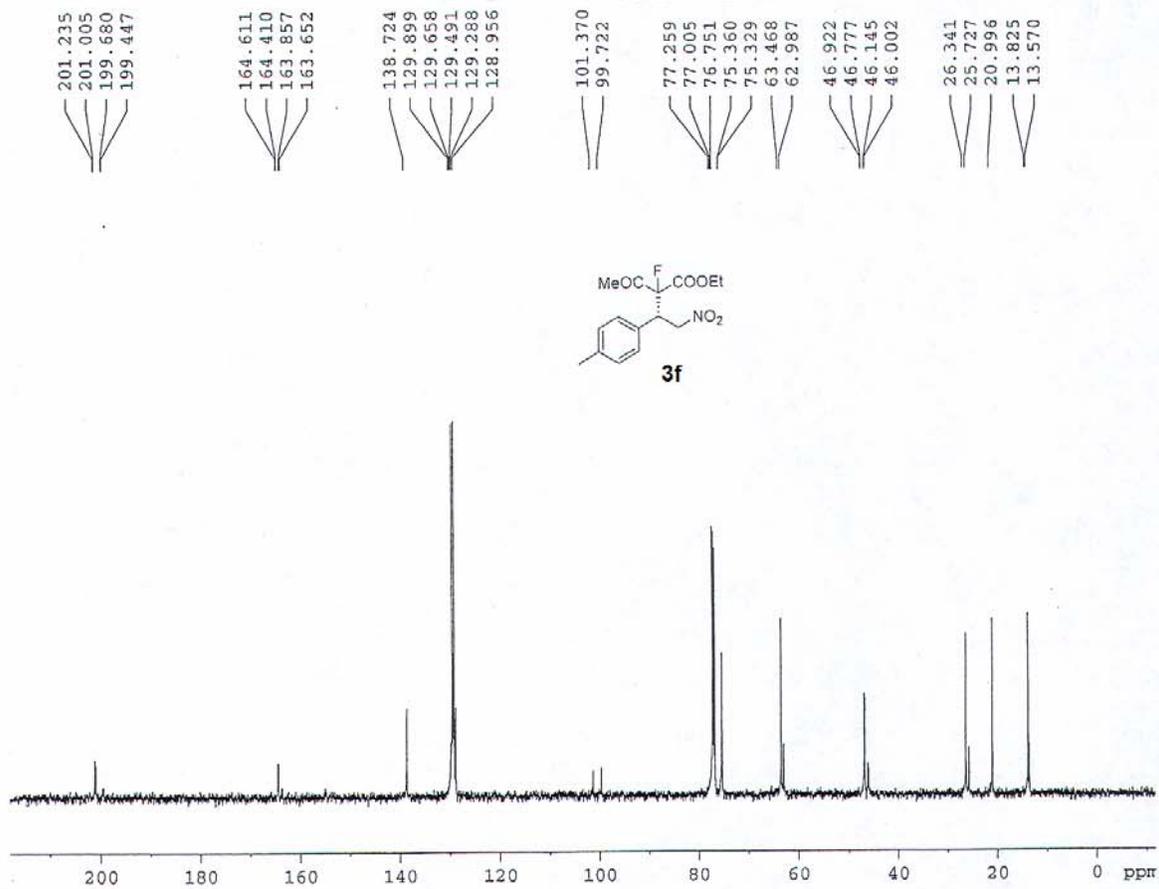
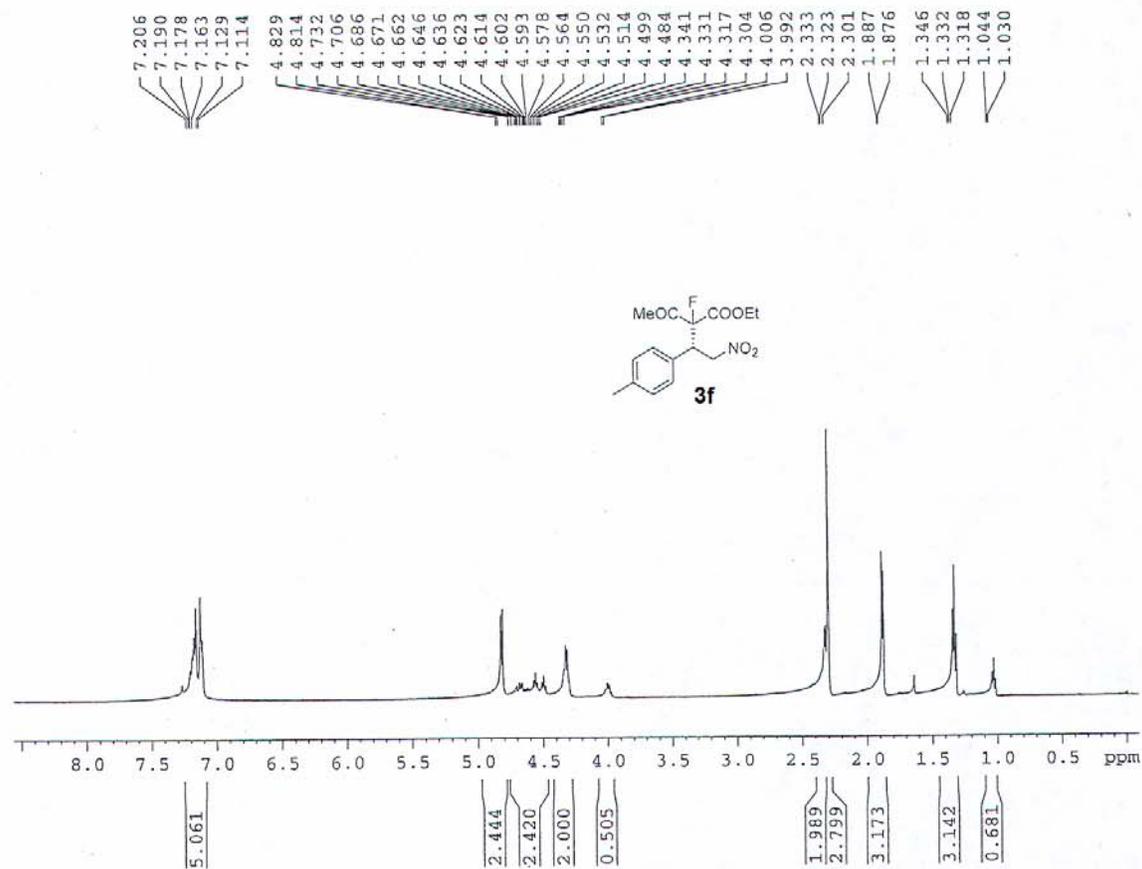


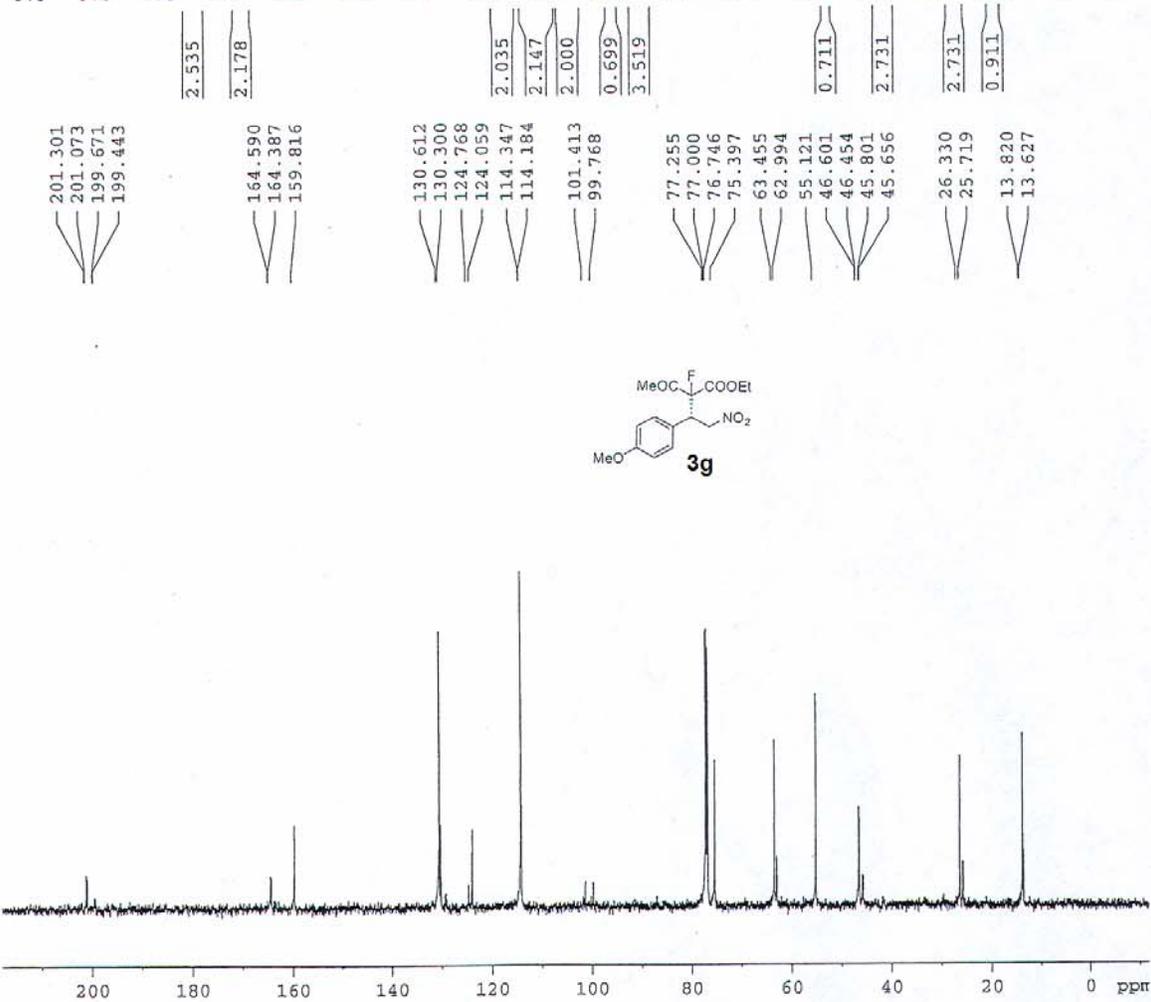
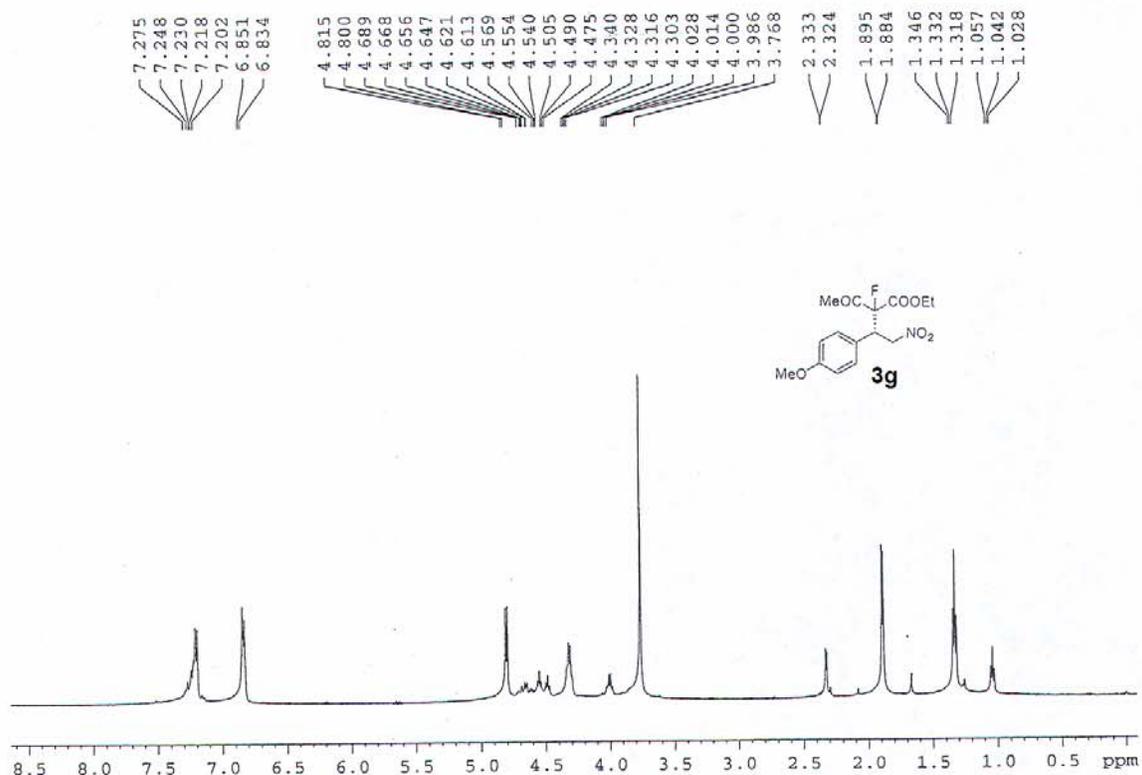


