Supporting Information

One-pot fluorination and asymmetric Michael addition promoted by recyacable fluorous organocatalysts

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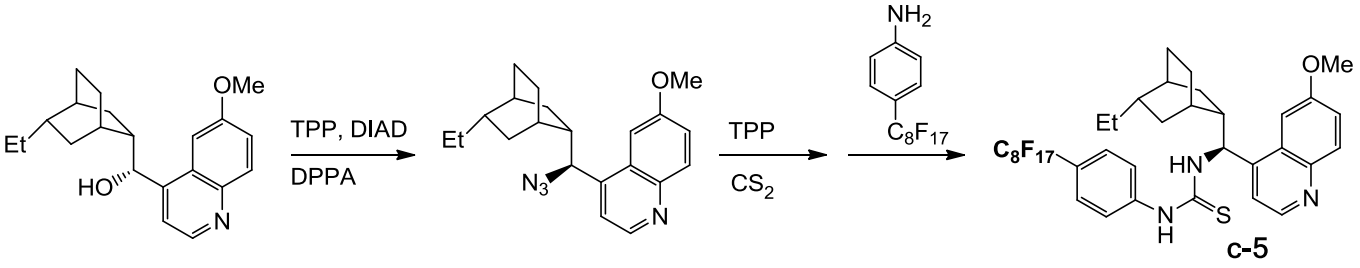
1. General Information

Chemicals and solvents were purchased from commercial suppliers and used as received. ^{1}H and ^{13}C NMR spectra were recorded on a Varian (300 MHz) NMR spectrometer or AMX500 (500 MHz) spectrometer. LC-MS were performed on an Agilent 2100 system with a C_{18} column (5.0 μm, 6.0 x 50 mm). The mobile phases were MeOH and water both containing 0.05% trifluoro acetic acid. A linear gradient was used to increase from 75:25 MeOH/water to 100% MeOH in 5 min at a flow rate of 0.7 mL/min. The UV detections were at 210 nm, 254 nm and 365 nm. Low resolution mass spectra were recorded in APCI (atmospheric pressure chemical ionization). All high resolution mass spectra were obtained on a Finnigan/MAT 95XL-T spectrometer. Flash chromatography separations were performed on YAMAZEN AI-580 system with Agela (12 g or 20 g, 230-400 μm mesh) silica gel. The enantiomeric excesses (ee) of products were determined by chiral phase HPLC analysis on an SHIMADZU LC-20AD system. For final products **3a-3s,** the diastereomeric ratio (dr) was determined by ^{1}H NMR of the crude reaction mixture before flash column chromatography. The enantiomeric excess (ee) was determined by chiral LC of samples after flash chromatography. The dr of the purified sample could be different from the crude sample.

2. Representative Synthetic Procedures



Synthesis of catalyst c-5: To a mixture of hydroquindinine (1.64 g, 5 mmol), triphenyl phosphine (TPP, 1.83 g, 7 mmol) in 30 mL of THF, diisopropyl azodicarboxylate (DIAD, 1.18 mL, 6 mmol) was added dropwise under nitrogen at 0 ºC. After stirring for 5 min, the mixture of diphenyl phosphorazidate (DPPA, 1.18 mL, 5.5 mmol) in 15 mL of THF was added slowly via injector to the reaction mixture. The reaction was stirred at 25 ºC for 12 h and then heated at 50 ºC for 3 h. A solution of TPP (1.8 g, 7

mmol) in 10 mL of THF was added. After stirring at 50 ºC for 3 h, CS_2 (1 mL) was added dropwise. After the reaction mixture was then stirred for 16 h, 4-perfluorooctylanline (1.5 g, 3 mmol) was added and heated under microwave at 120 ºC for 1 h. The concentrated reaction mixture was purified by F-SPE on a Fluoro*Flash*® silica gel cartridge (25 g) and eluted with 80:20 MeOH/H_2O and then 100% MeOH to give **c-5** (703 mg, 27%).

Representative procedure for one-pot flurination and Michael addition. Catalyst **c-5** (15 mg, 0.02 mmol) was added to a mixture of Selectfluor™ (35 mg, 0.1 mmol), nitroolefin (149 mg, 0.1 mmol) and ethyl benzoylacetate (192 mg, 0.1 mmol) in 0.5 mL of CH_3CN and 0.5 mL of toluene. After stirring at -20 ℃ for 48 h, the reaction mixture was loaded onto a 5 g Fluoro*Flash*® silica gel cartridge and eluted with 80:20 MeOH-H_2O and then MeOH. The catalyst recovered from the MeOH fraction. Product in the concentrated MeOH-H_2O fraction was extracted with EtOAc and then washed with 2 M HCl and water. The concentrated crude product was purified by flash column (6:1 hexane/EtOAc) to give **3a** (33 mg, 92%)

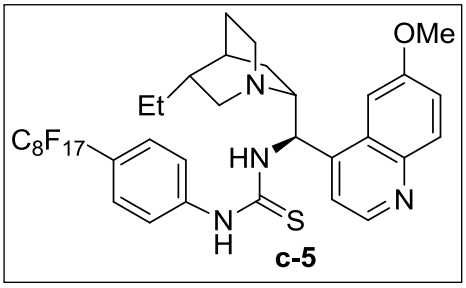
Representative procedures for one-pot and two-step fluorination and Michael addition with maleimides. Catalyst **c-5** (15 mg, 0.02 mmol) was added to a mixture of Selectfluor™ (35 mg, 0.1 mmol), and ethyl benzoylacetate (192 mg, 0.1 mmol) in 0.5 mL of CH_3CN and 0.5 mL CH_2Cl_2. After stirring at room temp for 24 h, *N*-ethylmaleimide (13 mg, 0.1 mmol) was added and the mixture was stirred at -20 ºC for 8 h. The reaction mixture was loaded onto a 5 g Fluoro*Flash*® silica gel cartridge for catalyst recovery following the same procedure described above. The crude product was purified by flash column (6:1 hexane/EtOAc) to give **3l** (31 mg, 93%).

Procedure for recycling organocatalyst c-5: The reaction mixture was loaded onto a 5 g Fluoro*Flash*® silica gel cartridge. The cartridge was eluted with 80:20 MeOH:H_2O for reaction product and then with MeOH for the organocatalyst. The MeOH:H_2O fraction was concentration and dried in vacuo at 50 ºC for 8 h to afford recovered organocatalyst **c-5.**

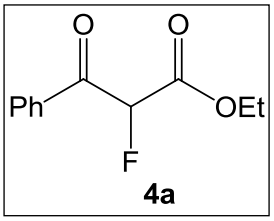
Synthesis of racemic α-fluoro-β-ketoester 4a: The mixture of Selectfluor^{TM} (570 mg, 1.6 mmol) and ethyl benzoylacetate (310 mg, 1.6 mmol) in 5 mL CH_3CN was stirred at 90 ℃ under microwave for 1 h. The mixture was extract with EtOAc. The organic layer was washed with 2 M HCl and water, and dried over Na_2SO_4. After concentration, the residue was purified by flash column (8:1 hexane/EtOAc) to give ethyl 2-fluoro-3-oxo-3-phenylpropanoate **4a** as a colorless oil **4a** (310 mg, 94%).

Synthesis of racemic fluorinated Michael addition product 3: To a mixture of nitroolefin (89 mg, 0.6 mmol) and α-fluoro-β-ketoester **4a** (105 mg, 0.5 mmol) in 1 mL of CH_3CN and 1 mL of toluene Et_3N (50 mg, 0.5 mmol) was added Et_3N (50 mg, 0.5 mmol). After stirring for at room temp for 24 h, the reaction mixture was extract with EtOAc. The organic layer was washed with 2 M HCl and water, and dried over Na_2SO_4. After concentration, the residue was purified by flash column (6:1 hexane/EtOAc) to give the addition product (172 mg, 96%).

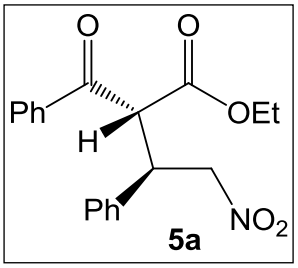
3. Analytical Data and Chiral HPLC Chromatogram



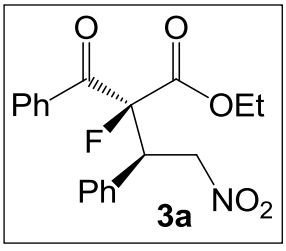
c-5: ^{1}H NMR ($CDCl_3$, 300 MHz) δ 0.91(t, *J* = 7.2 Hz, 3H), 0.98 (m, 1H), 1.14-1.16 (m, 1H), 1.41-1.55 (m, 6H), 2.97-3.06 (m, 4H), 4.02 (s, 3H), 3.32 (m, 1H), 6.38-6.42 (d, *J* = 10.5 Hz, 1H), 7.43-7.60 (m, 4H), 7.73-7.75 (d, *J* = 8.4 Hz, 2H), 7.93-7.96 (d, *J* = 9 Hz, 1H), 8.06 (d, 1H), 8.67-8.69 (d, *J* = 4.8 Hz, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 12.2, 26.0, 27.0, 27.2, 27.9, 38.4, 48.1, 50.1, 50.8, 56.3, 61.6, 103.8, 121.1, 123.2, 124.0, 128.4, 130.2, 131.2, 144.8, 145.1, 148.3, 159.6, 164.3, 170.6, 182.3, 198.3; MS (ACPI) m/z : 879.2 (M^++1); HRMS (EI) m/z: calcd. for $C_{35}H_{32}F_{17}N_4S$ $[M + H]^+$: 879.2025; Found: 879.2010.



4a (racemic), ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.15 (t, *J* = 7.2 Hz, 3H), 4.23 (q, *J* = 7.2 Hz, 2H), 5.70-5.87 (s, *J* = 48.9 Hz, 1H), 7.18-7.45 (m, 2H), 7.53 (m, 1H), 7.94-7.98 (m, 2H); MS (ACPI) m/z : 211.1 (M^++1).

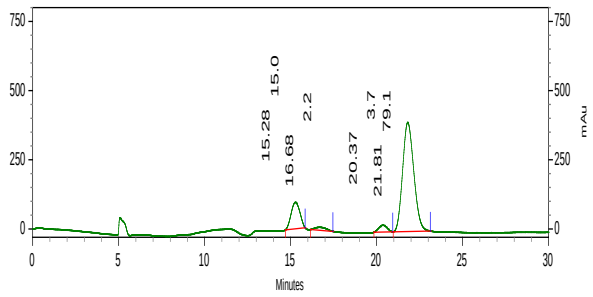


5a, ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.28 (t, *J* = 7.2 Hz, 3H), 4.24 (q, *J* = 7.2 Hz, 2H), 4.50 (m, 1H), 4.82 (m, 1H), 4.92 (m, 2H), 7.22-7.34 (m, 8H), 7.46-7.69 (m, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 13.9, 43.0, 56.3, 62.2, 127.9, 128.1, 128.3, 128.6, 128.7, 128.9, 128.9, 133.8, 134.2, 192.2; MS (ACPI) m/z : 342.1 (M^++1).



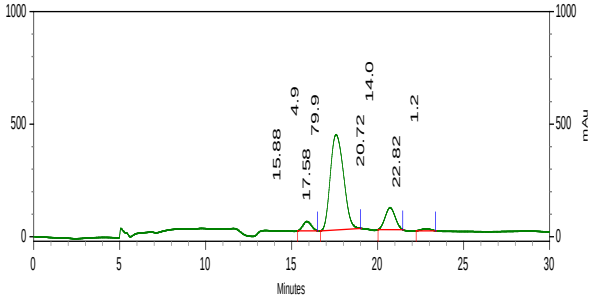
3a: 92% yield (6:1 dr), 95% ee. The enantiomeric excess was determined by HPLC on Venusil Chiral OD-H with hexane/i-PrOH (93:7) as the eluent. Flow rate: 0.4 mL/min, λ = 254nm: Ent-1 = 16.676 min, Ent-2 = 21.808 min. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.29 (t, *J* = 7.2 Hz, 3H), 4.34 (q, *J* = 7.2 Hz, 2H), 4.78 (m, 1H), 4.87 (m, 2H), 7.23-7.37 (m, 8H), 7.49-7.70 (m, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 13.6, 13.8, 46.8, 47.1, 47.7, 48.0, 63.1, 63.7, 75.7, 75.8, 99.4, 102.1, 128.4, 128.5, 128.7, 128.8, 129.0, 129.1, 129.2, 129.4,

129.7, 130.0, 130.1, 133.0, 133.2, 133.8, 134.1, 134.1, 134.7, 165.4, 165.7, 191.6, 192.0; MS (ACPI) m/z : 360.0 (M^++1).



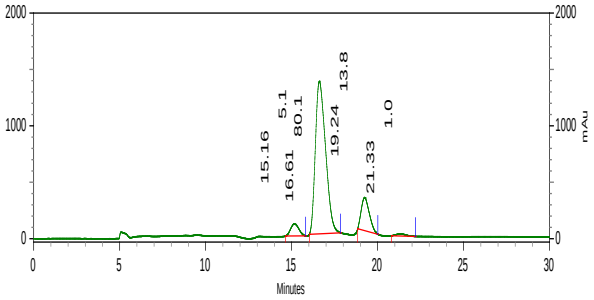
	Retention Time	Area %
	15.280	15.00
(Ent-1)	16.676	2.24
	20.368	3.67
(Ent-2)	21.808	79.10
	Totals	100.00

Chiral sample catalyzed by c-5



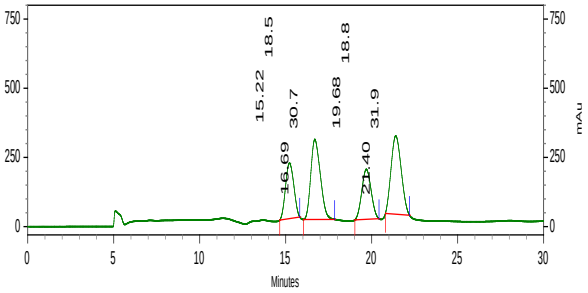
Retention Time	Area %
15.876	4.95
17.580	79.92
20.724	13.96
22.824	1.18
Totals	100.00

Chiral sample catalyzed by c-6



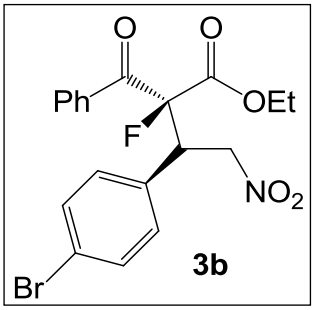
Retention Time	Area %
15.164	5.09
16.612	80.10
19.244	13.79
21.328	1.02
Totals	100.00

Chiral sample catalyzed by c-7

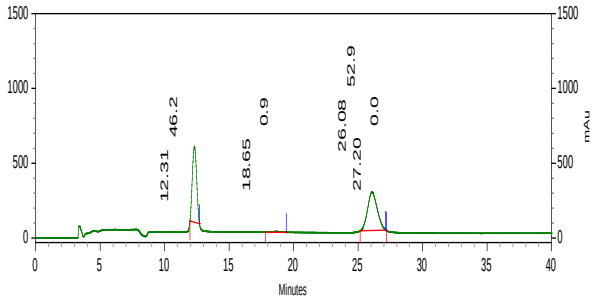


Retention Time	Area %
15.216	18.53
16.692	30.73
19.684	18.82
21.396	31.92
Totals	100.00

Racemic sample

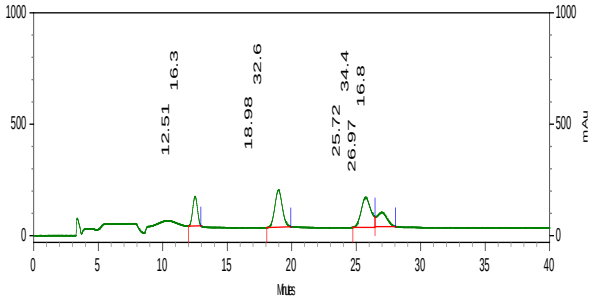


3b: 96% yield (5:1 dr), 96% ee. The enantiomeric excess was determined by HPLC on Venusil Chiral OD-H with hexane/i-PrOH (95:5) as the eluent. Flow rate: 0.6 mL/min, λ = 254nm: Ent.(1) = 18.652 min, Ent.(2) = 26.076 min. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.30 (t, *J* = 7.2 Hz, 3H), 4.36 (q, *J* = 7.2 Hz, 2H), 4.78 (m, 1H), 4.91 (m, 2H), 7.23-7.39 (m, 6H), 7.49-7.71 (m, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 14.0, 47.3, 47.6, 64.1, 75.7, 128.7, 129.1, 129.5, 129.6, 130.0, 131.3, 131.6, 131.6, 132.1, 132.3, 132.5, 134.3, 135.1, 200.5; MS (ACPI) m/z : 440.0 (M^++1).