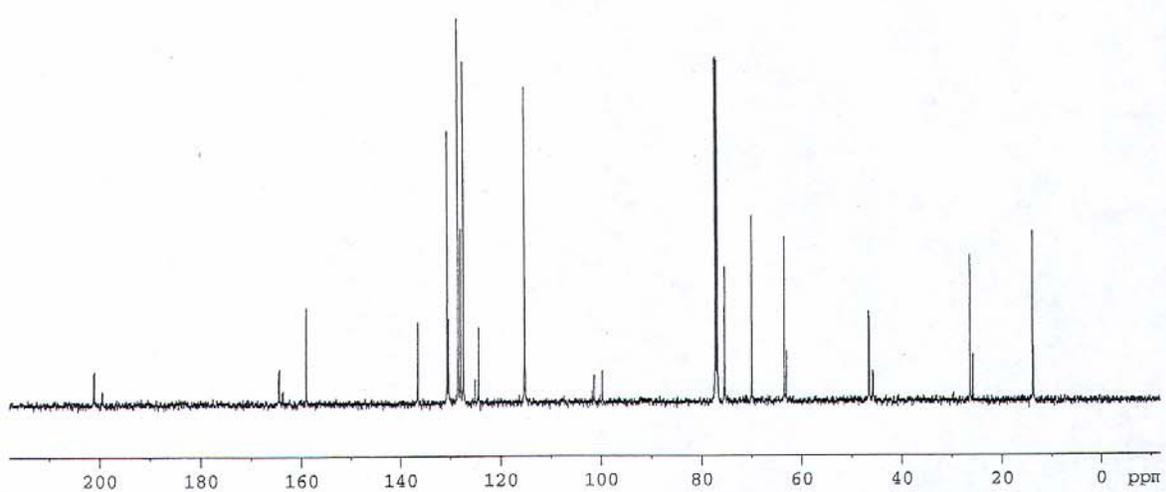
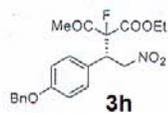
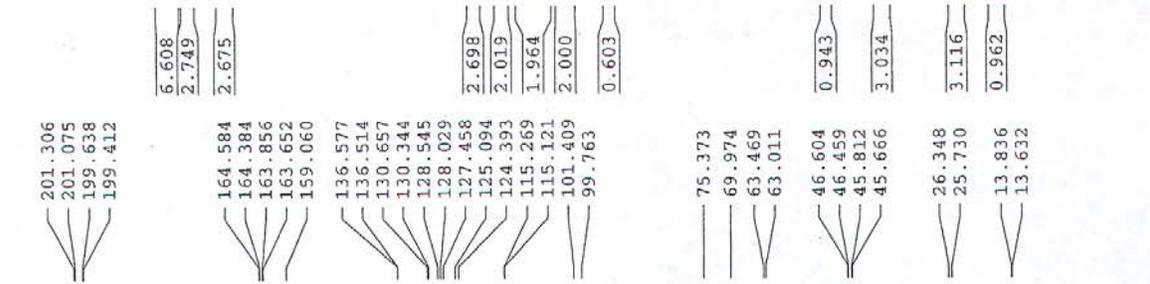
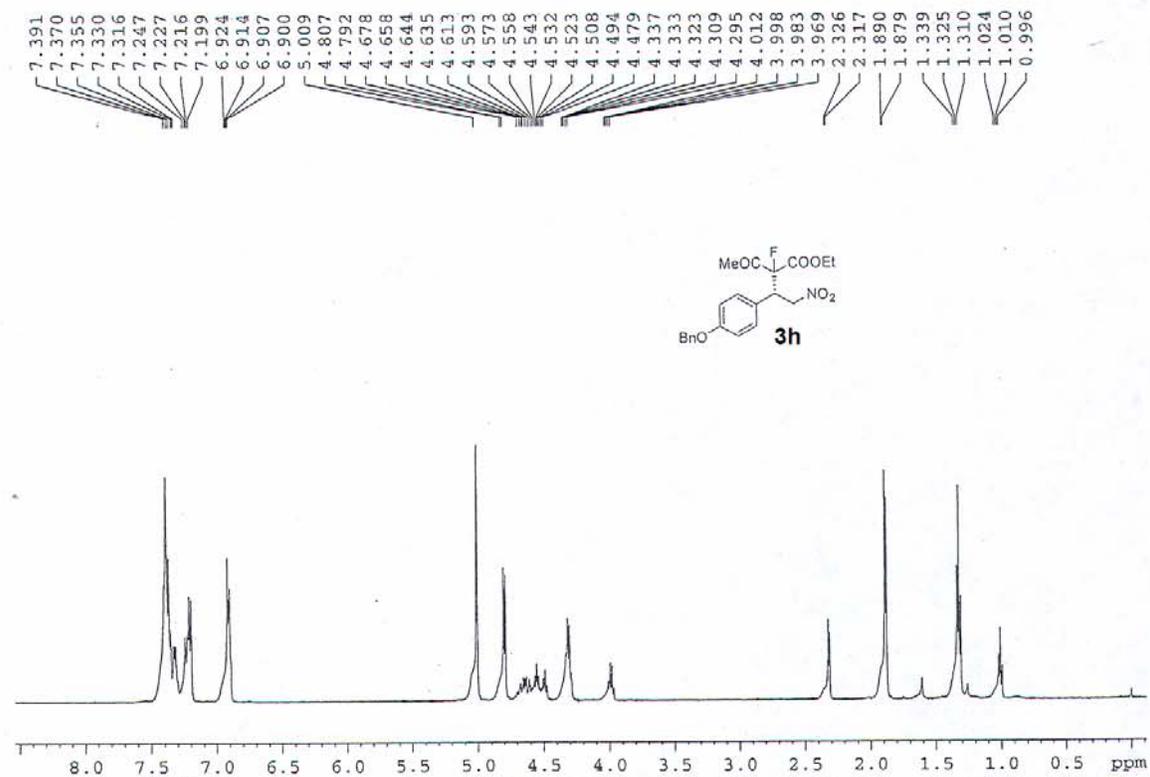


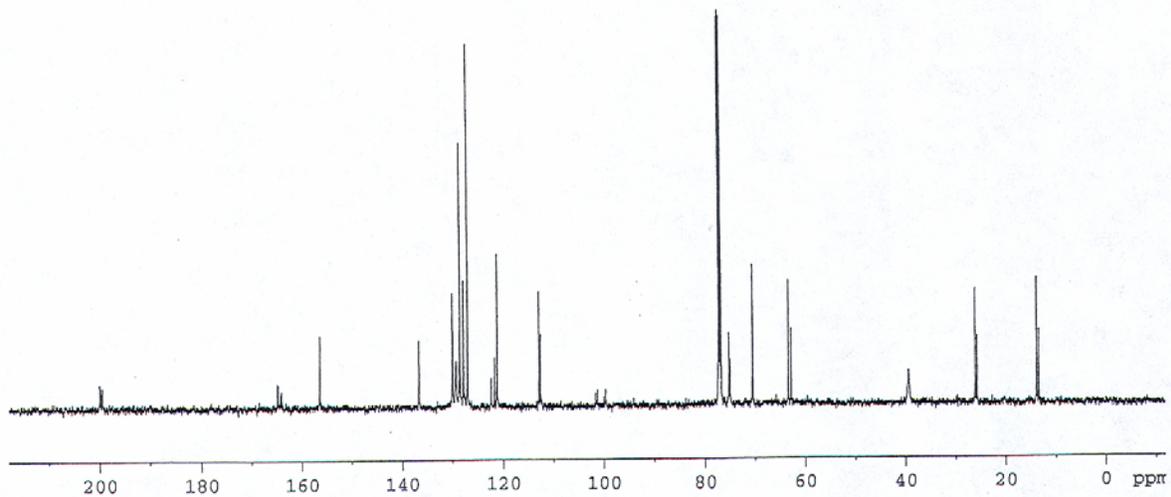
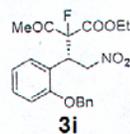
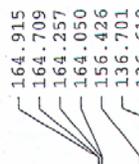
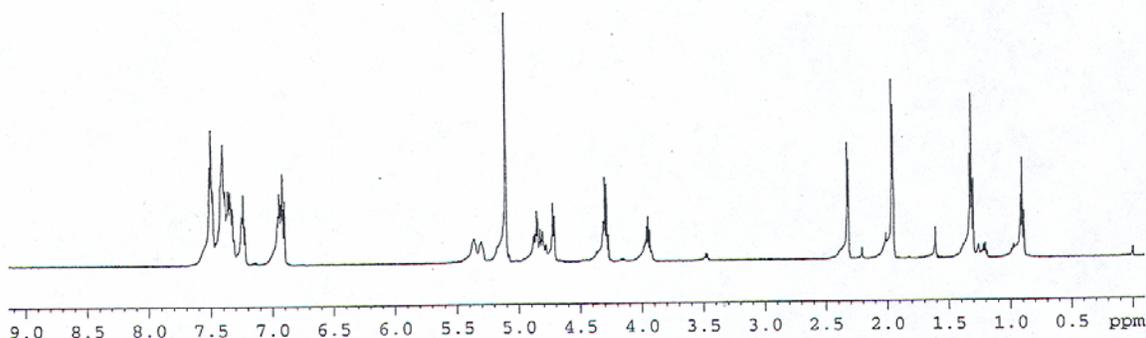
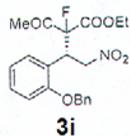
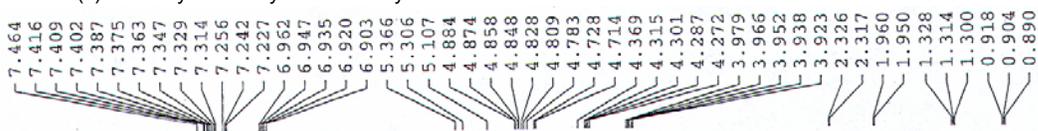