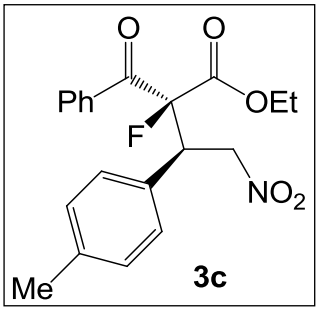
Retention Time	Area %
12.308	46.25
(Ent-1) 18.652	0.87
(Ent-2) 26.076	52.88
27.204	0.00
Totals	100.00

Chiral sample catalyzed by C5

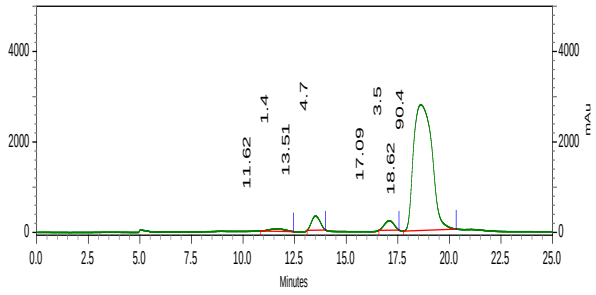


Retention Time	Area %
12.508	16.29
(Ent-1) 18.976	32.58
(Ent-2) 25.724	34.36
26.968	16.76
Totals	100.00

Racemic sample

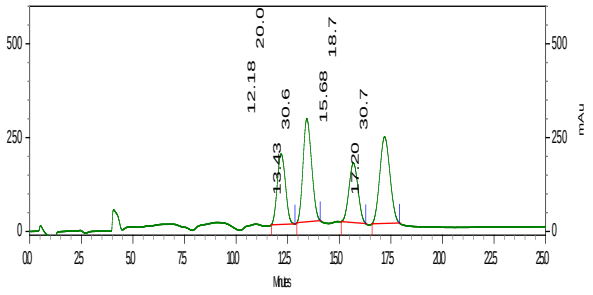


3c: 90% yield (3:1 dr), 91% ee. The enantiomeric excess was determined by HPLC on Venusil Chiral OD-H with hexane/i-PrOH (93:7) as the eluent. Flow rate: 0.6 mL/min, λ = 254nm: Ent-1 = 13.512 min, Ent.-2 = 18.616 min. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.28 (t, *J* = 7.2 Hz, 3H), 2.25 (s, 3H), 4.34 (q, *J* = 7.2 Hz, 2H), 4.78 (m, 1H), 4.88 (m, 2H), 7.03-7.73 (m, 8H), 8.08 (m, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 13.6, 13.8, 21.0, 21.1, 46.5, 46.7, 47.6, 63.1, 63.7, 75.8, 128.4, 128.8, 129.2, 129.3, 129.4, 129.5, 129.5, 130.0, 130.1, 133.7, 134.7, 138.3, 138.8, 165.4, 165.8, 170.2; MS (ACPI) m/z: 374.2 (M^{+}+1).



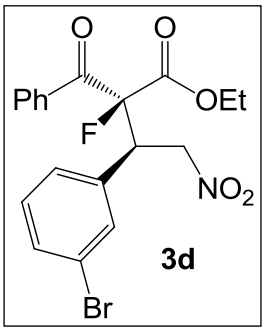
Retention Time	Area %
11.620	1.43
(Ent-1) 13.512	4.69
17.088	3.49
(Ent-2) 18.616	90.39
Totals	100.00

Chiral sample catalyzed by c-5

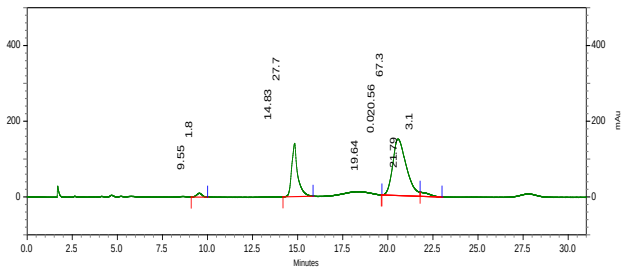


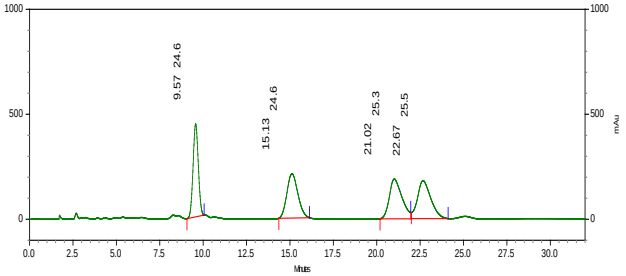
Retention Time	Area %
12.180	19.99
(Ent-1) 13.428	30.58
15.684	18.73
(Ent-2) 17.196	30.70
Totals	100.00

Racemic sample



3d: 91% yield (3:1 dr), 83% ee. The enantiomeric excess was determined by HPLC on Venusil Chiral OD-H with hexane/i-PrOH (98:2) as the eluent. Flow rate: 1.2 mL/min, λ = 254nm: Ent-1 = 20.564 min, Ent-2 = 21.788 min. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.04 (t, *J* = 7.2 Hz, 3H), 4.02 (q, *J* = 7.2 Hz, 2H), 4.79 (m, 1H), 4.91 (m, 2H), 7.23-7.68 (m, 7H), 8.07-8.11 (m, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 13.7, 46.4, 46.7, 63.4, 75.3, 75.4, 128.0, 128.9, 129.4, 130.1, 130.2, 130.4, 132.2, 132.7, 134.1, 134.9, 156.4; MS (ACPI) m/z: 440.0 (M^{+}+1).



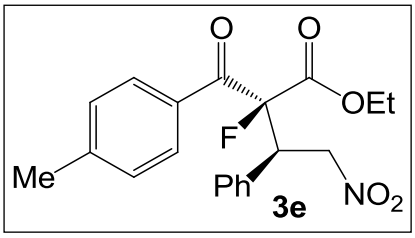


Retention Time	Area %
9.548	1.81
15.832	27.74
(Ent-1) 20.564	67.32
(Ent-2) 21.788	3.13
Totals	100.00

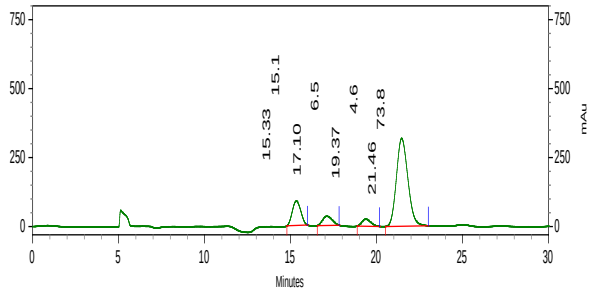
Chiral Sample catalyzed by C-5

Retention Time	Area %
9.568	24.62
15.128	24.60
(Ent-1) 21.020	25.26
(Ent-2) 22.668	25.51
Totals	100.00

Racemic sample

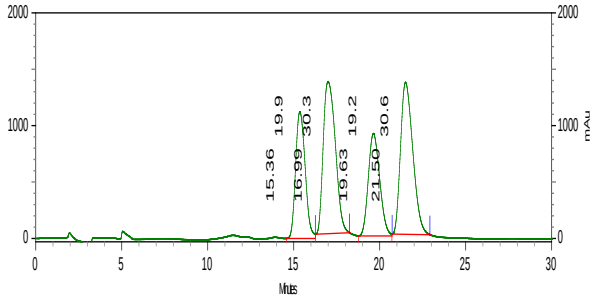


3e: 94% yield (4:1 dr), 85% ee. The enantiomeric excess was determined by HPLC on Venusil Chiral OD-H with hexane/i-PrOH (93:7) as the eluent. Flow rate: 0.6 mL/min, λ = 254nm: Ent-1 = 17.104 min, Ent-2 = 21.456 min. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.29 (t, *J* = 7.2 Hz, 3H), 2.38 (s, 3H), 4.33 (q, *J* = 7.2 Hz, 2H), 4.75 (m, 1H), 4.89 (m, 2H), 7.14-7.40 (m, 7H), 7.67 (d, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 13.6, 13.8, 21.7, 21.8, 47.0, 48.0, 63.6, 75.8, 117.2, 128.5, 128.7, 128.8, 128.9, 129.2, 129.4, 129.5, 129.5, 129.7, 129.7, 130.3, 133.4, 145.0 171.4, 172.4; MS (ACPI) m/z: 374.1 (M^{+}+1).



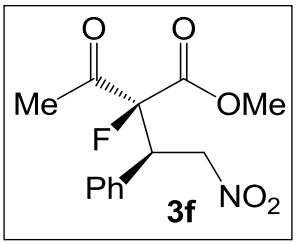
Retention Time	Area %
15.328	15.14
(Ent-1) 17.104	6.51
19.368	4.59
(Ent-2) 21.456	73.76
Totals	100.00

Chiral sample catalyzed by C-5

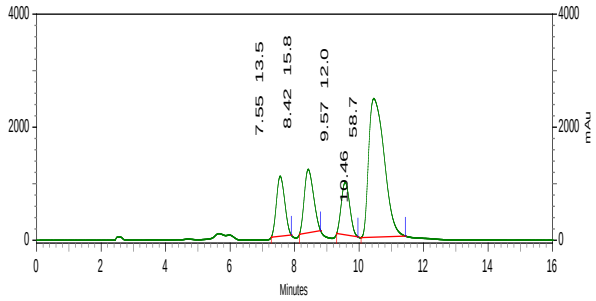


Retention Time	Area %
15.356	19.90
(Ent-1) 16.992	30.27
19.632	19.20
(Ent-2) 21.504	30.62
Totals	100.00

Racemic sample

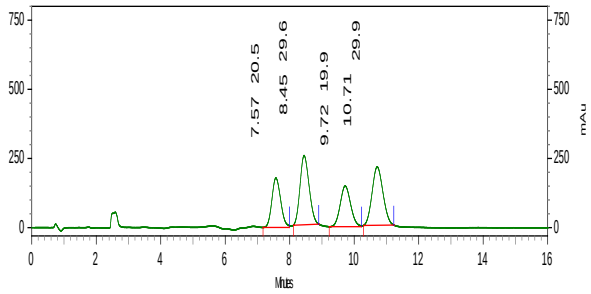


3f: 87% yield (2:1 dr), 57% ee. The enantiomeric excess was determined by HPLC on Venusil Chiral OD-H with hexane/i-PrOH (92:8) as the eluent. Flow rate: 0.8 mL/min, λ = 254nm: Ent-1 = 8.420 min, Ent-2 = 10.460 min. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.87 (s, 3H), 3.89 (s, 3H), 4.60 (dd, 1H), 4.84 (m, 2H), 7.29 (m, 5H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 26.4, 47.1, 47.3, 54.0, 75.1, 117.4, 129.0, 129.1, 129.5, 129.5, 199.5, 200.4; MS (ACPI) m/z: 284.1 (M^{+}+1).



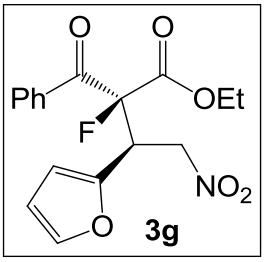
Retention Time	Area %
7.552	13.47
(Ent-1) 8.420	15.80
9.568	12.04
(Ent-2) 10.460	58.69
Totals	100.00

Chiral sample catalyzed by c-5

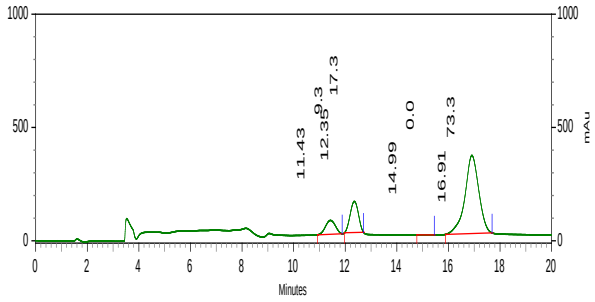


Retention Time	Area %
7.572	20.54
(Ent-1) 8.448	29.64
9.720	19.95
(Ent-2) 10.712	29.87
Totals	100.00

Racemic sample

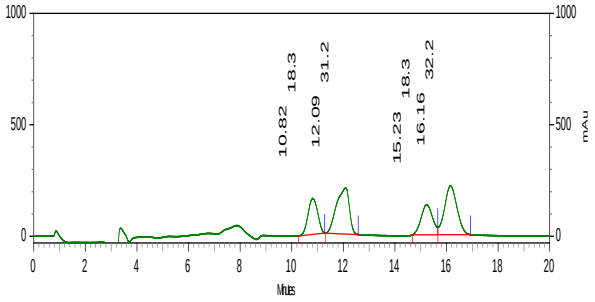


3g: 96% yield (3:1 dr), 62% ee. The enantiomeric excess was determined by HPLC on Venusil Chiral OD-H with hexane/i-PrOH (95:5) as the eluent. Flow rate: 0.6 mL/min, λ = 254nm:Ent-1 = 12.352 min, Ent-2 = 16.908 min. ^{1}H NMR ($CDCl_3$, 75 MHz) δ 1.29 (t, *J* = 7.2 Hz, 3H), 4.34 (q, *J* = 7.2 Hz, 2H), 4.86 (m, 1H), 5.01 (m, 2H), 6.25-6.39 (dd, 2H), 7.26-7.64 (m, 5H), 7.84 (m, 2H), 8.06 (m, 1H); ^{13}C NMR ($CDCl_3$, 300 MHz) δ 13.8, 13.8, 41.3, 41.5, 42.0, 42.2, 63.4, 63.7, 73.4, 73.5, 73.6, 97.8, 100.6, 110.5, 110.7, 110.7, 110.8, 117.2, 128.5, 128.8, 129.4, 129.5, 130.0, 130.1, 133.6, 134.1, 134.8, 143.1, 143.1, 143.6, 146.7, 146.9, 163.9, 164.3, 165.0, 165.4, 172.4; MS (ACPI) m/z : 350.1 (M^{+}+1).



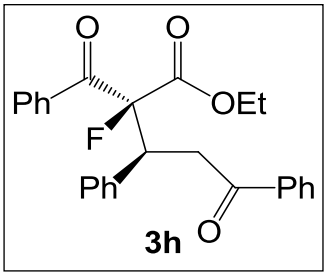
Retention Time		Area %
	11.428	9.34
(Ent-1)	12.352	17.32
	14.988	0.02
(Ent-2)	16.908	73.32
	Totals	100.00

Chiral sample catalyzed by c-5

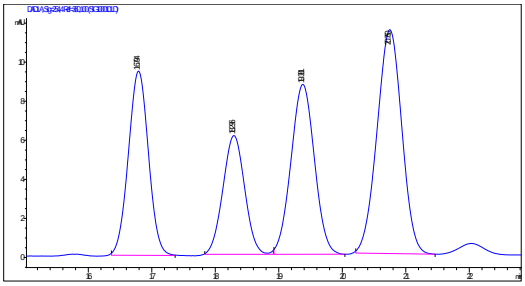


Retention Time		Area %
	10.816	18.30
(Ent-1)	12.092	31.24
	15.232	18.28
(Ent-2)	16.156	32.18
	Totals	100.00

Racemic sample

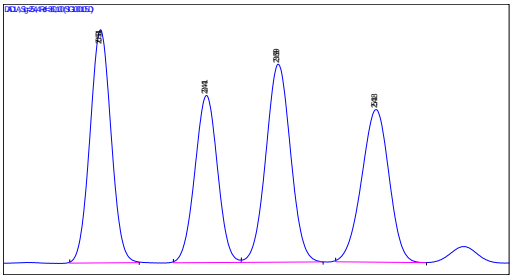


3h: 59% yield (3:1 dr), 36% ee. The enantiomeric excess was determined by HPLC on ULTRON ES-OVM PREP with KH_2PO_4/MeOH (73:27) as the eluent. Flow rate: 1.5 mL/min, λ = 254nm: Ent-1 = 18.296 min, Ent-2 = 20.750 min. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.27 (t, *J* = 7.2 Hz, 3H), 3.43 (m, 1H), 3.76 (m, 1H), 4.31 (q, *J* = 7.2 Hz, 2H), 4.80 (m, 1H), 7.11-7.69 (m, 13H), 7.90-8.12 (m, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 13.7, 13.9, 39.6, 45.0, 45.2, 62.7, 63.2, 127.3, 127.8, 128.2, 128.3, 128.5, 128.6, 128.7, 129.1, 129.2, 129.6, 129.9, 130.0, 130.1, 133.1, 133.2, 133.3, 134.2, 172.4, 196.5; MS (ACPI) m/z : 419.1 (M^++1). HRMS (EI) m/z: calcd. for $C_{26}H_{24}FO_4$ $[M + H]^+$: 419.1695; Found: 419.1667.



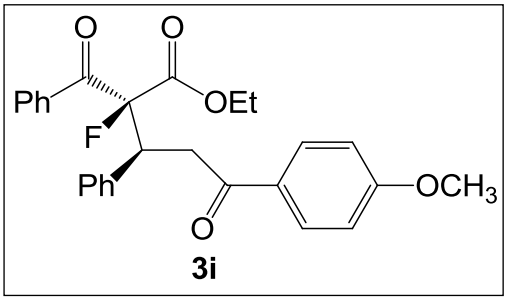
Retention Time	Area %
16.794	23.4
(Ent-1) 18.296	16.4
19.381	25.2
(Ent-2) 20.750	35.1
Totals	100.00

Chiral sample catalyzed by c-5



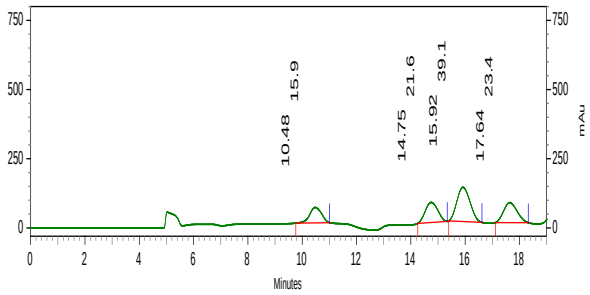
Retention Time	Area %
20.581	27.7
(Ent-1) 22.441	21.3
23.699	27.4
(Ent-2) 25.418	23.5
Totals	100.00

Racemic sample



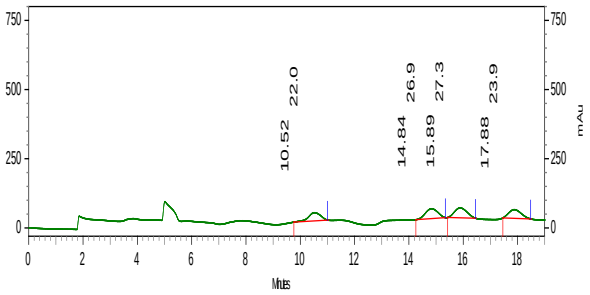
3i

3i: 59% yield (3:1 dr), 36% ee. The enantiomeric excess was determined by HPLC on ULTRON ES-OVM PREP with KH_2PO_4/MeOH (73:27) as the eluent. Flow rate: 1.5 mL/min, λ = 254nm: t_{minor} = 18.296 min, t_{major} = 20.750 min. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.26 (t, *J* = 7.2 Hz, 3H), 3.32 (m, 1H), 3.63 (m, 1H), 3.83 (s, 3H), 4.29 (q, *J* = 7.2 Hz, 2H), 4.79 (m, 1H), 6.88 (m, 2H), 7.10-7.50 (m, 8H), 7.61-7.90 (m, 4H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 13.9, 39.1, 45.1, 55.5, 63.2, 113.6, 113.7, 127.3, 128.2, 128.6, 129.1, 129.2, 129.9, 130.4, 133.2, 195.0, 200.3; MS (ACPI) m/z : 449.1 (M^++1).



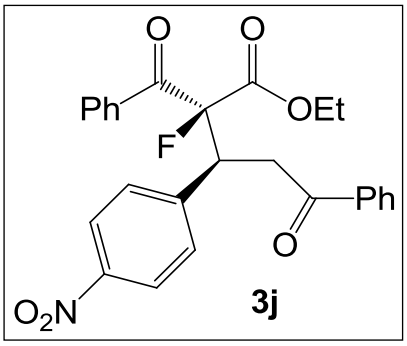
Retention Time		Area %
	10.480	15.91
(Ent-1)	14.752	21.62
(Ent-2)	15.920	39.11
	17.644	23.36
	Totals	100.00

Chiral sample catalyzed by c-5

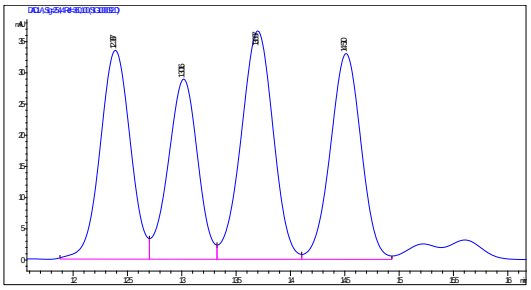


Retention Time		Area %
	10.524	21.96
(Ent-1)	14.836	26.88
(Ent-2)	15.888	27.27
	17.884	23.89
	Totals	100.00

Racemic sample

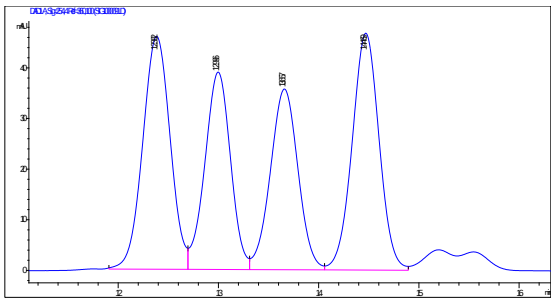


3j: 71% yield (2:1 dr), 20% ee. The enantiomeric excess was determined by HPLC on ULTRON ES-OVM PREP with KH_2PO_4/MeOH (73:27) as the eluent. Flow rate: 1.5 mL/min, λ = 254nm: Ent-1 = 13.106 min, Ent-2 = 13.697 min. ^{1}H NMR ($CDCl_3$, 75 MHz) δ 1.26 (t, *J* = 7.2 Hz, 3H), 3.22 (m, 1H), 3.68 (m, 1H), 4.33 (q, *J* = 7.2 Hz, 2H), 4.89 (m, 1H), 7.35-7.68 (m, 9H), 7.76-7.90 (m, 2H), 8.04-8.18 (m, 3H); ^{13}C NMR ($CDCl_3$, 300 MHz) δ 13.8, 14.1, 39.3, 39.7, 63.0, 63.6, 117.3, 123.3, 123.4, 128.0, 128.5, 128.7, 128.8, 128.8, 129.3, 130.0, 130.1, 130.7, 130.9, 133.5, 134.0, 134.6, 163.8, 164.1, 195.8; MS (ACPI) m/z : 464.1 (M^++1).



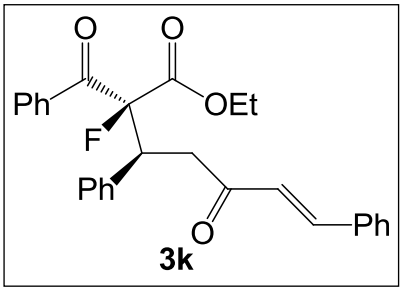
Retention Time	Area %
12.387	24.2
(Ent-1) 13.016	20.1
(Ent-2) 13.697	30.1
14.510	25.6
Totals	100.00

Chiral sample catalyzed by c-5

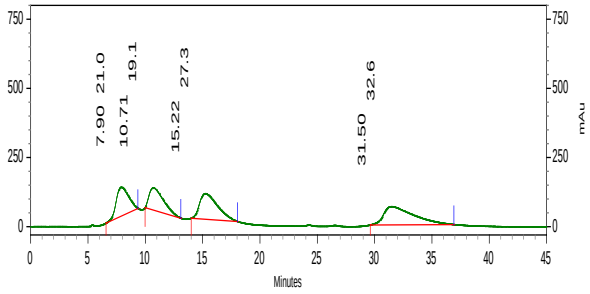


Retention Time	Area %
12.382	27.5
(Ent-1) 12.996	22.1
(Ent-2) 13.657	21.9
14.469	27.5
Totals	100.00

Racemic sample

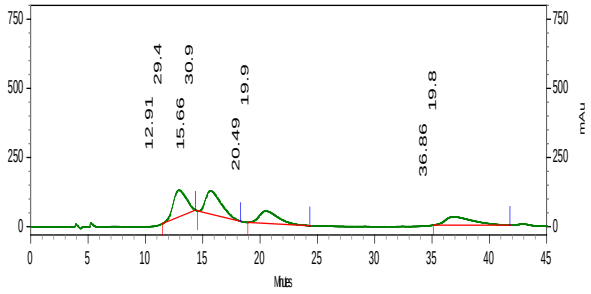


3k: 42% yield (4:1 dr), 8% ee. The enantiomeric excess was determined by HPLC on Venusil Chiral OD-H with hexane/i-PrOH (92:8) as the eluent. Flow rate: 0.8 mL/min, λ = 254nm: Ent-1 = 15.224 min, Ent-2 = 31.496 min. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.29 (t, *J* = 7.2 Hz, 3H), 3.06 (m, 1H), 3.35 (m, 1H), 4.32 (q, *J* = 7.2 Hz, 2H), 4.70 (m, 1H), 6.65 (d, *J* = 16.2 Hz, 1H), 7.10-7.53 (m, 14H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 13.7, 13.9, 41.6, 45.0, 45.3, 63.2, 125.8, 127.4, 127.8, 128.2, 128.3, 128.3, 128.6, 128.9, 129.1, 129.2, 129.5, 129.8, 129.9, 130.0, 130.0, 130.5, 130.6, 133.3, 134.2, 137.5, 142.9, 143.1, 196.4; MS (ACPI) m/z: 445.2 (M^{+}+1).



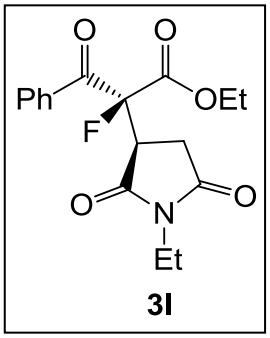
Retention Time		Area %
	7.904	21.01
	10.708	19.13
(Ent-1)	15.224	27.27
(Ent-2)	31.496	32.59
	Totals	100.00

Chiral sampl catalyzed by c-5



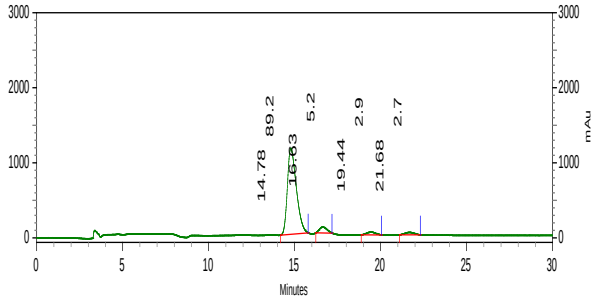
Retention Time		Area %
	12.912	29.43
(Ent-1)	15.664	30.86
	20.492	19.91
(Ent-2)	36.856	19.80
	Totals	100.00

Racemic sample



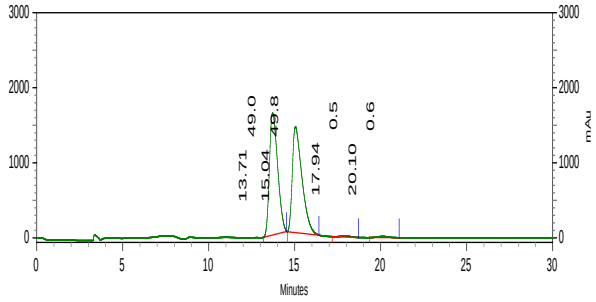
3l

3l: 93% yield (> 20:1 dr), 90% ee. The enantiomeric excess was determined by HPLC on Venusil Chiral OD-H with hexane/i-PrOH (95:5) as the eluent. Flow rate: 0.5 mL/min, λ = 254nm: Ent-1 = 14.780 min, Ent-2 = 16.632 min. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.16 (t, *J* = 7.2 Hz, 3H), 1.28 (t, *J* = 7.2 Hz, 3H), 2.58 (dd, *J* = 18.3 Hz, 1H), 3.05 (dd, *J* = 18.3 Hz, 1H), 3.61 (q, *J* = 7.2 Hz, 2H), 4.14 (m, 1H), 4.42 (m, *J* = 7.2 Hz, 2H), 7.46-7.51 (m, 2H), 7.62 (m, 1H), 8.12 (m, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 12.9, 13.8, 30.8, 34.1, 44.9, 63.5, 128.5, 128.8, 130.1, 130.2, 134.8, 168.5, 174.8; MS (ACPI) m/z : 336.1 (M^++1). HRMS (EI) m/z: calcd. for $C_{17}H_{19}FO_5$ [M + H]$^+$: 336.1247; Found: 336.1250.



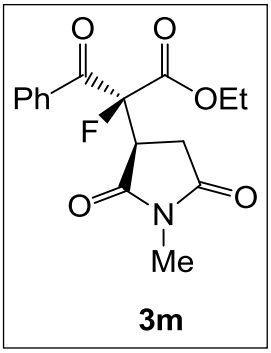
Retention Time		Area %
(Ent-1)	14.780	89.20
(Ent-2)	16.632	5.20
	19.440	2.91
	21.684	2.69
	Totals	100.00

Chiral sample catalyzed by c-5



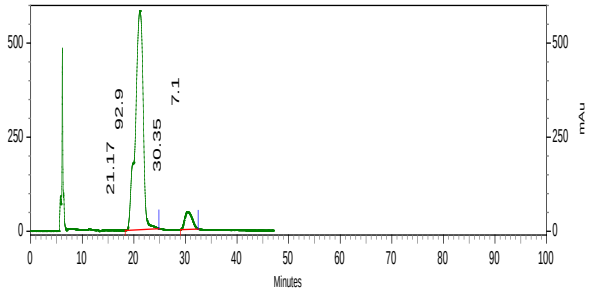
Retention Time		Area %
(Ent-1)	13.712	49.03
(Ent-2)	15.040	49.85
	17.944	0.53
	20.096	0.59
	Totals	100.00

Racemic sample



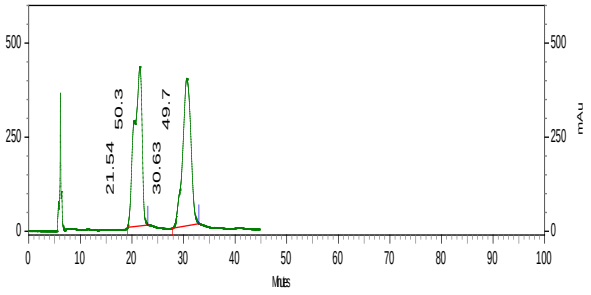
3m

3m: 91% yield (> 20:1 dr), 86% ee. The enantiomeric excess was determined by HPLC on Venusil Chiral OA with hexane/i-PrOH (90:10) as the eluent. Flow rate: 1.0 mL/min, λ = 254 nm: Ent-1 = 21.172 min, Ent-2 = 30.348 min. ^{1}H NMR ($CDCl_3$, 75 MHz) δ 1.24 (t, *J* = 7.2 Hz, 3H), 2.55 (dd, *J* = 18.3 Hz, 1H), 2.98 (s, 3H), 3.05 (dd, *J* = 18.3 Hz, 1H), 4.15 (m, 1H), 4.39 (m, *J* = 7.2 Hz, 2H), 4.67 (q, *J* = 15 Hz, 2H), 7.46 (m, 2H), 7.59 (m, 2H), 7.62 (m, 1H), 8.09 (m, 1H); ^{13}C NMR ($CDCl_3$, 300 MHz) δ 13.7, 25.0, 30.7, 30.7, 44.9, 45.1, 63.4, 97.6, 100.3, 128.8, 130.1, 130.1, 132.4, 132.5, 134.8, 165.0, 165.4, 174.6, 174.9, 189.2, 189.5, 200.4; MS (ACPI) m/z : 322.1 (M^{+}+1).