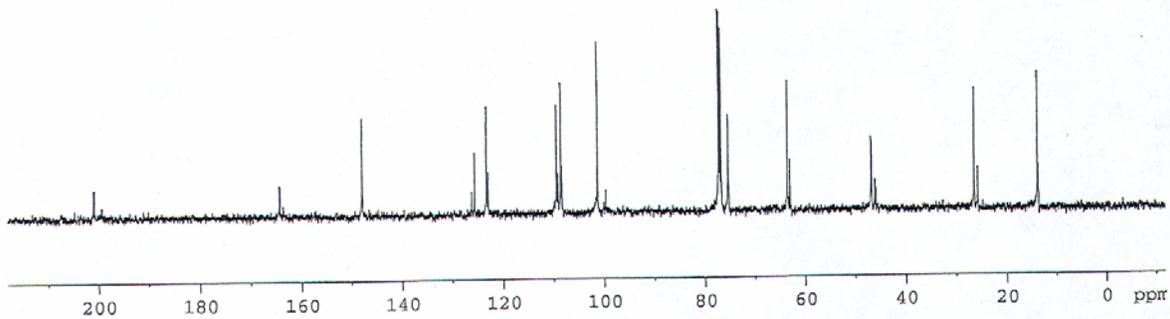
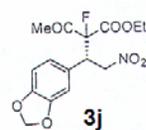
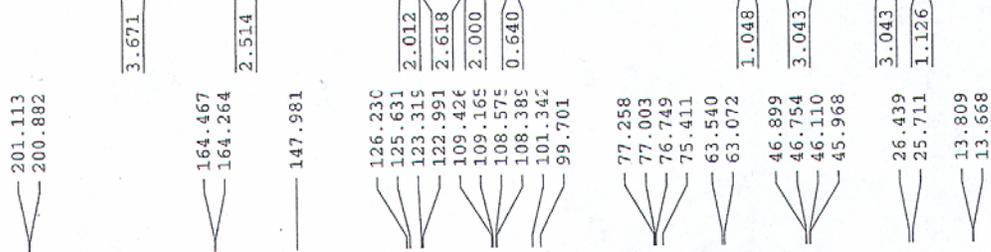
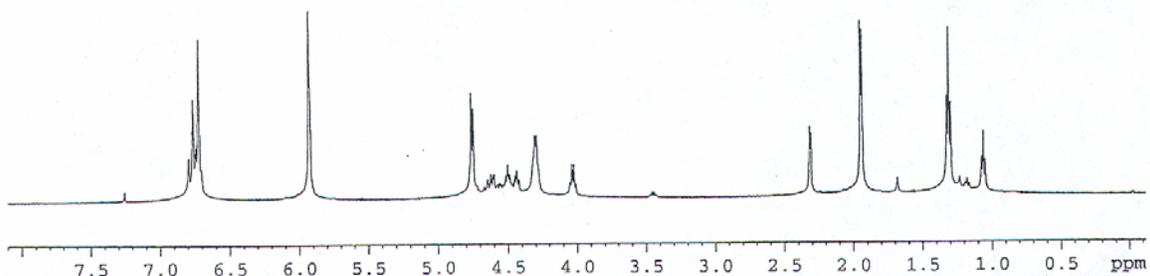
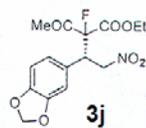
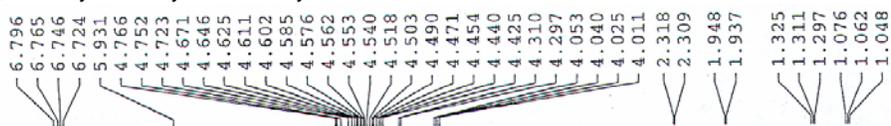