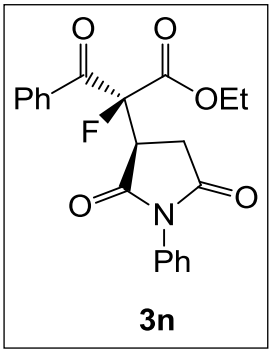
Retention Time	Area %
(Ent-1) 21.172	92.95
(Ent-2) 30.348	7.05
Totals	100.00

Chiral sample catalyzed by c-5



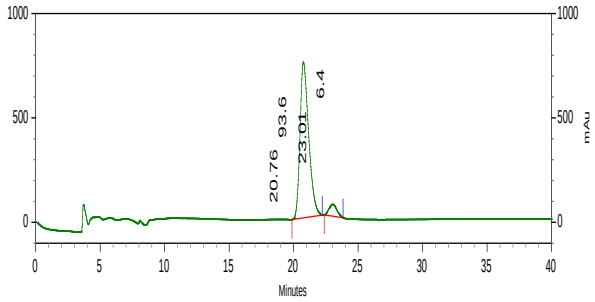
Retention Time	Area %
(Ent-1) 21.540	50.31
(Ent-2) 30.632	49.69
Totals	100.00

Racemic sample



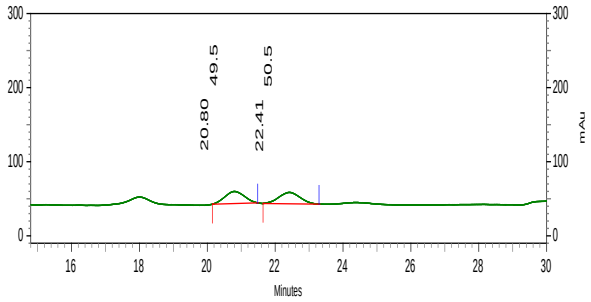
3n

3n: 90% yield (> 20:1 dr), 87% ee. The enantiomeric excess was determined by HPLC on Venusil Chiral OD-H with hexane/i-PrOH (94:6) as the eluent. Flow rate: 0.5 mL/min, λ = 254 nm: Ent-1 = 20.760 min, Ent-2 = 23.008 min. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.24 (t, *J* = 7.2 Hz, 3H), 2.77 (dd, *J* = 18.3 Hz, 1H), 3.25 (dd, *J* = 18.3 Hz, 1H), 4.24 (m, 1H), 4.42 (m, *J* = 7.2 Hz, 2H), 7.27 (m, 2H), 7.28-7.59 (m, 5H), 7.60 (m, 2H), 8.14 (m, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 13.8, 14.1, 20.9, 30.8, 30.9, 45.0, 45.4, 60.3, 63.5, 97.9, 100.6, 126.4, 128.8, 128.8, 129.1, 130.1, 130.1, 131.3, 132.5, 132.5, 134.1, 134.8, 164.9, 165.3, 173.7, 173.9, 189.2, 189.5; MS (ACPI) m/z : 384.1 (M^++1).



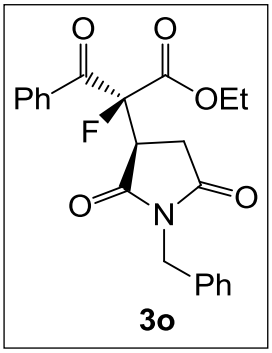
Retention Time		Area %
(Ent-1)	20.760	93.56
(Ent-2)	23.008	6.44
	Totals	100.00

Chiral sample catalyzed by c-5



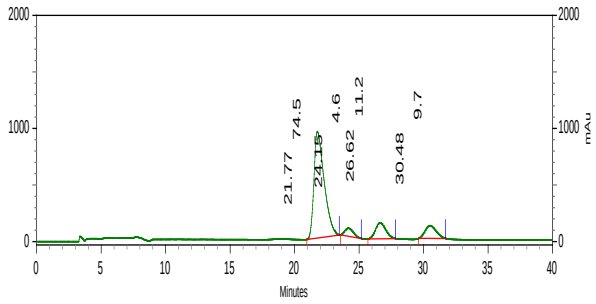
Retention Time		Area %
(Ent-1)	20.804	49.49
(Ent-2)	22.412	50.51
	Totals	100.00

Racemic sample



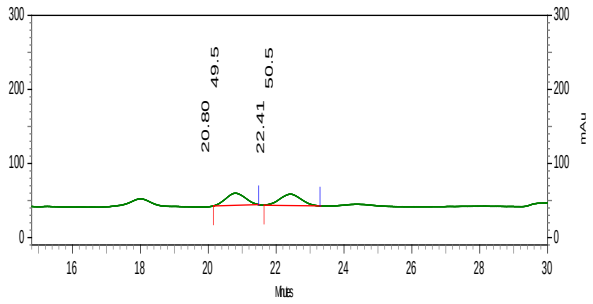
3o

3o: 96% yield (> 20:1 dr), 90% ee. The enantiomeric excess was determined by HPLC on Venusil Chiral OD-H with hexane/i-PrOH (94:6) as the eluent. Flow rate: 0.5 mL/min, λ = 254nm: Ent-1 = 21.768 min, Ent-2 = 24.152 min. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.27 (t, *J* = 7.2 Hz, 3H), 2.58 (dd, *J* = 18.3 Hz, 1H), 3.05 (dd, *J* = 18.3 Hz, 1H), 4.14 (m, 1H), 4.42 (m, *J* = 7.2 Hz, 2H), 4.67 (q, *J* = 15 Hz, 2H), 7.25-7.37 (m, 5H), 7.46-7.51 (m, 2H), 7.62 (m, 1H), 8.12 (m, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 13.8, 30.8, 42.7, 45.0, 45.3, 63.5, 128.0, 128.6, 128.7, 128.8, 130.1, 130.2, 134.9, 174.6; MS (ACPI) m/z : 398.1 (M^++1).



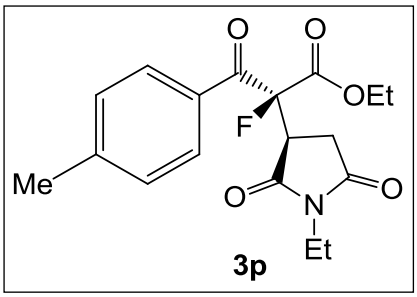
Retention Time		Area %
(Ent-1)	21.768	74.46
(Ent-2)	24.152	4.63
	26.620	11.20
	30.484	9.70
	Totals	100.00

Chiral sample catalyzed by c-5



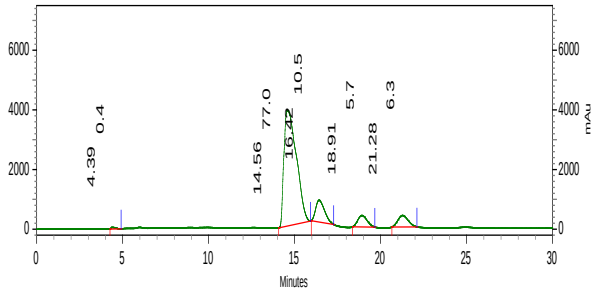
Retention Time		Area %
(Ent-1)	23.816	49.69
(Ent-2)	26.144	50.31
	26.620	0
	30.484	0
	Totals	100.00

Racemic sample



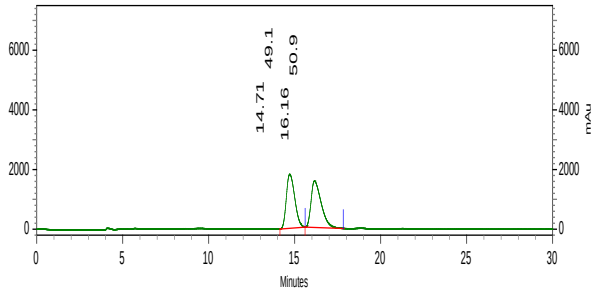
3p

3p: 92% yield (> 20:1 dr), 77% ee. The enantiomeric excess was determined by HPLC on Venusil Chiral OD-H with hexane/i-PrOH (95:5) as the eluent. Flow rate: 0.5 mL/min, λ = 254nm: Ent-1 = 14.564 min, Ent-2 = 16.420 min. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.26 (t, *J* = 7.2 Hz, 3H), 1.31 (t, *J* = 7.5 Hz, 3H), 2.42 (s, 3H), 2.58 (dd, *J* = 18.6 Hz, 1H), 3.06 (dd, *J* = 18.6 Hz, 1H), 3.58 (q, J = 7.5 Hz, 2H), 4.13 (m, 1H), 4.42 (m, *J* = 7.2 Hz, 2H), 7.27 (d, *J* = 0.9 Hz, 2H), 8.02 (d, *J* = 1.8 Hz, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 12.9, 13.8, 21.8, 34.1, 44.9, 45.2, 63.4, 117.2, 129.5, 130.3, 130.4, 146.2, 163.8, 171.9, 172.4, 174.9; MS (ACPI) m/z : 350.1 (M^{+}+1).



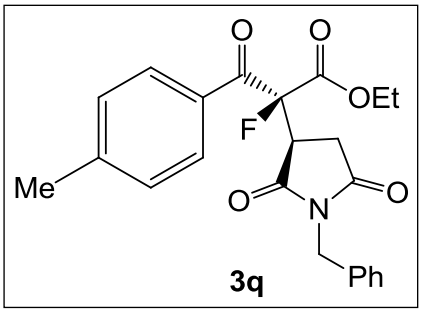
Retention Time		Area %
(Ent-1)	14.564	77.05
(Ent-2)	16.420	10.47
	18.912	5.73
	21.276	6.35
	Totals	100.00

Chiral sample catalyzed by c-5



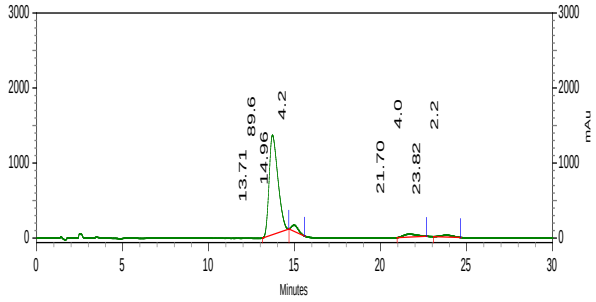
Retention Time		Area %
(Ent-1)	14.712	49.05
(Ent-2)	16.156	50.95
	18.912	0
	21.276	0
	Totals	100.00

Racemic sample



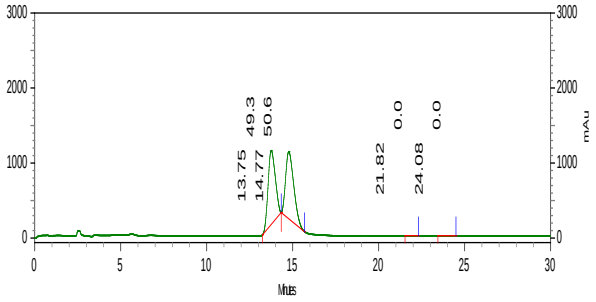
3q

3q: 95% yield (> 20:1 dr), 91% ee. The enantiomeric excess was determined by HPLC on Venusil Chiral OD-H with hexane/i-PrOH (94:6) as the eluent. Flow rate: 0.5 mL/min, λ = 254nm: Ent-1= 13.712 min, Ent-2 = 14.956 min. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.26 (t, *J* = 7.2 Hz, 3H), 2.42 (s, 3H), 2.58 (dd, *J* = 18.3 Hz, 1H), 3.05 (dd, *J* = 18.3 Hz, 1H), 4.14 (m, 1H), 4.68 (m, *J* = 14.1 Hz, 2H), 7.26-7.37 (m, 7H), 8.01 (m, *J* = 1.8 Hz, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 13.8, 21.8, 30.8, 45.0, 45.3, 63.3, 97.8, 100.5, 128.0, 128.5, 128.5, 128.6, 129.5, 130.0, 130.1, 130.3, 130.4, 135.2, 146.2, 165.2, 165.5, 172.1, 172.4, 174.4, 174.7, 188.7, 189.0; MS (ACPI) m/z : 412.2 (M^{+}+1).



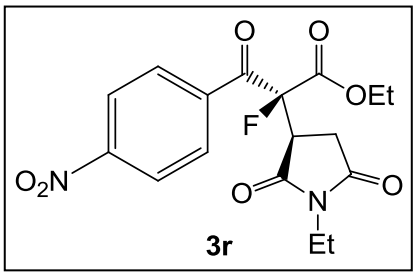
Retention Time		Area %
(Ent-1)	13.712	89.60
(Ent-2)	14.956	4.18
	21.704	4.03
	23.820	2.19
	Totals	100.00

Chiral sample catalyzed by c-5



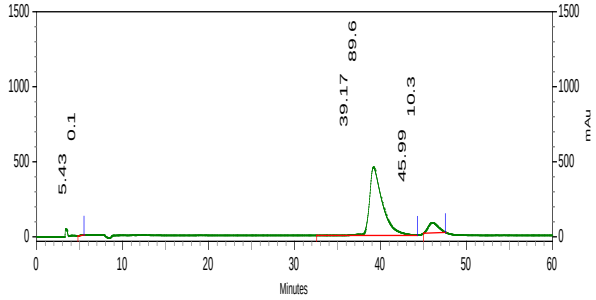
Retention Time		Area %
(Ent-1)	13.752	49.31
(Ent-2)	14.772	50.63
	21.824	0.02
	24.080	0.04
	Totals	100.00

Racemic sample



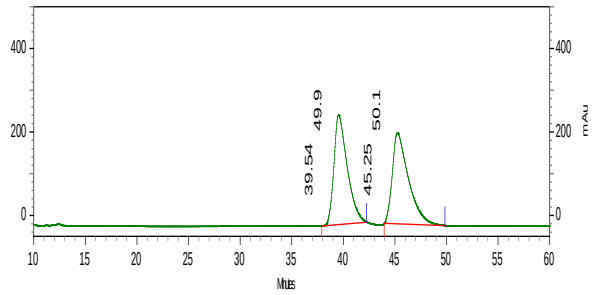
3r

3r: 98% yield (> 20:1 dr), 80% ee. The enantiomeric excess was determined by HPLC on Venusil Chiral OD-H with hexane/i-PrOH (94:6) as the eluent. Flow rate: 0.8 mL/min, λ = 254nm: Ent-1 = 39.172 min, Ent-2 = 45.988 min. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.22 (t, *J* = 7.2 Hz, 3H), 1.33 (t, *J* = 7.2 Hz, 3H), 2.55 (dd, *J* = 18.3 Hz, 1H), 3.06 (dd, *J* = 18.3 Hz, 1H), 3.60 (q, *J* = 7.2 Hz, 2H), 4.12 (m, 1H), 4.43 (m, *J* = 7.2 Hz, 2H), 8.28-8.35 (m, 4H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 12.9, 13.9, 30.6, 30.7, 34.2, 44.7, 45.0, 63.9, 123.9, 131.3, 131.4, 171.4, 174.3; MS (ACPI) m/z : 381.1 (M^{+}+1).