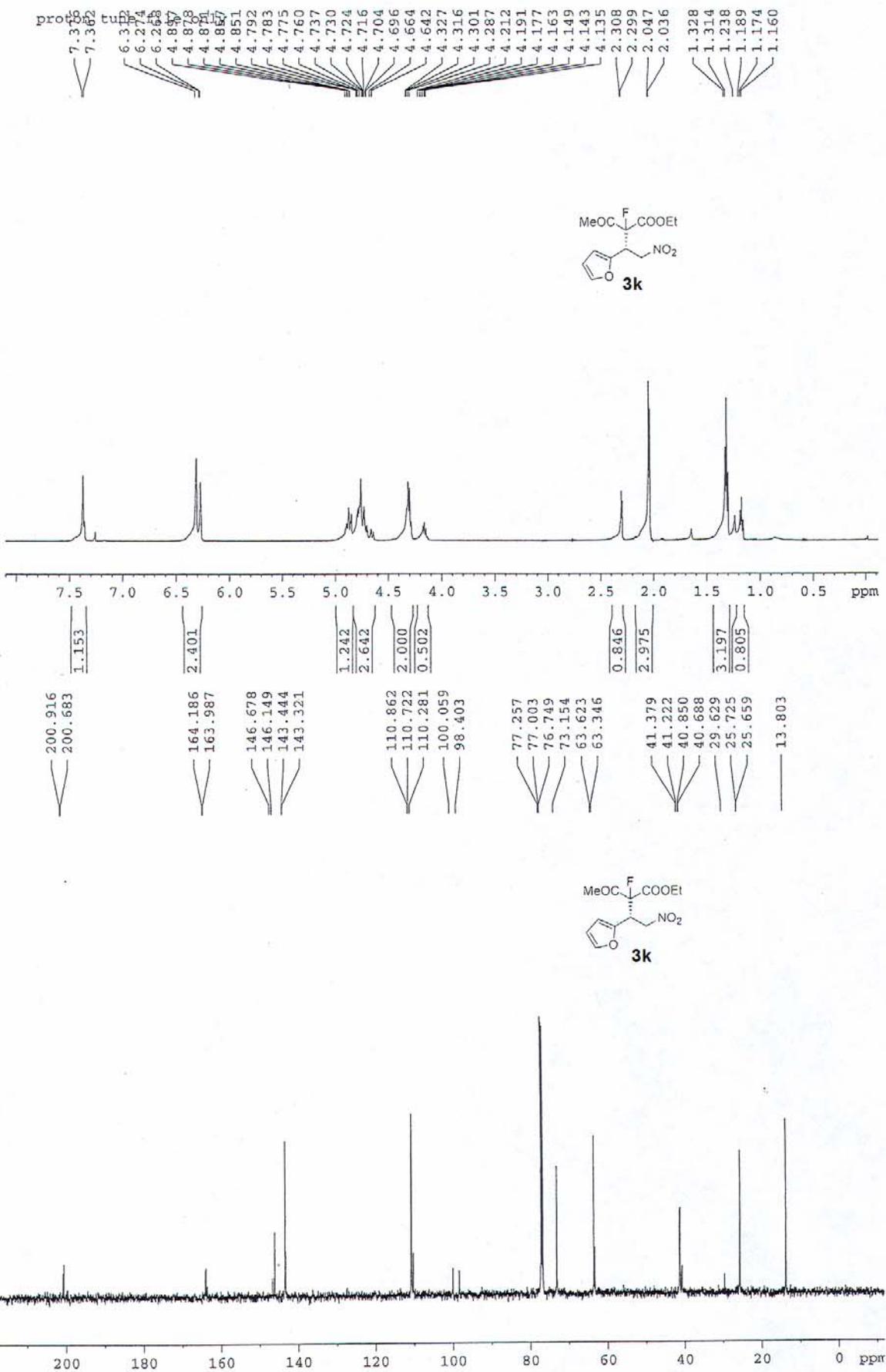


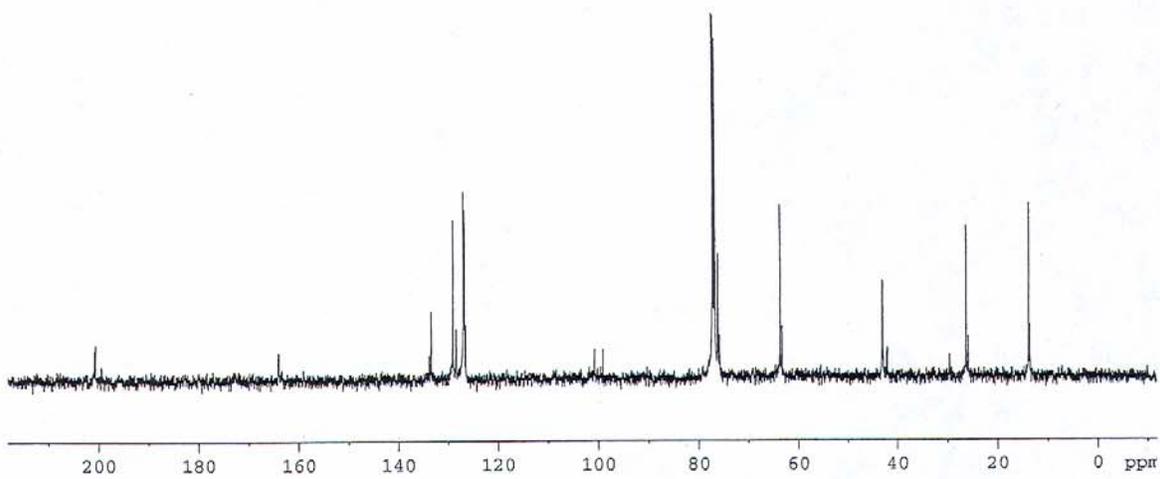
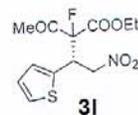
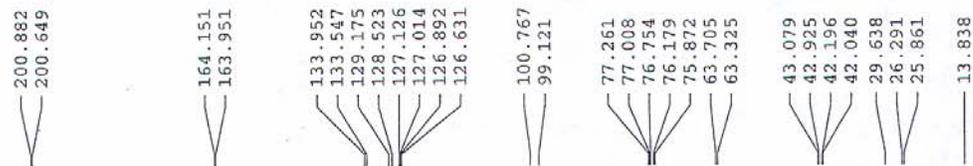
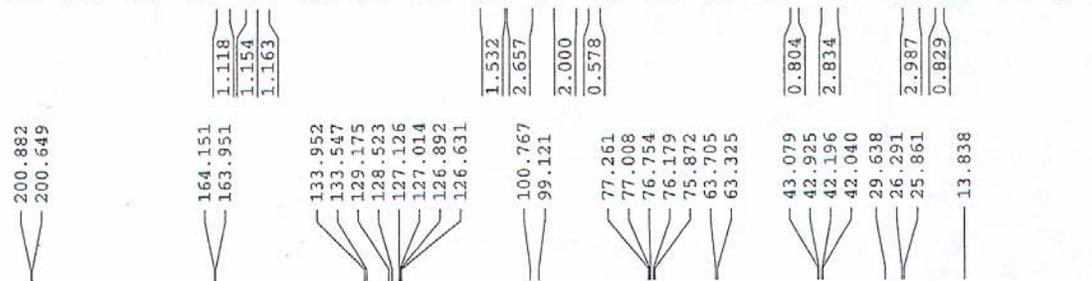
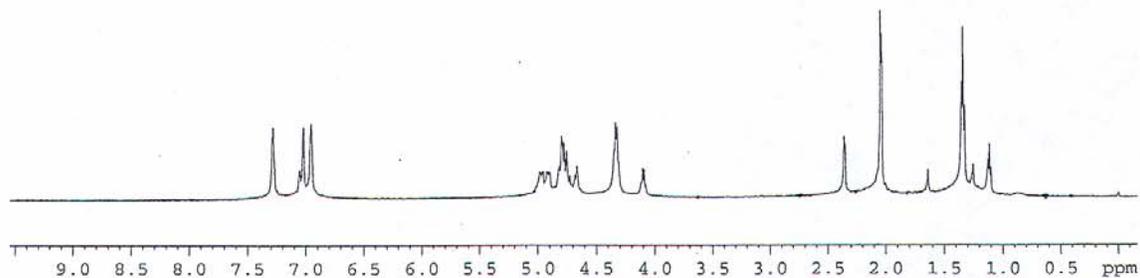
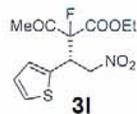
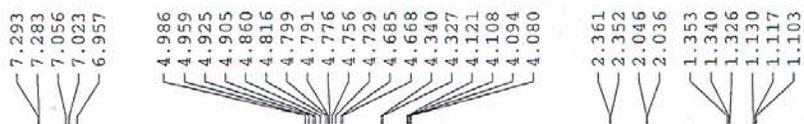