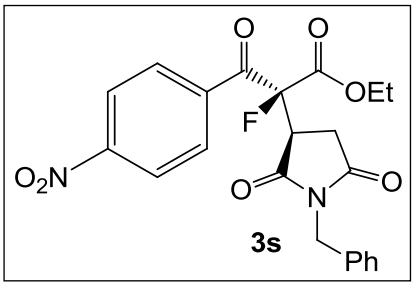
Retention Time	Area %
(Ent-1) 39.172	89.60
(Ent-2) 45.988	10.27
Totals	100.00

Chiral sample catalyzed by c-5

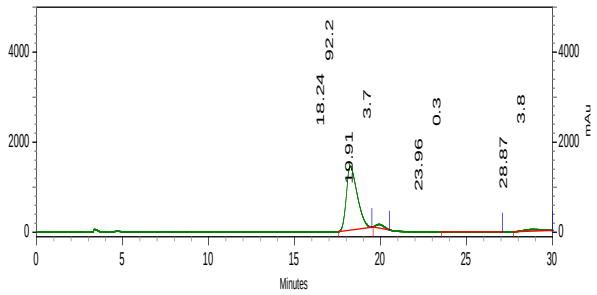


Retention Time	Area %
(Ent-1) 39.544	49.89
(Ent-2) 45.248	50.11
Totals	100.00

Racemic sample

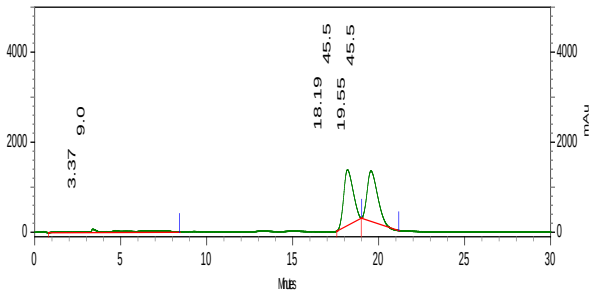


3s: 96% yield (> 20:1 dr), 94% ee. The enantiomeric excess was determined by HPLC on Venusil Chiral OD-H with hexane/i-PrOH (94:6) as the eluent. Flow rate: 0.8 mL/min, λ = 254nm: Ent-1 = 18.236 min, Ent-2 = 19.908 min. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.29 (t, *J* = 7.2 Hz, 3H), 2.57 (dd, *J* = 18.3 Hz, 1H), 3.07 (dd, *J* = 18.3 Hz, 1H), 4.13 (m, 1H), 4.46 (m, *J* = 7.2 Hz, 2H), 4.68 (q, J = 13.4 Hz, 2H), 7.27-7.38 (m, 5H), 7.46-7.51 (m, 5H), 8.27-8.34 (m, 4H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 13.8, 30.5, 42.7, 44.7, 44.9, 63.8, 97.7, 100.4, 123.8, 128.0, 128.5, 128.6, 131.2, 131.3, 135.1, 136.7, 150.9, 164.3, 164.7, 173.7, 174.1, 188.3, 188.6; MS (ACPI) m/z : 443.2 (M^{+}+1).



Retention Time		Area %
(Ent-1)	18.236	92.18
(Ent-2)	19.908	3.73
	23.964	0.28
	28.868	3.80
	Totals	100.00

Chiral sample catalyzed by c-5



Retention Time		Area %
(Ent-1)	18.188	45.49
(Ent-2)	19.552	45.49
	23.964	0.28
	28.868	3.80
	Totals	100.00

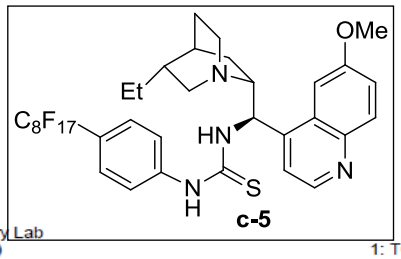
Racemic sample

4. HRMS and LC-MS spectra

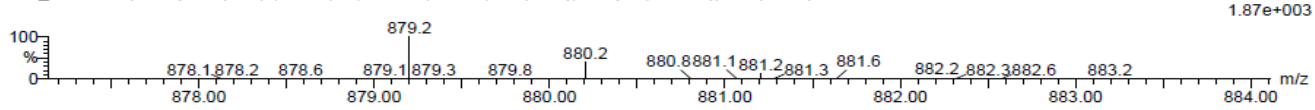
Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 10.0 PPM / DBE: min = -1.5, max = 150.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

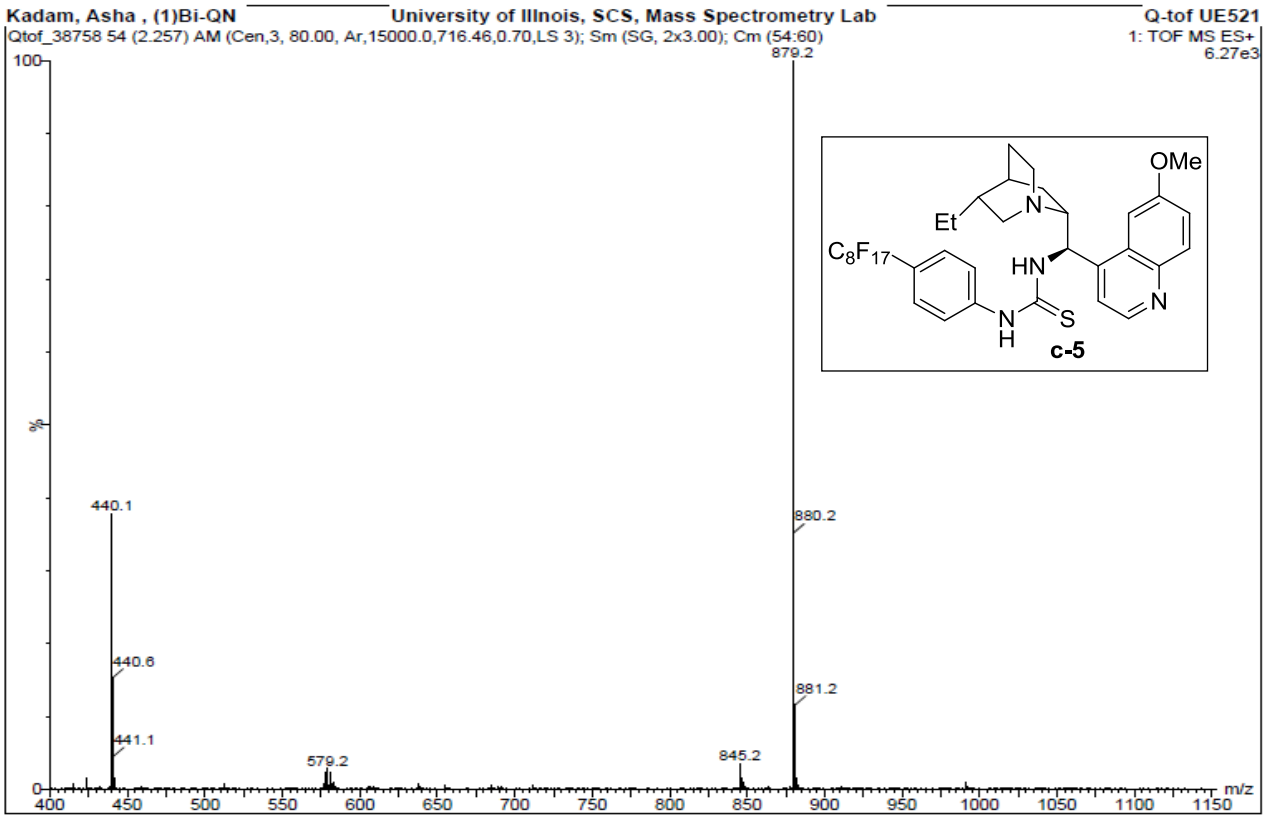
Monoisotopic Mass, Even Electron Ions
83 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-150 H: 0-200 N: 3-5 O: 1-5 F: 17-17 S: 1-1



Kadam, Asha , (1)Bi-QN University of Illnois, SCS, Mass Spectrometry Lab Q-tof UE521
Qtof_38758 69 (2.900) AM (Cen,3, 80.00, Ar,15000.0,716.46,0.70,LS 3); Sm (SG, 2x3.00); Cm (69:82) 1: TOF MS ES+
1.87e+003



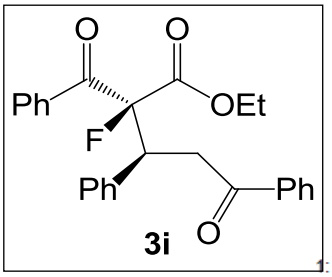
Minimum:				-1.5		
Maximum:		5.0	10.0	150.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
879.2010	879.2025	-1.5	-1.7	13.5	1.5	C35 H32 N4 O F17 S



Elemental Composition Report

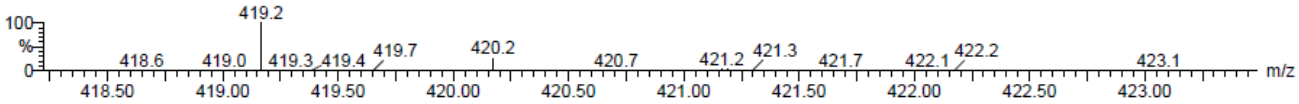
Single Mass Analysis

Tolerance = 10.0 PPM / DBE: min = -1.5, max = 150.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3



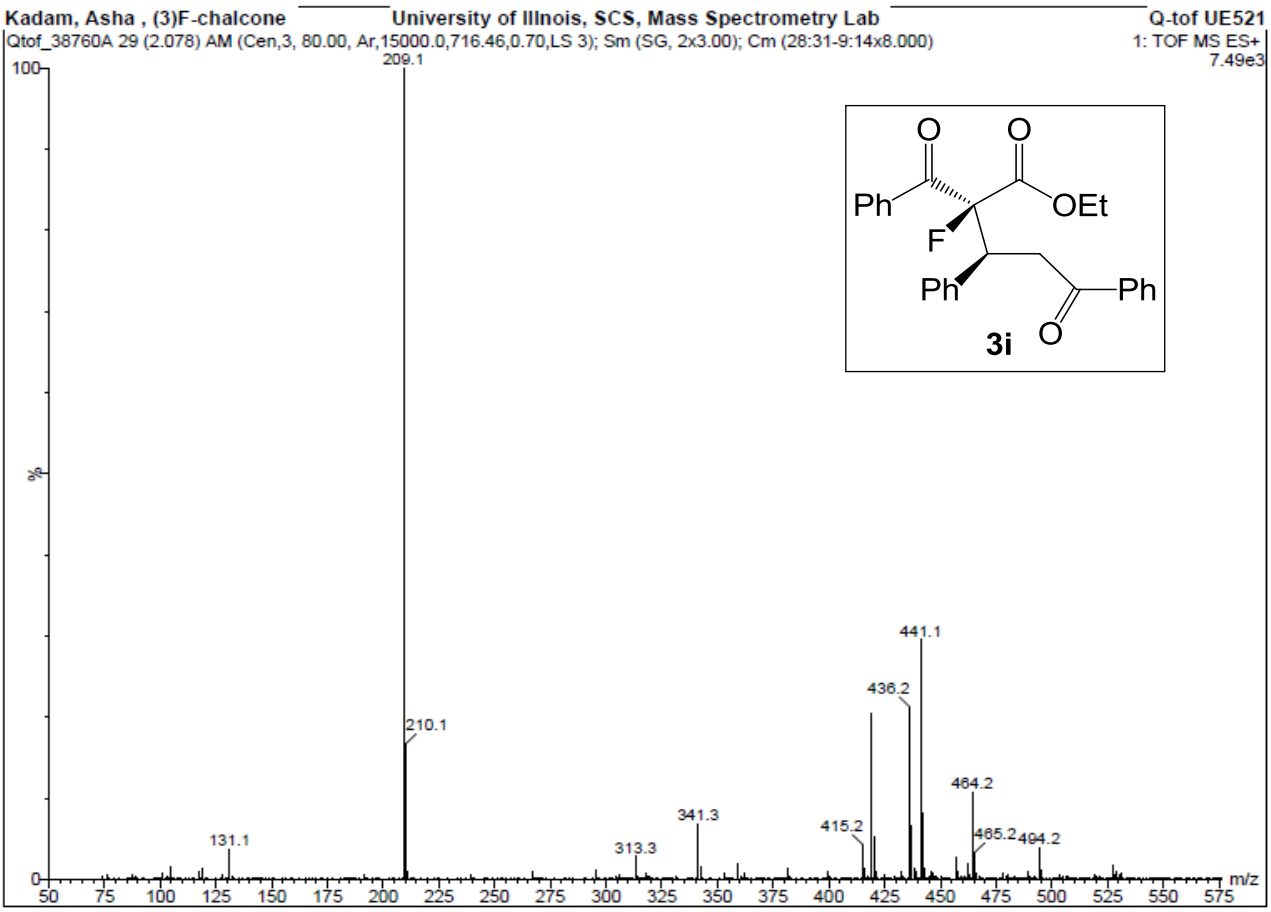
Monoisotopic Mass, Even Electron Ions
25 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-150 H: 0-200 O: 1-5 F: 1-1

Kadam, Asha , (3)F-chalcone — University of Illnois, SCS, Mass Spectrometry Lab — Q-tof UE521
Qtof_38760A 30 (2.150) AM (Cen,3, 80.00, Ar,15000.0,716.46,0.70,LS 3); Sm (SG, 2x3.00); Cm (30) — 1: TOF MS ES+ 3.97e+002

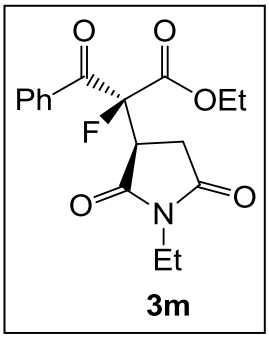


Minimum:				-1.5		
Maximum:		5.0	10.0	150.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
419.1667	419.1659	0.8	1.9	14.5	0.9	C26 H24 O4 F

Kadam, Asha , (3)F-chalcone — University of Illnois, SCS, Mass Spectrometry Lab — Q-tof UE521
Qtof_38760A 29 (2.078) AM (Cen,3, 80.00, Ar,15000.0,716.46,0.70,LS 3); Sm (SG, 2x3.00); Cm (28:31-9:14x8.000) — 1: TOF MS ES+ 7.49e3



Elemental Composition Report



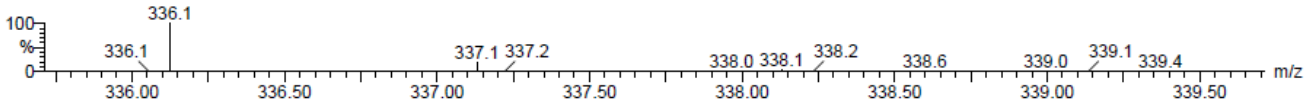
3m

Single Mass Analysis

Tolerance = 10.0 PPM / DBE: min = -1.5, max = 150.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

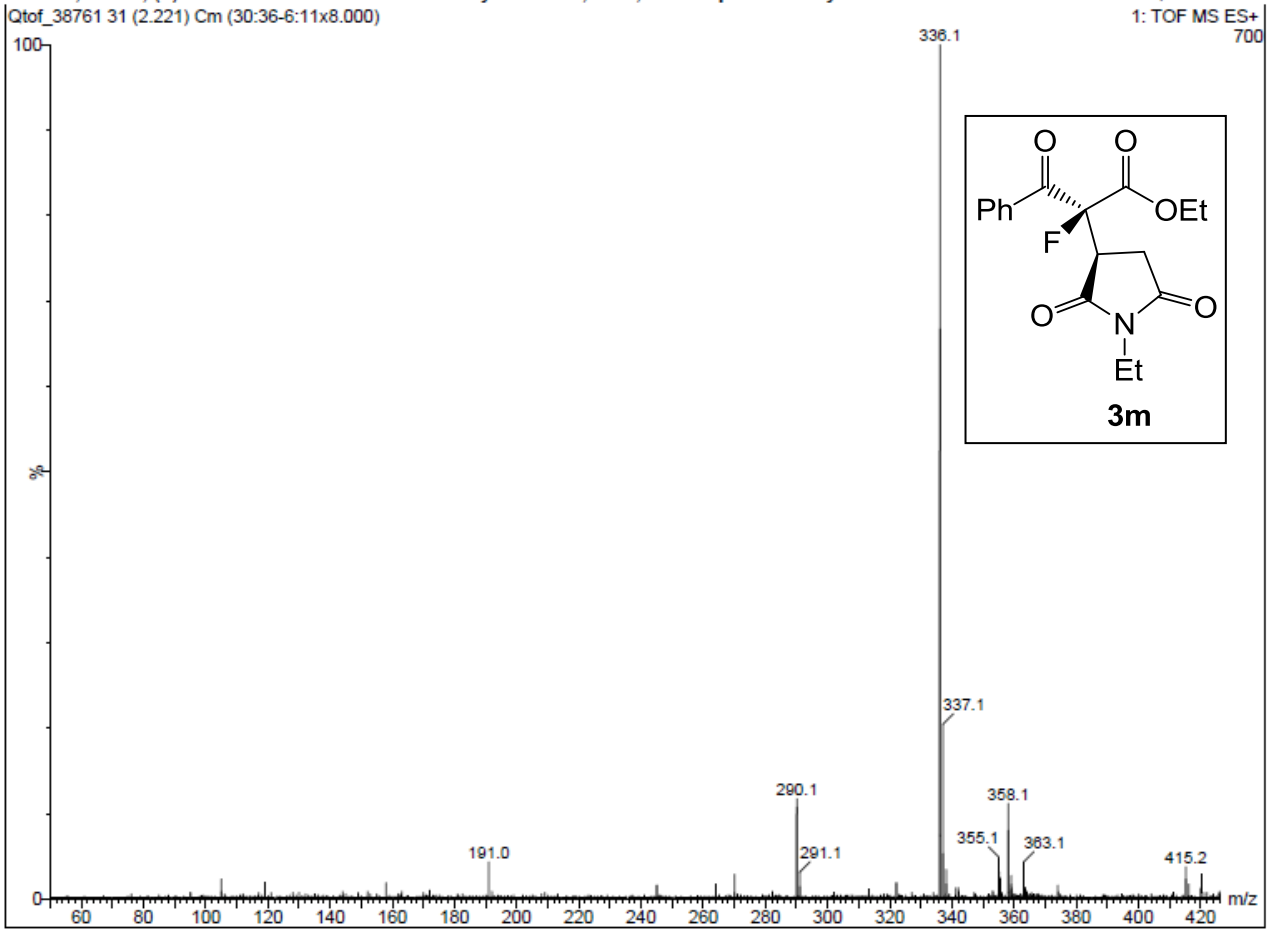
Monoisotopic Mass, Even Electron Ions
96 formula(e) evaluated with 2 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-150 H: 0-200 N: 1-5 O: 1-5 F: 1-1

Kadam, Asha , (4)F-malonide — University of Illnois, SCS, Mass Spectrometry Lab — Q-tof UE521
Qtof_38761 32 (2.293) AM (Cen,3, 80.00, Ar,15000.0,716.46,0.70,LS 3); Sm (SG, 2x3.00); Cm (32) — 1: TOF MS ES+ 1.08e+003

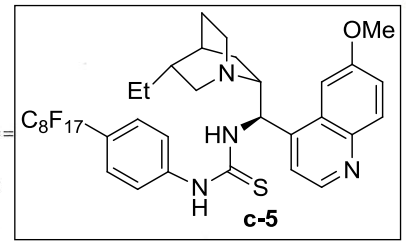


Minimum:				-1.5		
Maximum:		5.0	10.0	150.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
336.1250	336.1247	0.3	0.9	8.5	1.1	C17 H19 N O5 F
	336.1261	-1.1	-3.3	13.5	3.2	C18 H15 N5 O F

Kadam, Asha , (4)F-malonide — University of Illnois, SCS, Mass Spectrometry Lab — Q-tof UE521
Qtof_38761 31 (2.221) Cm (30:36-6:11x8.000) — 1: TOF MS ES+ 700



3m

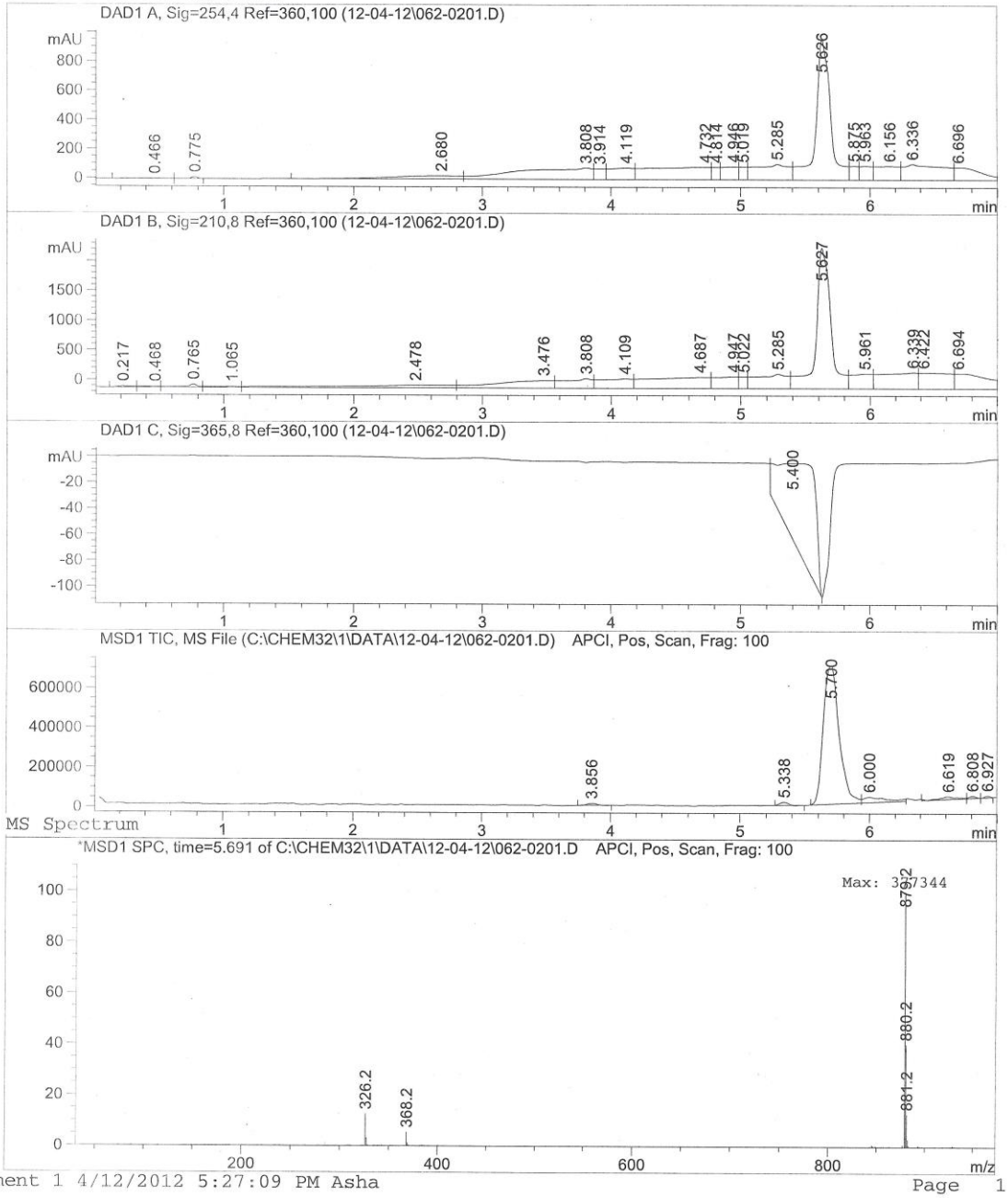


Print of all graphic windows
Data File : C:\CHEM32\1\DATA\12-04-12\062-0201.D
Sample Name : hqqd-hqtu-3

Acq. Operator : Asha
Acq. Instrument : Instrument 1
Injection Date : 4/12/2012 4:26:30 PM
Seq. Line : 2
Location : Vial 62
Inj : 1
Inj Volume : 4 µl

Method : C:\CHEM32\1\METHODS\CINCHONA.M
Last changed : 3/23/2012 1:40:38 PM by Asha

Current Chromatogram(s)