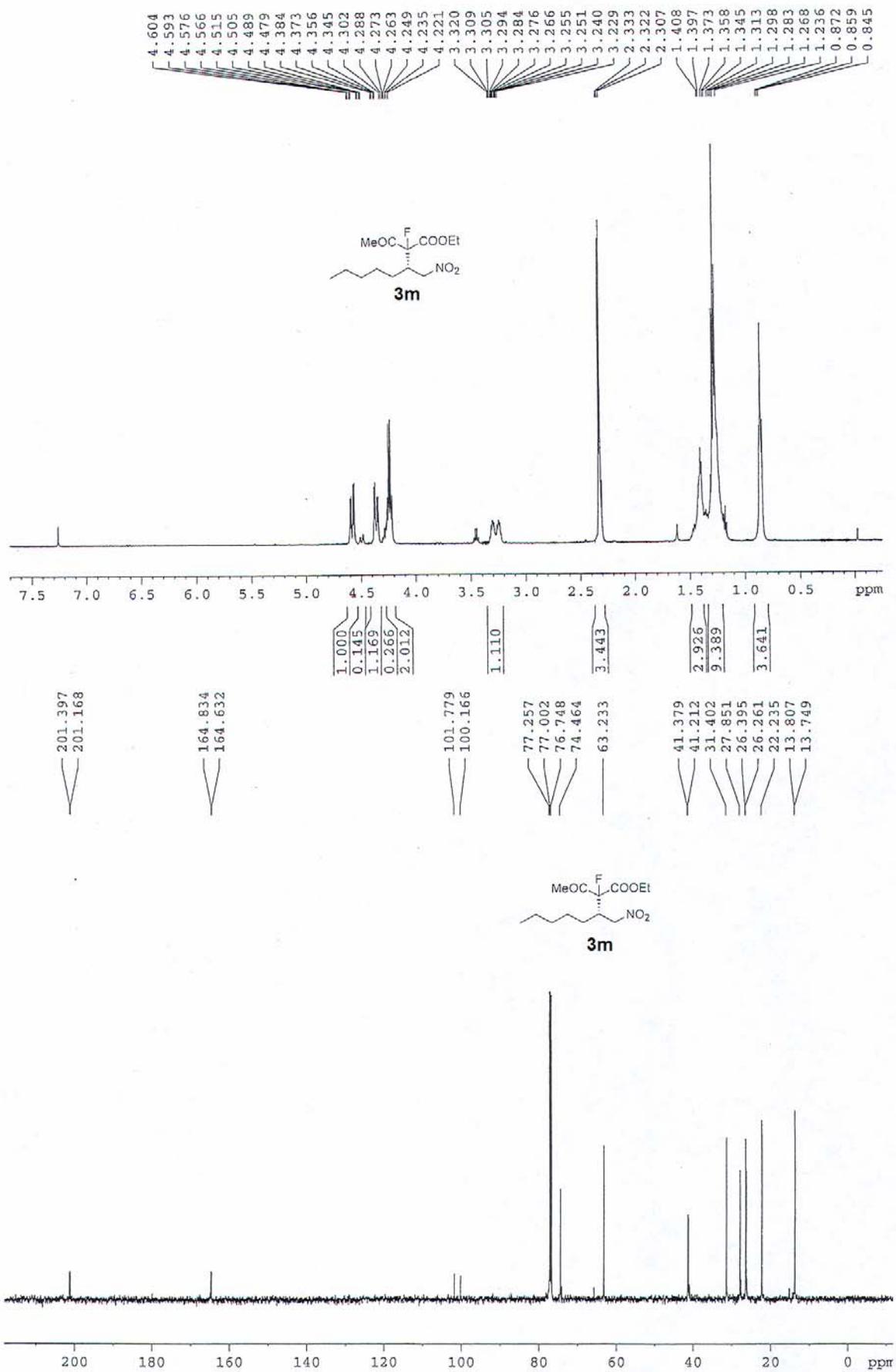


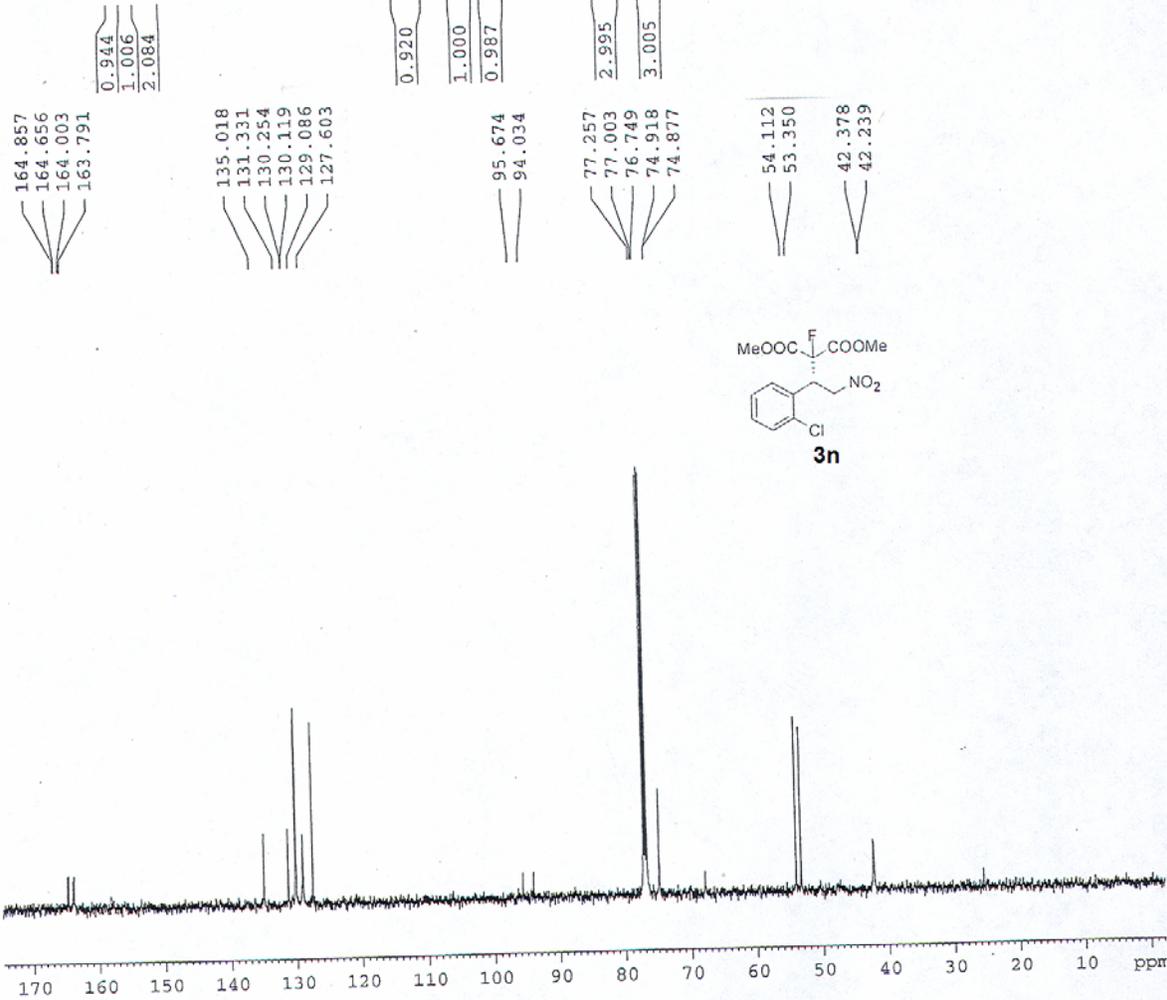
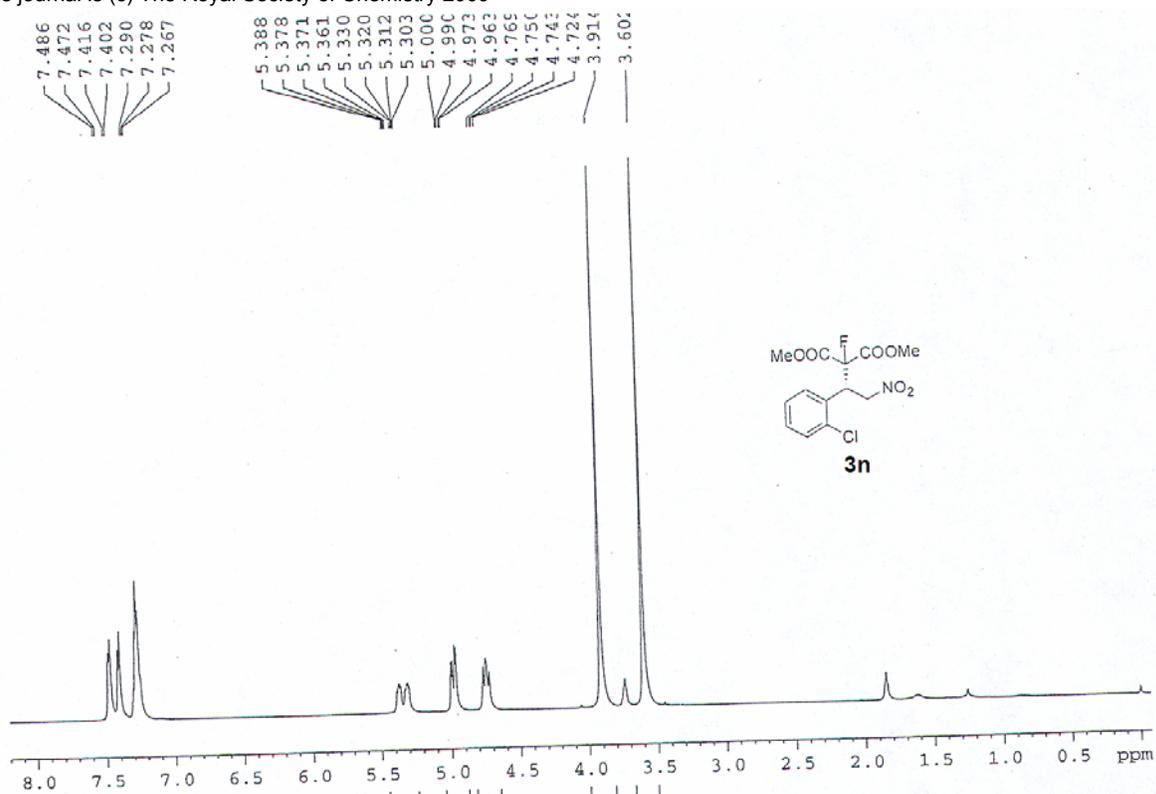


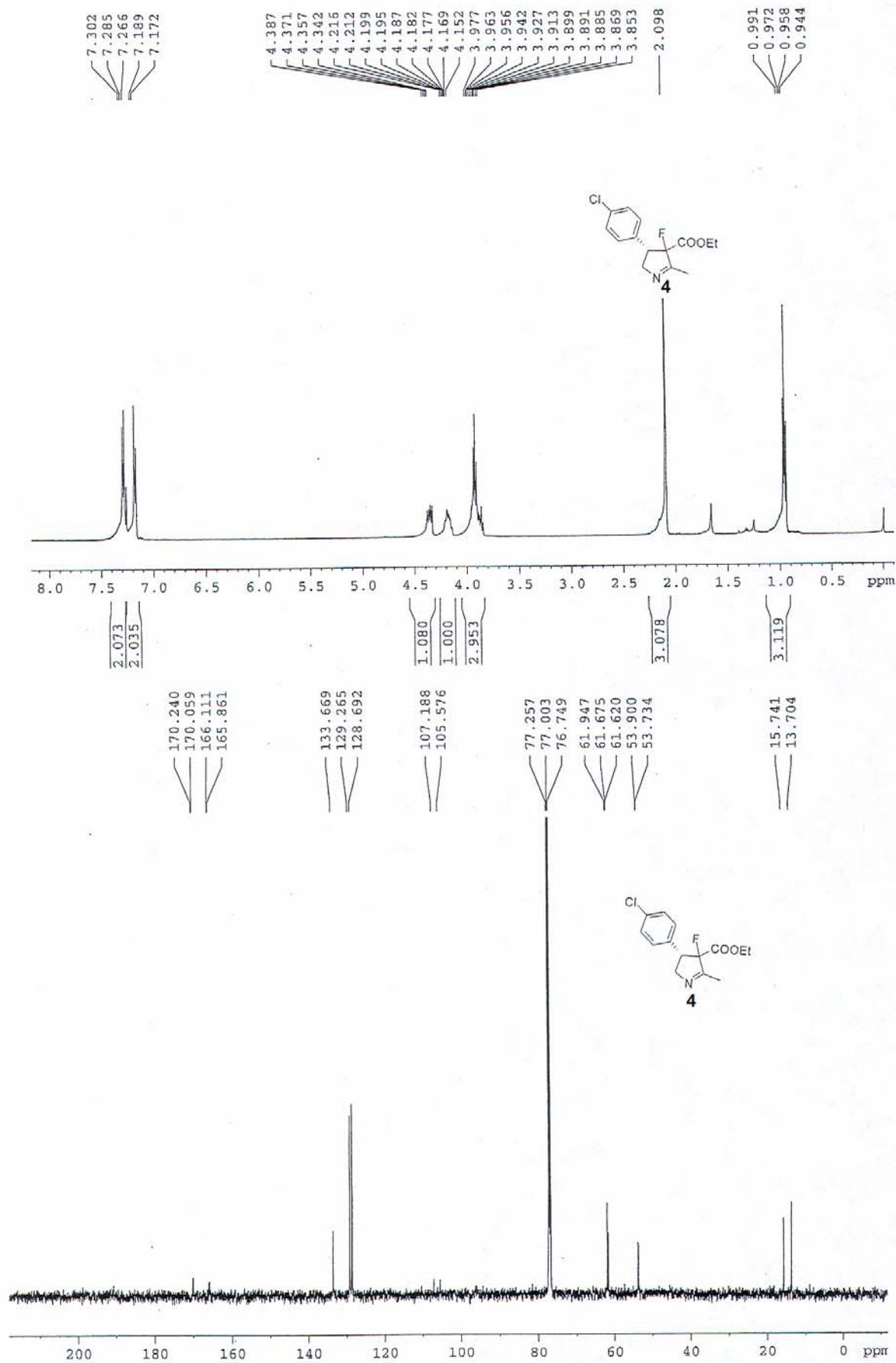






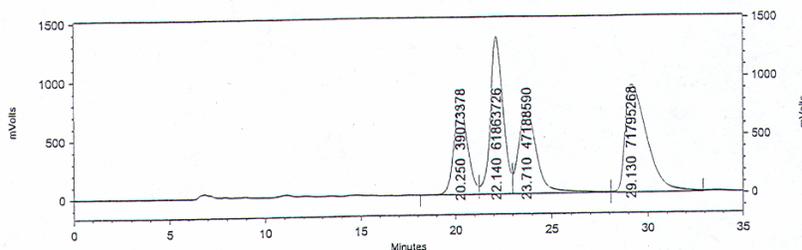
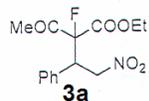






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Department of Chemistry

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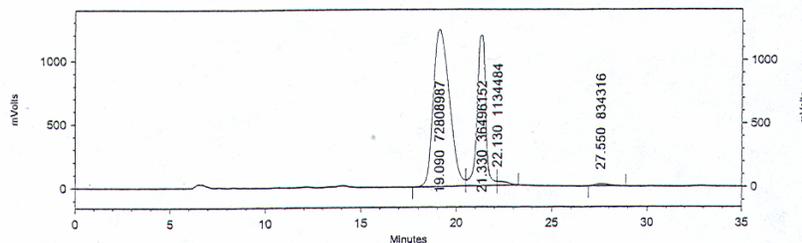
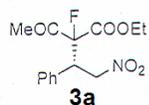


SPD-10Avp
Ch1-220nm Results

PK #	RT	Area	Area %
1	20.250	39073378	17.767
2	22.140	61863726	28.130
3	23.710	47188590	21.457
4	29.130	71795268	32.646
Totals		219920962	100.000

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Method Name: C:\EZStart\Projects\WeiWang\aa0.met
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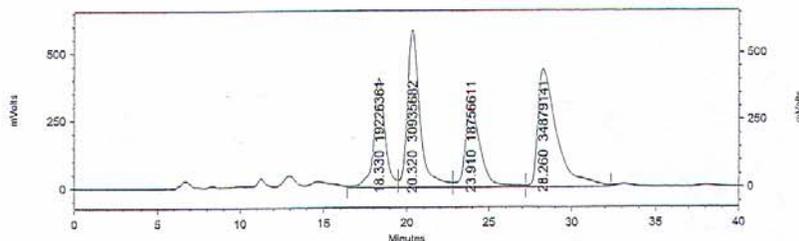
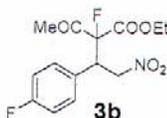


SPD-10Avp
Ch1-220nm Results

PK #	RT	Area	Area %
1	19.090	72808987	65.432
2	21.330	36496152	32.798
3	22.130	1134484	1.020
4	27.550	834316	0.750
Totals		111273939	100.000

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Method Name: C:\EZStart\Projects\WeiWang\aa0.met
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SPD-10Avp

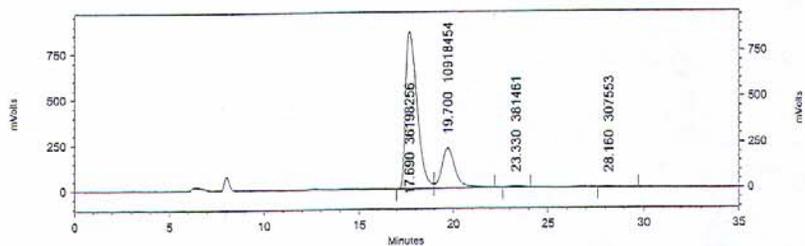
Ch1-220nm Results

PK #	RT	Area	Area %
1	18.330	19226361	18.523
2	20.320	30935682	29.804
3	23.910	18756611	18.070
4	28.260	34879141	33.603

Totals		103797795	100.000
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Method Name: C:\EZStart\Projects\WeiWang\aa4.met
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SPD-10Avp

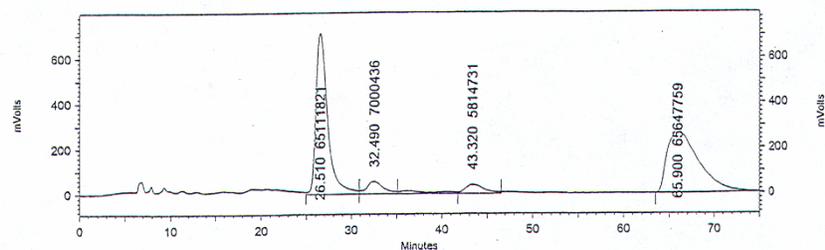
Ch1-220nm Results

PK #	RT	Area	Area %
1	17.690	36198256	75.720
2	19.700	10918454	22.839
3	23.330	381461	0.798
4	28.160	307553	0.643

Totals		47805724	100.000
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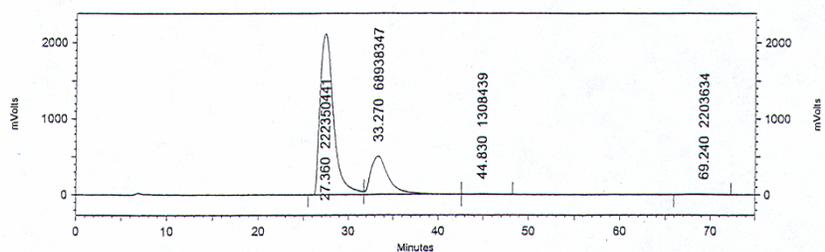


SPD-10Avp
Ch1-220nm Results

Pk #	RT	Area	Area %
1	26.510	65111821	45.350
2	32.490	7000436	4.876
3	43.320	5814731	4.050
4	65.900	65647759	45.724
Totals		143574747	100.000

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Method Name: C:\EZStart\Projects\WeiWang\zls1256-4.met
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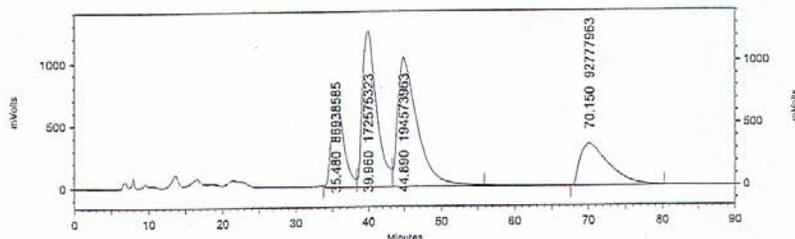


SPD-10Avp
Ch1-220nm Results

Pk #	RT	Area	Area %
1	27.360	222350441	75.424
2	33.270	68938347	23.385
3	44.830	1308439	0.444
4	69.240	2203634	0.747
Totals		294800861	100.000

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Method Name: C:\EZStart\Projects\WeiWang\zls1268.met
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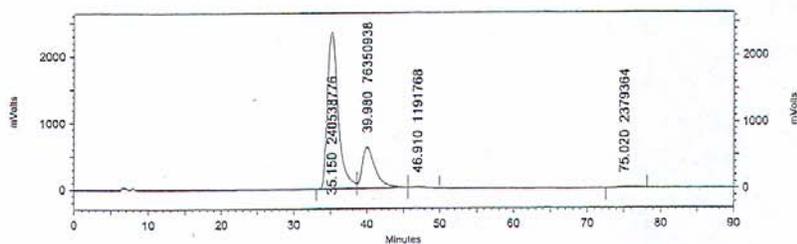
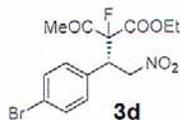


SPD-10Avp
 Chl-220nm Results

Pk #	RT	Area	Area %
1	35.480	86938585	15.898
2	39.960	172575323	31.557
3	44.890	194573963	35.580
4	70.150	92777963	16.965
Totals		546865834	100.000

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 Department of Chemistry

Method Name: C:\EZStart\Projects\WeiWang\aa4.met
 Data File: C:\EZStart\Projects\WeiWang\hlv17d1.dat
 Date Acquired: 8/5/2007 1:36:24 PM Date Printed: 01/17/2008 03:50:10 PM
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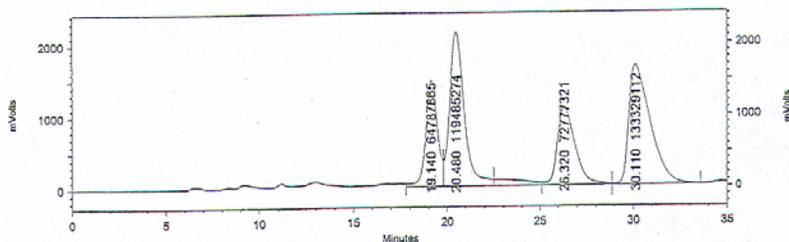
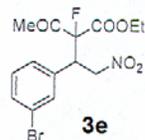


SPD-10Avp
 Chl-220nm Results

Pk #	RT	Area	Area %
1	35.150	240538776	75.060
2	39.980	76350938	23.825
3	46.910	1191768	0.372
4	75.020	2379364	0.742
Totals		320460846	100.000

University Of New Mexico
 Department of Chemistry

Method Name: C:\EZStart\Projects\WeiWang\aa0.met
 Data File: C:\EZStart\Projects\WeiWang\hlu77d1.dat
 Date Acquired: 7/26/2007 11:50:07 AM Date Printed: 01/17/2008 03:52:29 PM
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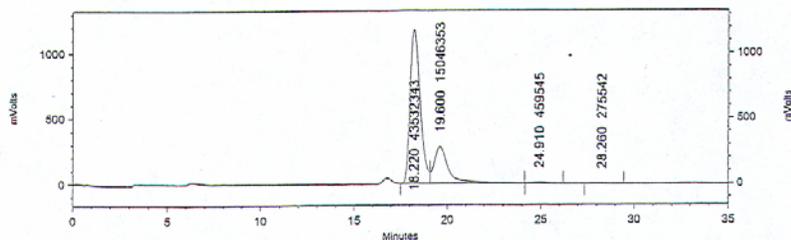
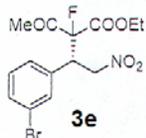


SPD-10Avp
 Ch1-220nm Results

Pk #	RT	Area	Area %
1	19.140	64787865	16.596
2	20.480	119485274	30.607
3	26.320	72777321	18.643
4	30.110	133329112	34.154
Totals		390379572	100.000

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 Department of Chemistry

Method Name: C:\EZStart\Projects\WeiWang\aa4.met
 Data File: C:\EZStart\Projects\WeiWang\hlv18d2.dat
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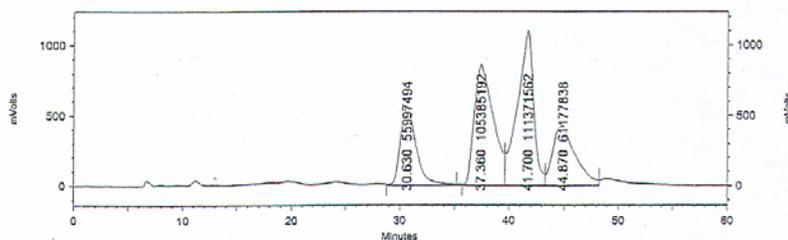
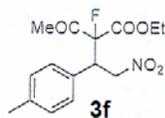


SPD-10Avp
 Ch1-220nm Results

Pk #	RT	Area	Area %
1	18.220	43532343	73.393
2	19.600	15046353	25.367
3	24.910	459545	0.775
4	28.260	275542	0.465
Totals		59313783	100.000

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 Department of Chemistry

Method Name: C:\EZStart\Projects\WeiWang\aa0.met
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 Sample ID: hlu78d

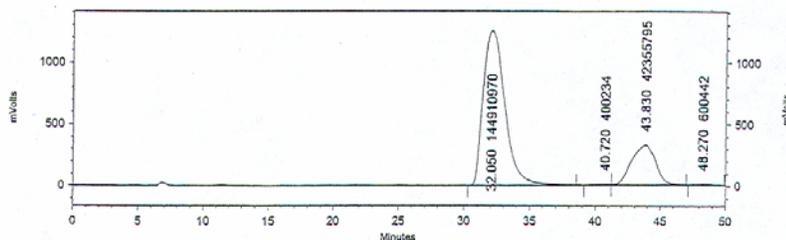
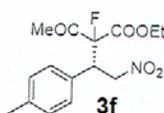


SPD-10Avp
 Ch1-220nm Results

PK #	RT	Area	Area %
1	30.630	55997494	16.789
2	37.360	105385192	31.559
3	41.700	111371562	33.352
4	44.870	61177838	18.320
Totals		333932086	100.000

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 Department of Chemistry

Method Name: C:\EZStart\Projects\WeiWang\zls1256-4.met
 Data File: C:\EZStart\Projects\WeiWang\hlv29d1.dat
 Date Acquired: 8/6/2007 8:55:49 PM Date Printed: 01/17/2008 03:58:04 PM
 Sample ID: hlv29d

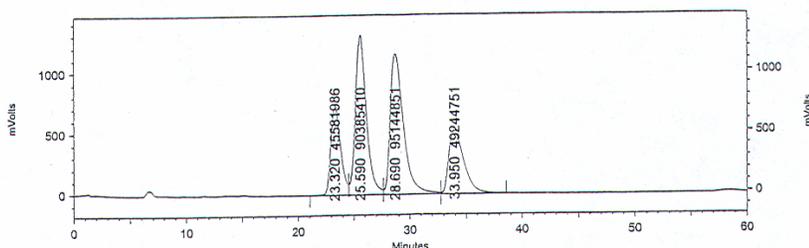


SPD-10Avp
 Ch1-220nm Results

PK #	RT	Area	Area %
1	32.050	144910970	76.971
2	40.720	400234	0.213
3	43.830	42355795	22.498
4	48.270	600442	0.319
Totals		188267441	100.000