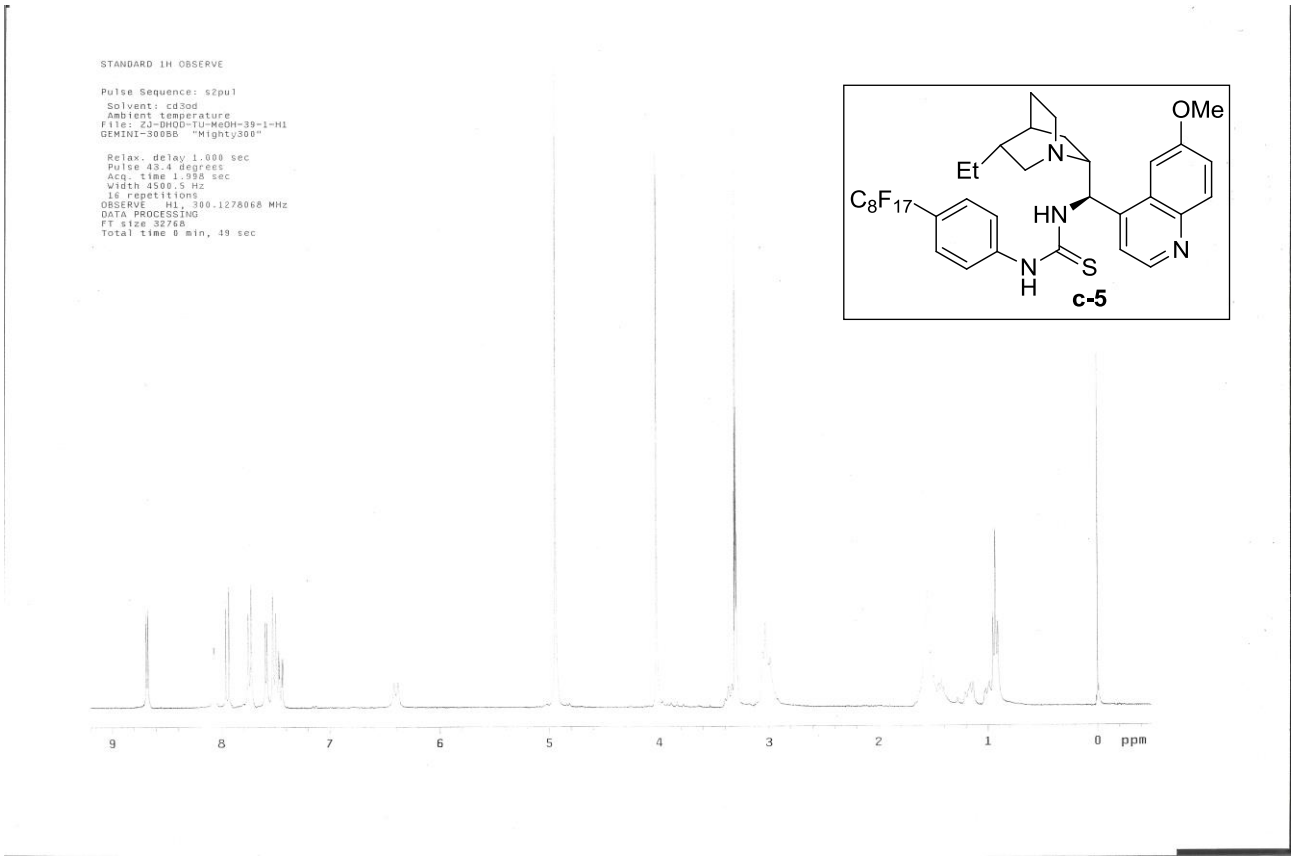


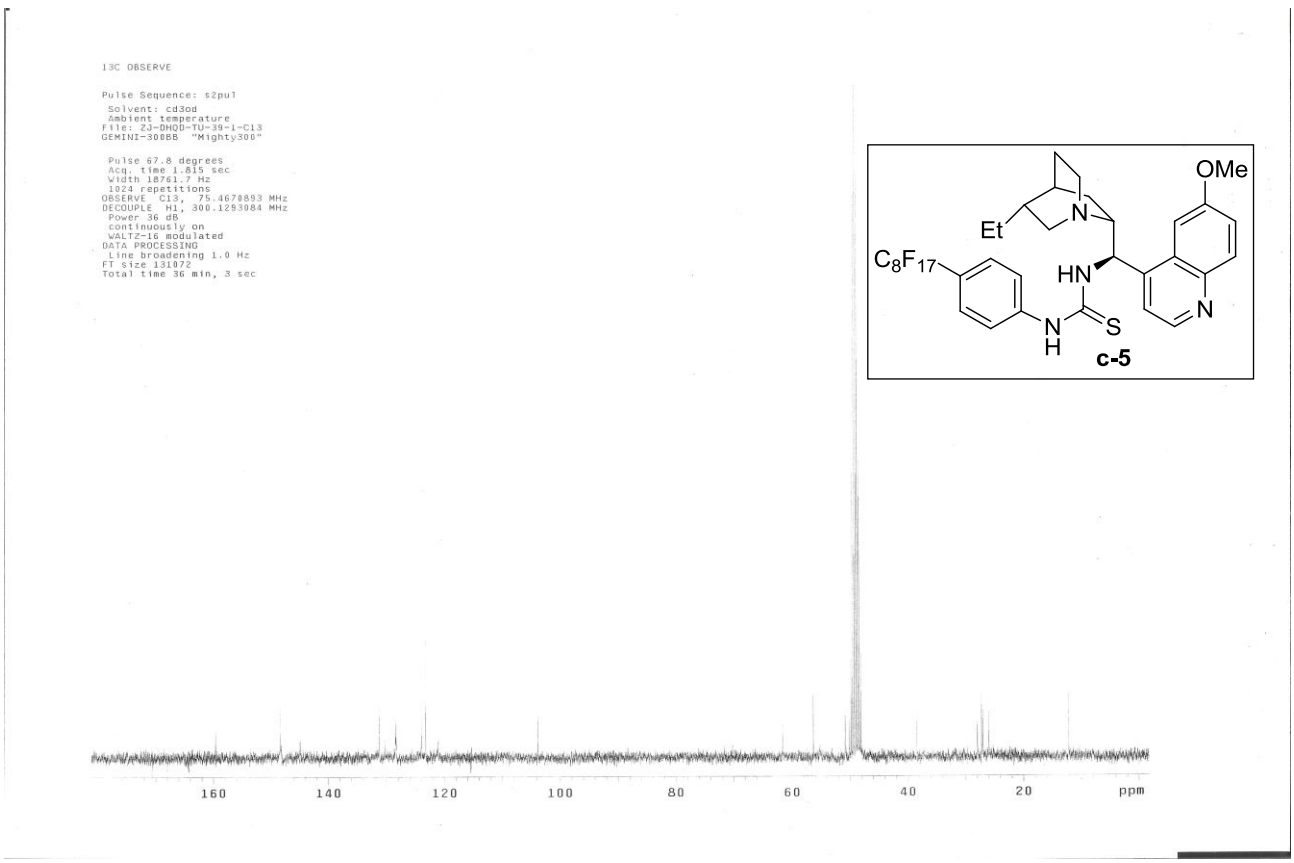
MS Spectrum

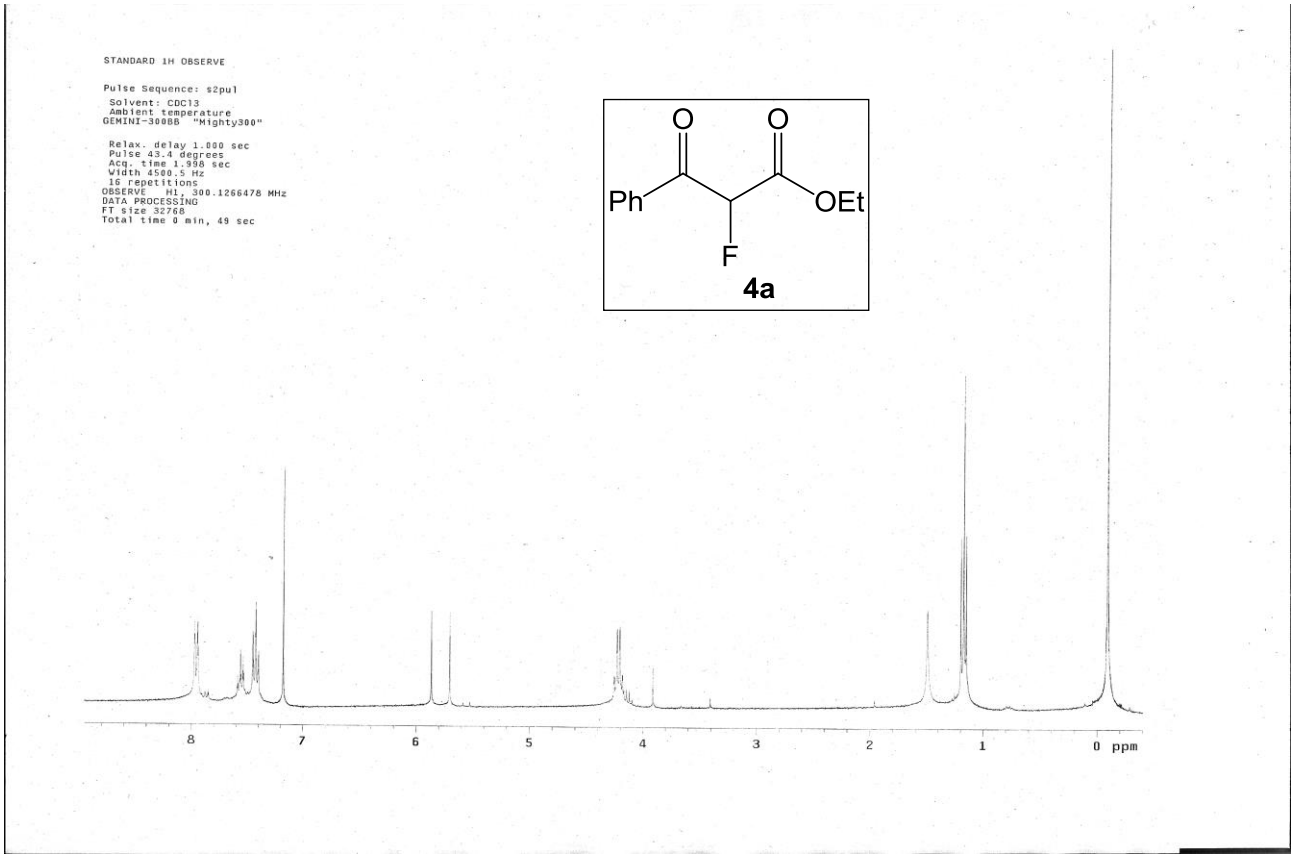
Instrument 1 4/12/2012 5:27:09 PM Asha

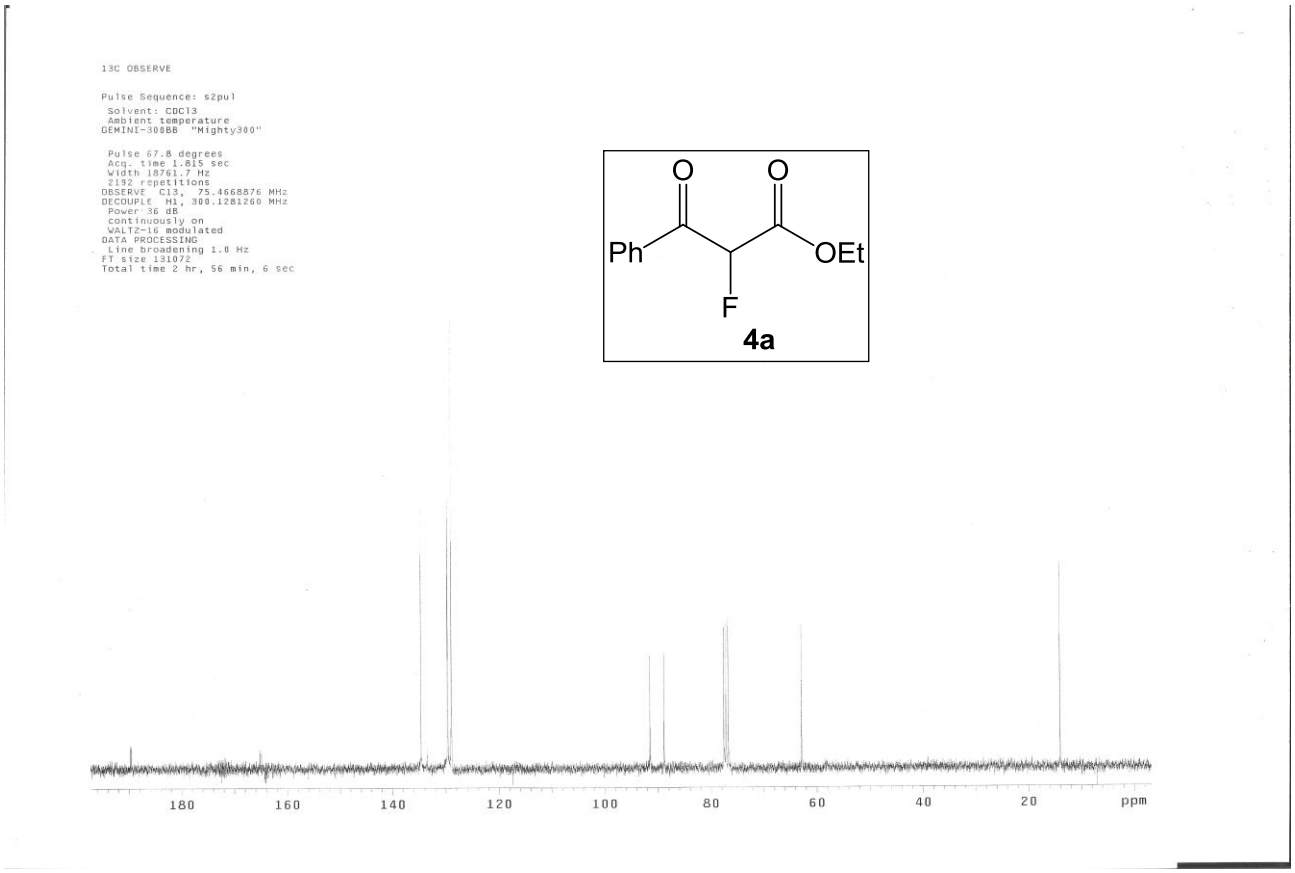
Page 1 of 1

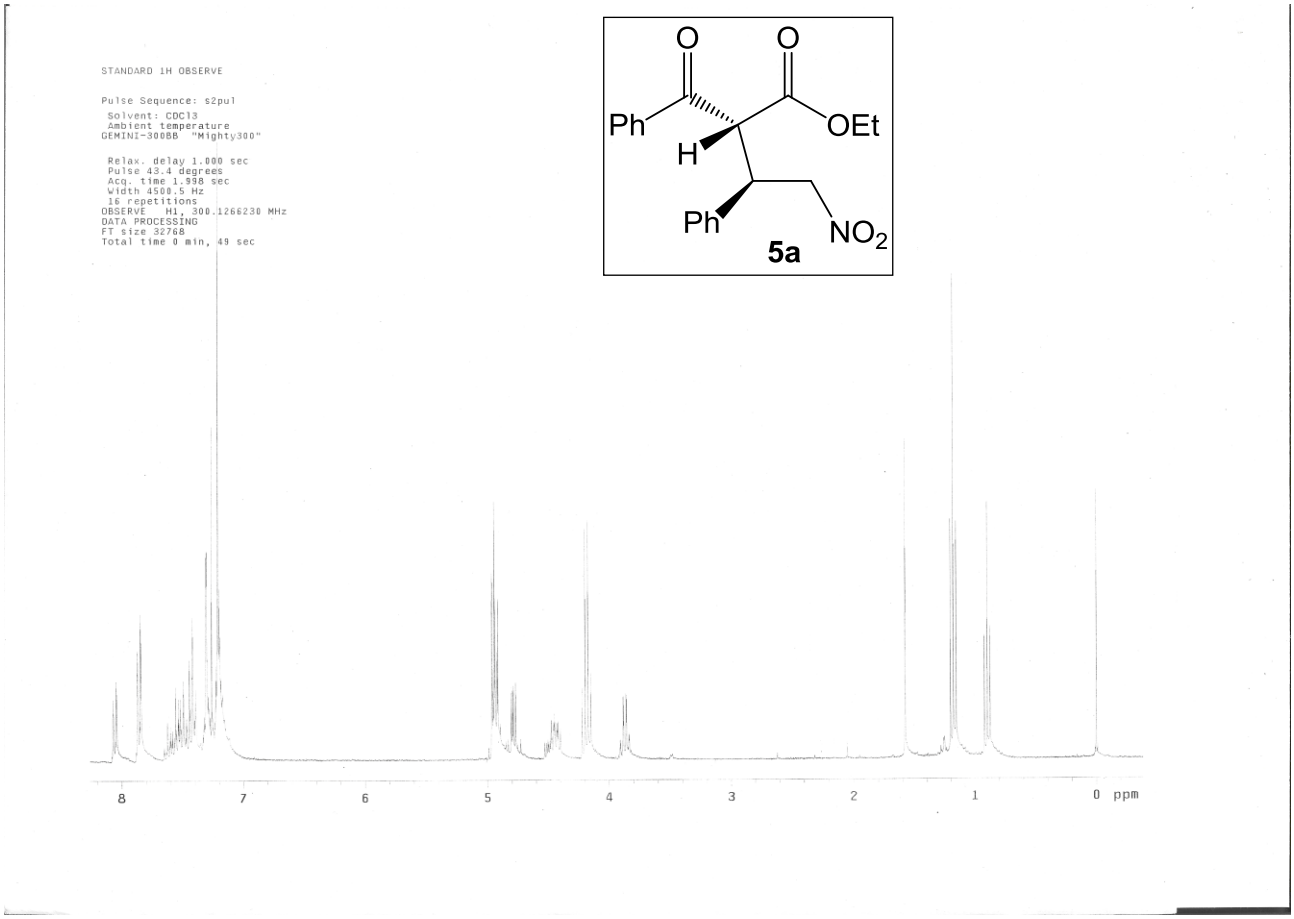
5. NMR Spectra

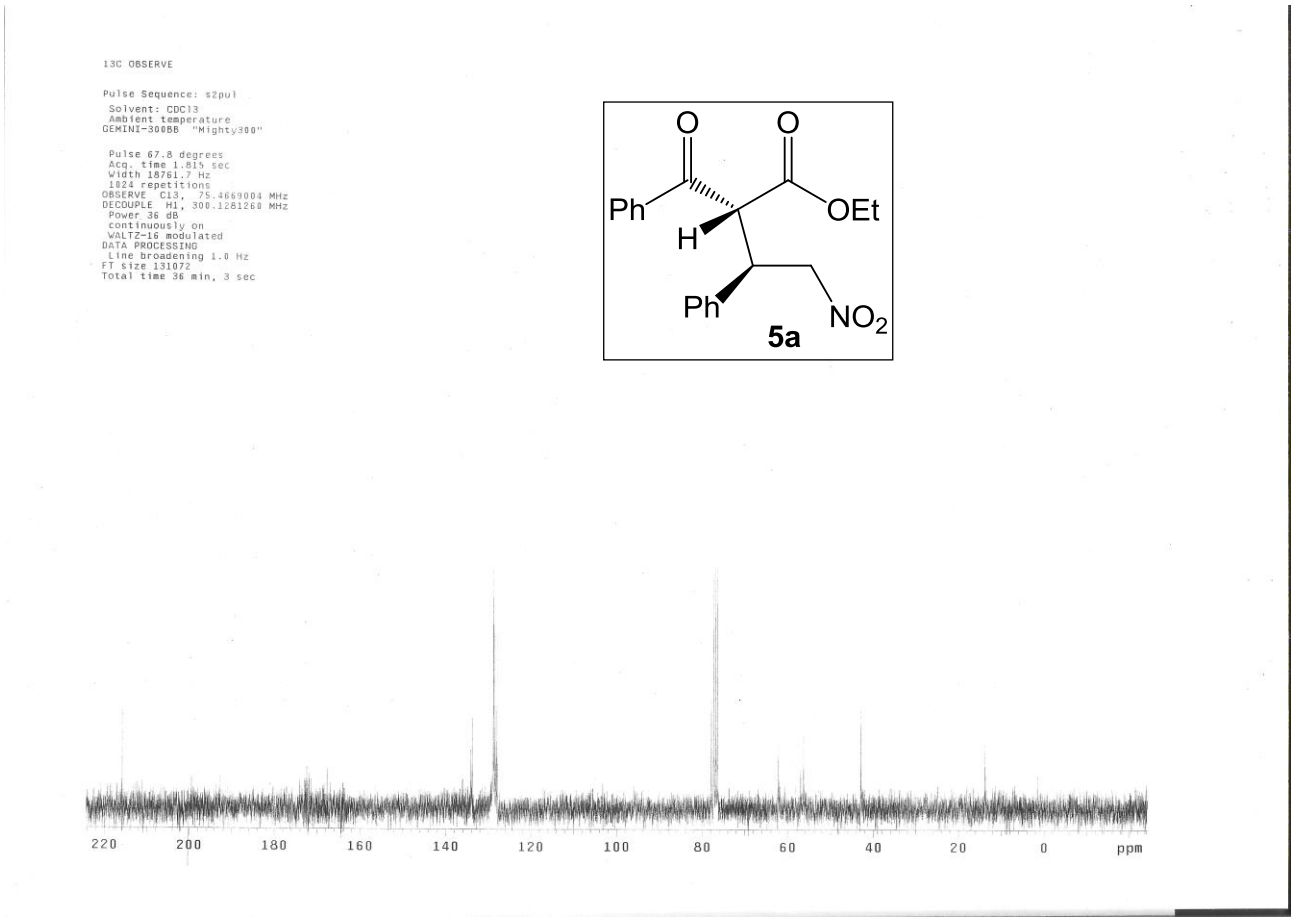


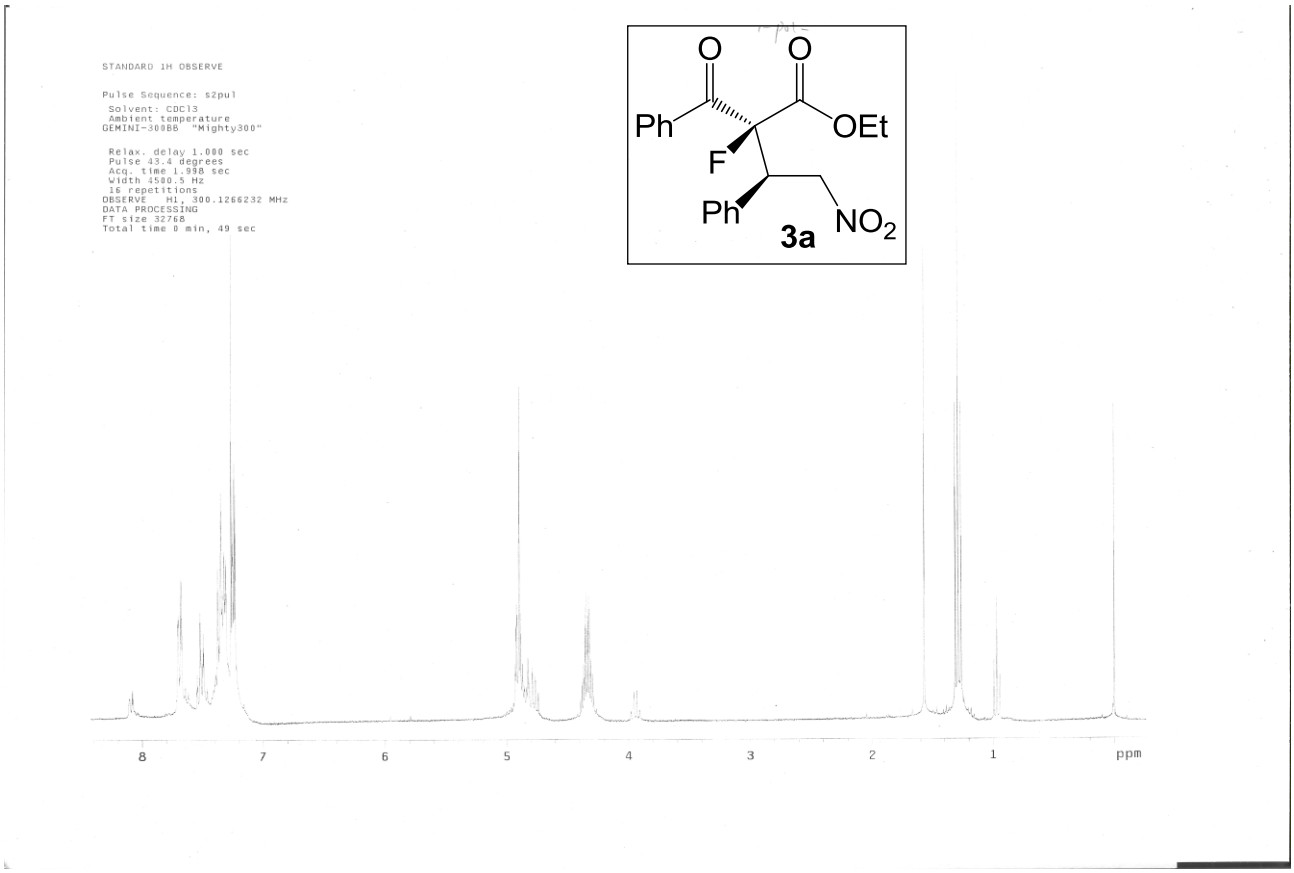


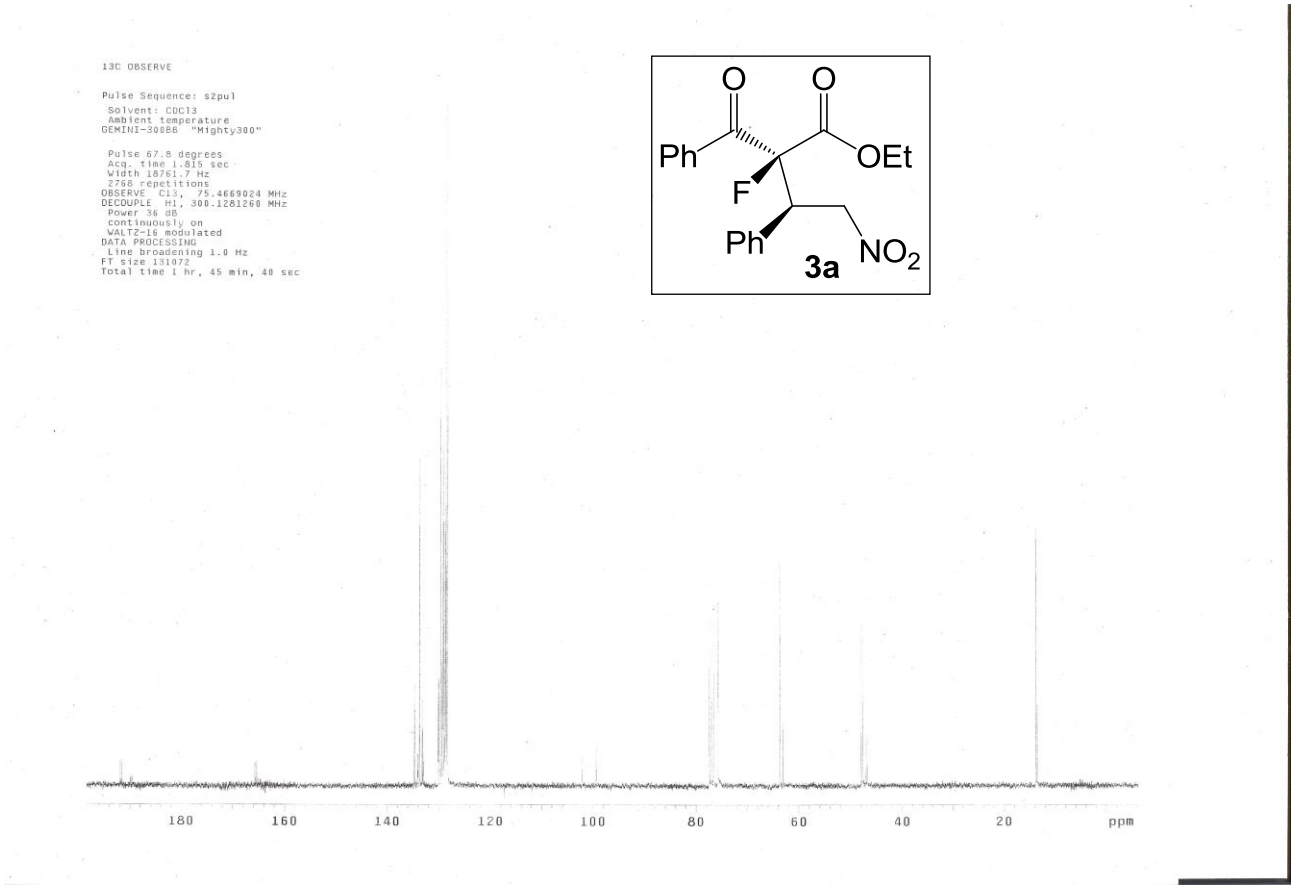

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "Mighty300"
Relax. delay 1.000 sec
Pulse 43.4 degrees
Acq. time 1.998 sec
Width 4500.5 Hz
16 repetitions