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 Department of Chemistry

Method Name: C:\EZStart\Projects\WeiWang\zls1275.met
 Data File: C:\EZStart\Projects\WeiWang\hlu93d1.dat
 Date Acquired: 8/2/2007 12:53:30 PM Date Printed: 01/17/2008 04:07:12 PM
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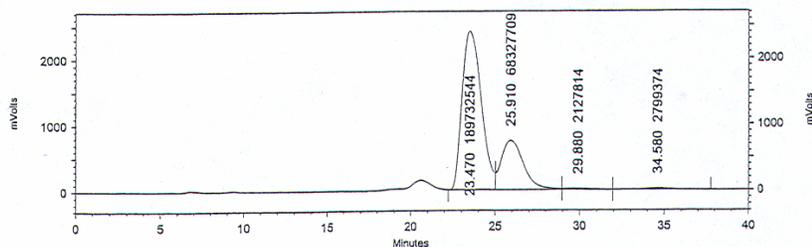
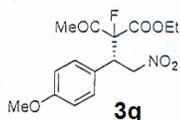


SPD-10Avp
 Ch1-220nm Results

Pk #	RT	Area	Area %
1	23.320	45581986	16.259
2	25.590	90385410	32.239
3	28.690	95144851	33.937
4	33.950	49244751	17.565
Totals		280356998	100.000

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 Department of Chemistry

Method Name: C:\EZStart\Projects\WeiWang\zls1256-4.met
 Data File: C:\EZStart\Projects\WeiWang\hlv23d1.dat
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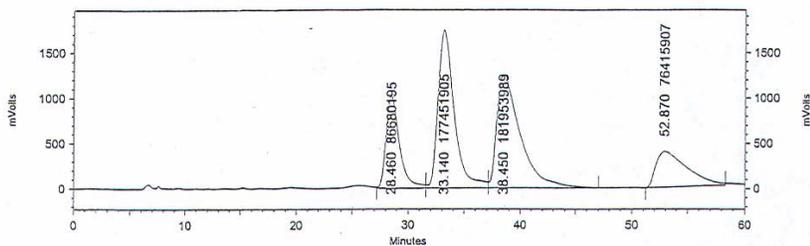


SPD-10Avp
 Ch1-220nm Results

Pk #	RT	Area	Area %
1	23.470	189732544	72.145
2	25.910	68327709	25.981
3	29.880	2127814	0.809
4	34.580	2799374	1.064
Totals		262987441	100.000

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Department of Chemistry

Method Name: C:\EZStart\Projects\WeiWang\zls1275.met
Data File: C:\EZStart\Projects\WeiWang\hlv1d1.dat
Date Acquired: 8/2/2007 1:54:42 PM Date Printed: 01/17/2008 04:10:30 PM
Sample ID: hlvd

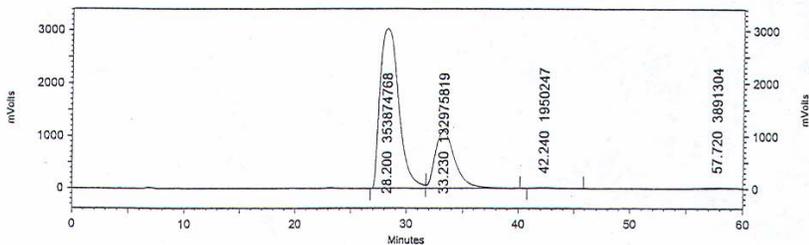
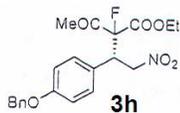


SPD-10Avp
Ch1-220nm Results

Pk #	RT	Area	Area %
1	28.460	86680195	16.589
2	33.140	177451905	33.962
3	38.450	181953989	34.824
4	52.870	76415907	14.625
Totals		522501996	100.000

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Department of Chemistry

Method Name: C:\EZStart\Projects\WeiWang\zls1256-4.met
Data File: C:\EZStart\Projects\WeiWang\hlv24d1.dat
Date Acquired: 8/6/2007 5:16:09 PM Date Printed: 01/17/2008 04:11:00 PM
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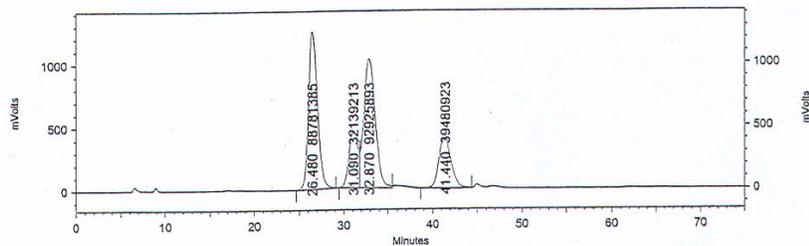
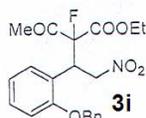


SPD-10Avp
Ch1-220nm Results

Pk #	RT	Area	Area %
1	28.200	353874768	71.825
2	33.230	132975819	26.990
3	42.240	1950247	0.396
4	57.720	3891304	0.790
Totals		492692138	100.000

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Method Name: C:\EZStart\Projects\WeiWang\zls1256-4.met
 Data File: C:\EZStart\Projects\WeiWang\hlv27d3.dat
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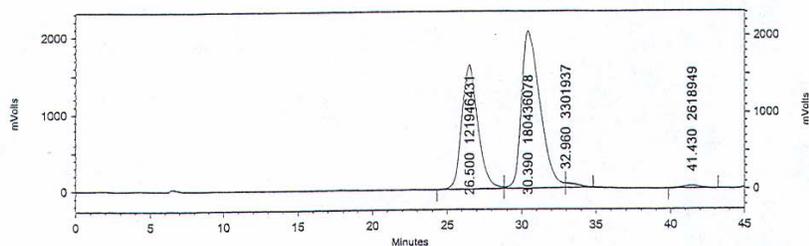
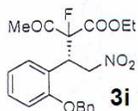


SPD-10Avp
 Ch1-220nm Results

PK #	RT	Area	Area %
1	26.480	88781385	35.046
2	31.090	32139213	12.687
3	32.870	92925893	36.682
4	41.440	39480923	15.585
Totals		253327414	100.000

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Method Name: C:\EZStart\Projects\WeiWang\aa6.met
 Data File: C:\EZStart\Projects\WeiWang\hlv28d1.dat
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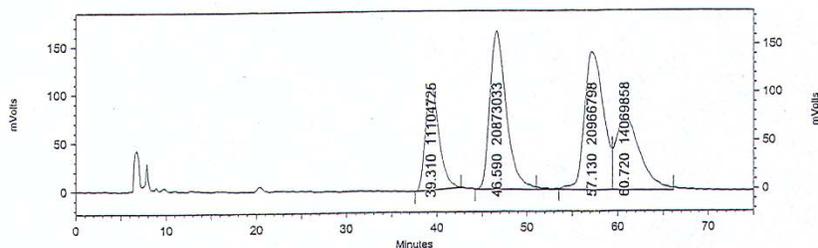
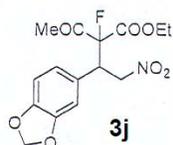


SPD-10Avp
 Ch1-220nm Results

PK #	RT	Area	Area %
1	26.500	121946431	39.554
2	30.390	180436078	58.525
3	32.960	3301937	1.071
4	41.430	2618949	0.849
Totals		308303395	100.000

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 Department of Chemistry

Method Name: C:\EZStart\Projects\WeiWang\aa2.met
 Data File: C:\EZStart\Projects\WeiWang\hlv2d2.dat
 Date Acquired: 8/2/2007 9:36:56 PM Date Printed: 01/17/2008 04:20:02 PM
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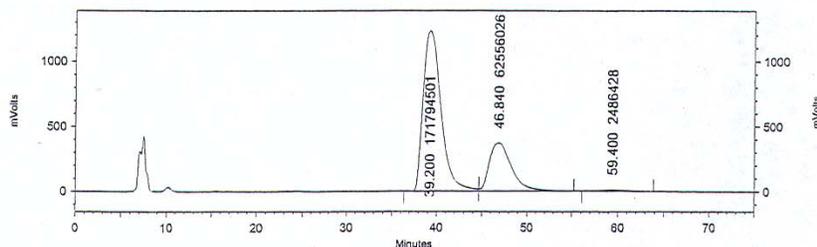
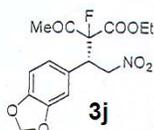


SPD-10Avp
 Ch1-220nm Results

Pk #	RT	Area	Area %
1	39.310	11104725	16.571
2	46.590	20873033	31.147
3	57.130	20966798	31.287
4	60.720	14069858	20.995
Totals		67014414	100.000

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Method Name: C:\EZStart\Projects\WeiWang\aa6.met
 Data File: C:\EZStart\Projects\WeiWang\hlv30d1.dat
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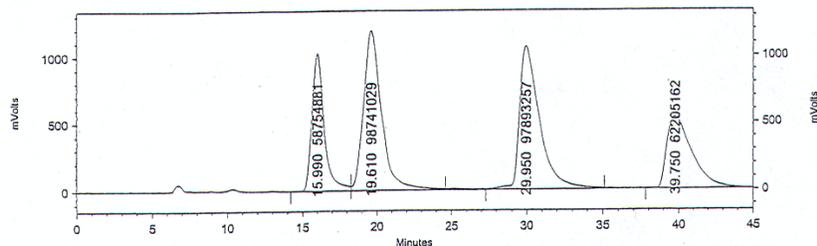
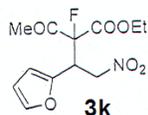


SPD-10Avp
 Ch1-220nm Results

Pk #	RT	Area	Area %
1	39.200	171794501	72.537
2	46.840	62556026	26.413
3	59.400	2486428	1.050
Totals		236836955	100.000

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 Department of Chemistry

Method Name: C:\EZStart\Projects\WeiWang\zls1275.met
 Data File: C:\EZStart\Projects\WeiWang\hlv13d1.dat
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 Sample ID: hlv13d



SPD-10Avp

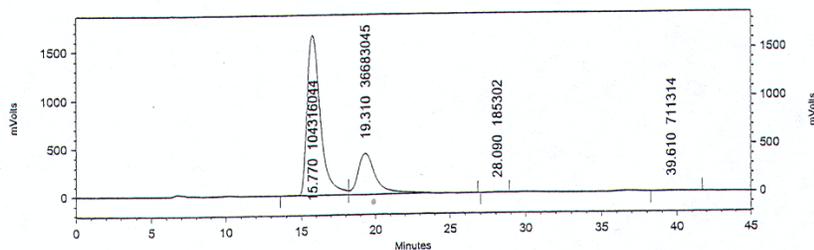
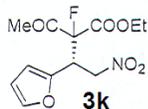
Ch1-220nm Results

PK #	RT	Area	Area %
1	15.990	58754881	18.500
2	19.610	98741029	31.090
3	29.950	97893257	30.823
4	39.750	62205162	19.586

Totals		317594329	100.000
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 Department of Chemistry

Method Name: C:\EZStart\Projects\WeiWang\zls1256-4.met
 Data File: C:\EZStart\Projects\WeiWang\hlv25d1.dat
 Date Acquired: 8/7/2007 11:48:48 AM Date Printed: 01/17/2008 04:13:02 PM
 Sample ID: hlv25d



SPD-10Avp

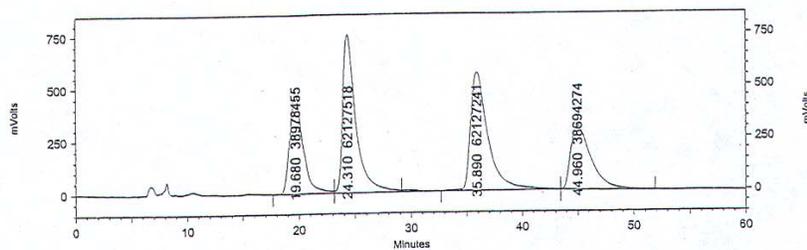
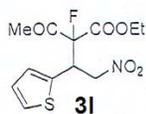
Ch1-220nm Results

PK #	RT	Area	Area %
1	15.770	104316044	73.516
2	19.310	36683045	25.852
3	28.090	185302	0.131
4	39.610	711314	0.501

Totals		141895705	100.000
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Method Name: C:\EZStart\Projects\WeiWang\zls1275.met
 Data File: C:\EZStart\Projects\WeiWang\hlv14d1.dat
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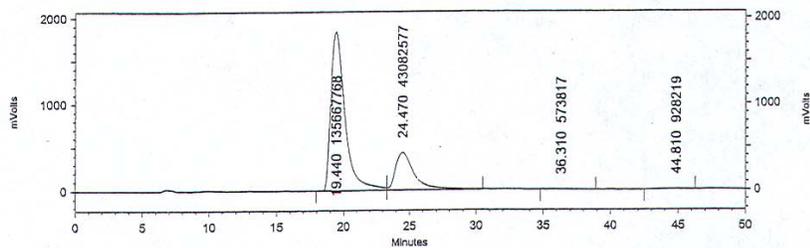
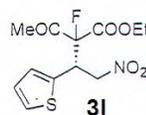


SPD-10Avp
 Ch1-220nm Results

Pk #	RT	Area	Area %
1	19.680	38978455	19.303
2	24.310	62127518	30.767
3	35.890	62127241	30.767
4	44.960	38694274	19.162
Totals		201927488	100.000

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Method Name: C:\EZStart\Projects\WeiWang\zls1256-4.met
 Data File: C:\EZStart\Projects\WeiWang\hlv26d1.dat
 Date Acquired: 8/7/2007 12:59:36 PM Date Printed: 01/17/2008 04:15:25 PM
 Sample ID: hlv26d

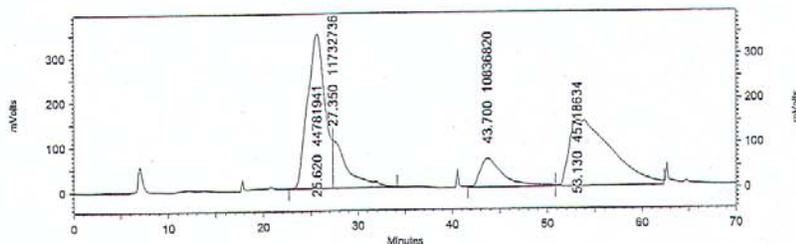
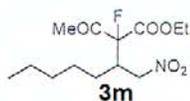


SPD-10Avp
 Ch1-220nm Results

Pk #	RT	Area	Area %
1	19.440	135667768	75.265
2	24.470	43082577	23.901
3	36.310	573817	0.318
4	44.810	928219	0.515
Totals		180252381	100.000

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Method Name: C:\EZStart\Projects\WeiWang\aa8.met
 Data File: C:\EZStart\Projects\WeiWang\hlw25d6.dat
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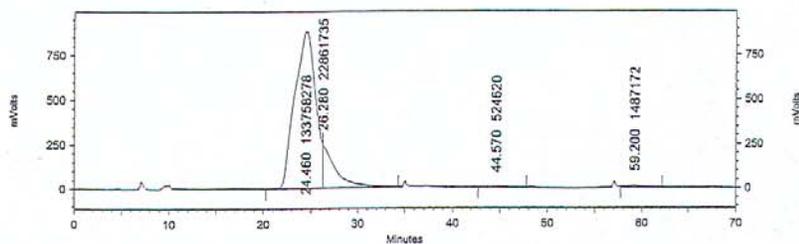
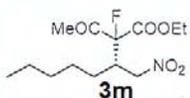


SPD-10Avp
 Ch1-220nm Results

PK #	RT	Area	Area %
1	25.620	44781941	39.605
2	27.350	11732736	10.377
3	43.700	10836820	9.584
4	53.130	45718634	40.434
Totals		113070131	100.000

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Method Name: C:\EZStart\Projects\WeiWang\aa8.met
 Data File: C:\EZStart\Projects\WeiWang\hlw22d2.dat
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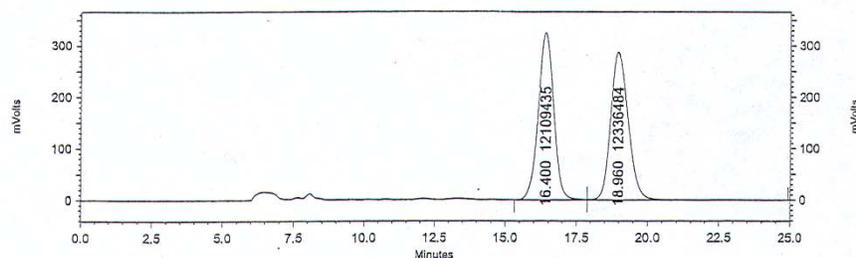
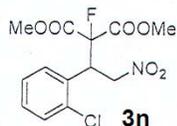


SPD-10Avp
 Ch1-220nm Results

PK #	RT	Area	Area %
1	24.460	133758278	84.320
2	26.280	22861735	14.412
3	44.570	524620	0.331
4	59.200	1487172	0.937
Totals		158631805	100.000

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Method Name: C:\EZStart\Projects\WeiWang\zls1139.met
 Data File: C:\EZStart\Projects\WeiWang\hlt55d2.dat
 Date Acquired: 5/19/2007 3:43:08 PM Date Printed: 01/17/2008 04:34:39 PM
 Sample ID: hlt55d



SPD-10Avp

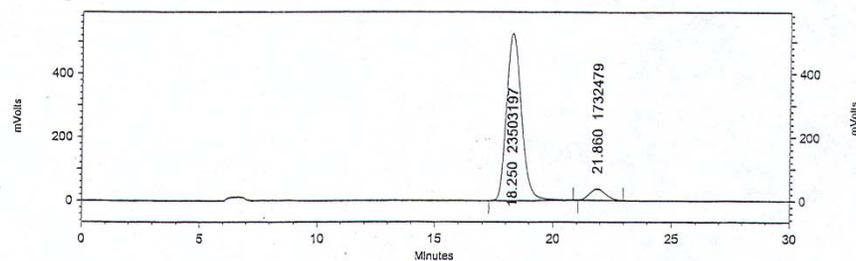
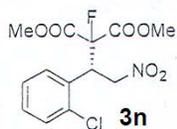
Ch1-235nm Results

PK #	RT	Area	Area %
1	16.400	12109435	49.536
2	18.960	12336484	50.464

Totals		24445919	100.000
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Method Name: C:\EZStart\Projects\WeiWang\zls10100.met
 Data File: C:\EZStart\Projects\WeiWang\hlt69d1.dat
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 Sample ID: hlt69d



SPD-10Avp

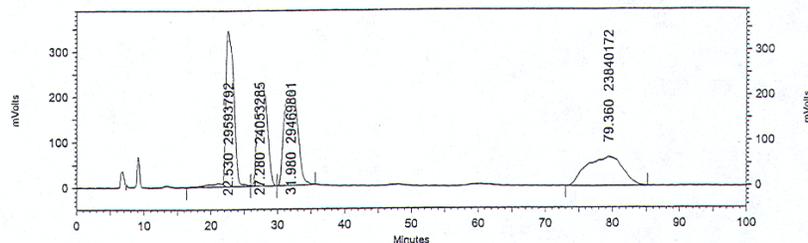
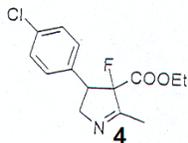
Ch1-235nm Results

PK #	RT	Area	Area %
1	18.250	23503197	93.135
2	21.860	1732479	6.865

Totals		25235676	100.000
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 Department of Chemistry

Method Name: C:\EZStart\Projects\WeiWang\zls1368.met
 Data File: C:\EZStart\Projects\WeiWang\hlw13d8.dat
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 Sample ID: hlw13d



SPD-10Avp

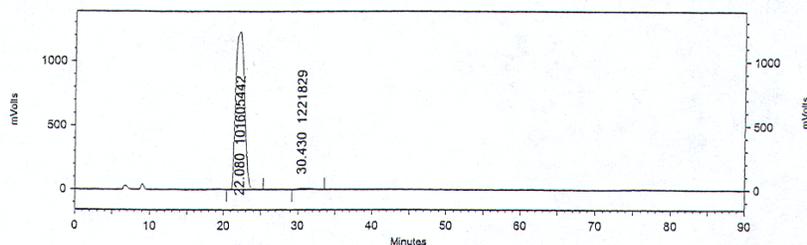
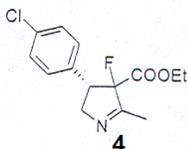
Ch1-220nm Results

Pk #	RT	Area	Area %
1	22.530	29593792	27.669
2	27.280	24053285	22.489
3	31.980	29469801	27.553
4	79.360	23840172	22.289

Totals		106957050	100.000
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Method Name: C:\EZStart\Projects\WeiWang\zls1368.met
 Data File: C:\EZStart\Projects\WeiWang\hlw10d2.dat
 Date Acquired: 9/27/2007 10:41:22 AM Date Printed: 01/17/2008 05:15:12 PM
 Sample ID: hlw10d



SPD-10Avp

Ch1-220nm Results

Pk #	RT	Area	Area %
1	22.080	101605442	98.812
2	30.430	1221829	1.188

Totals		102827271	100.000
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