OBSERVE H1, 300.1266478 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 49 sec
O
O
Ph
OEt
F
4a
8
7
6
5
4
3
2
1
0 ppm

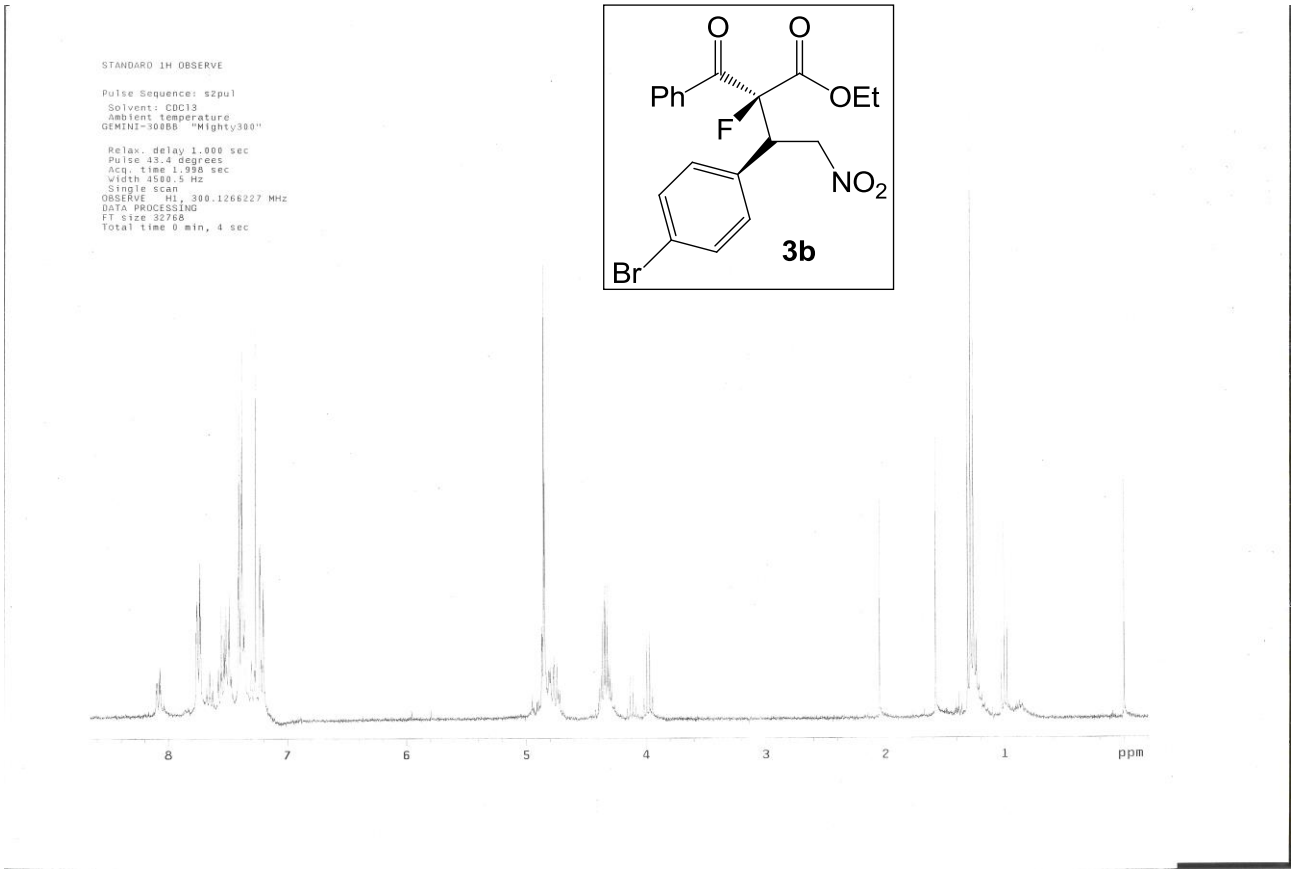

13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "Mighty300"
Pulse 67.8 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
2192 repetitions
OBSERVE C13, 75.4668876 MHz
DECOUPLE H1, 300.1281260 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 2 hr, 56 min, 6 sec
O
O
Ph
OEt
F
4a
180
160
140
120
100
80
60
40
20
ppm

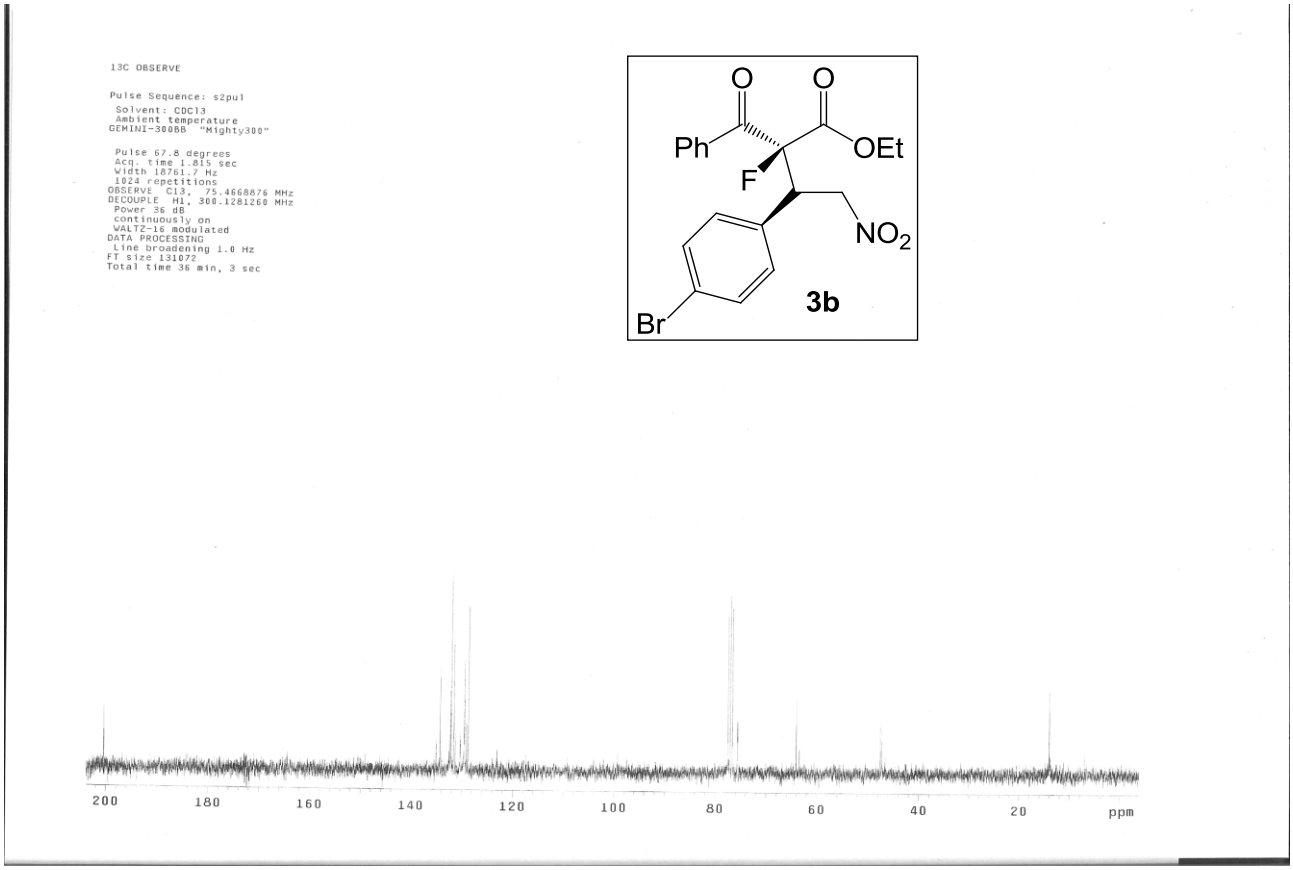


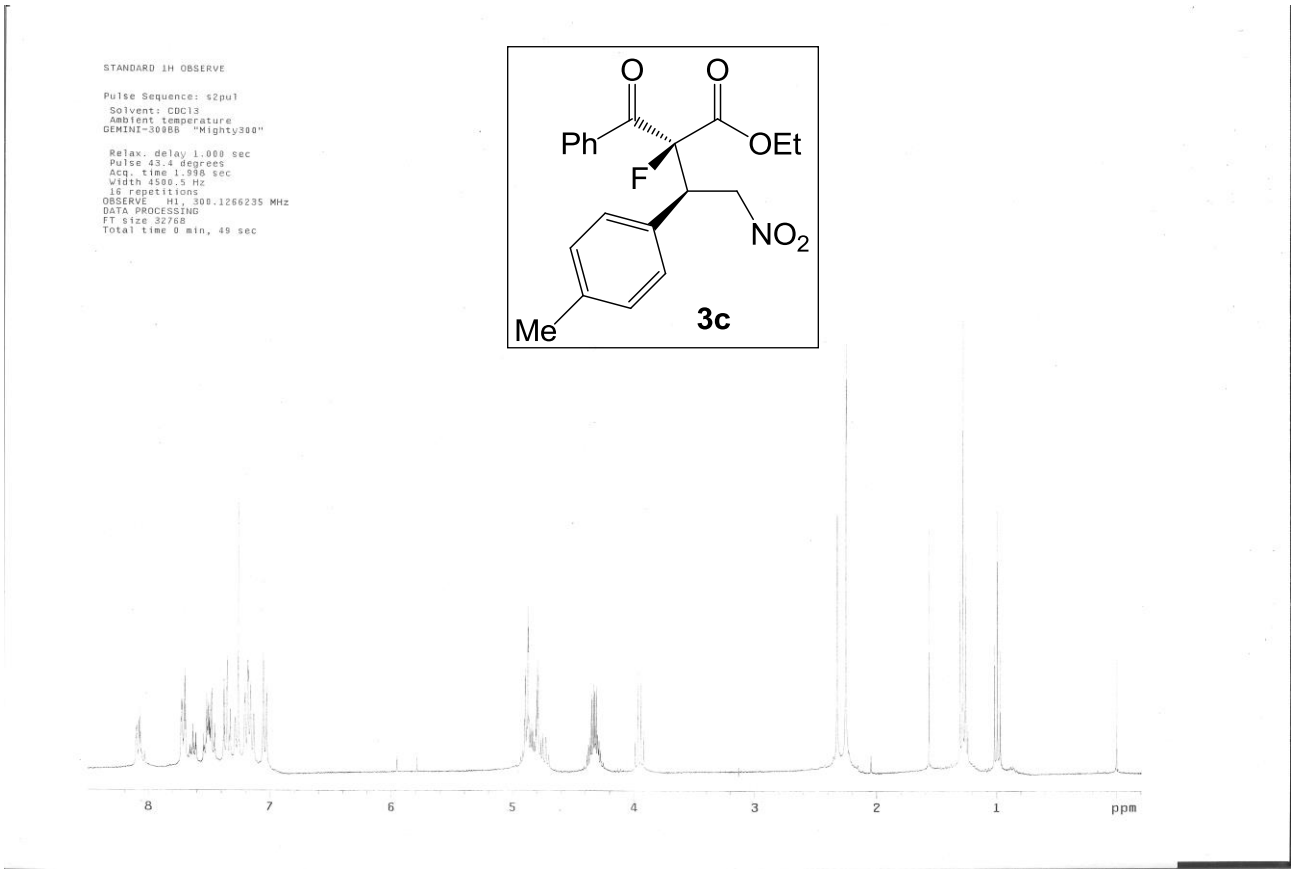


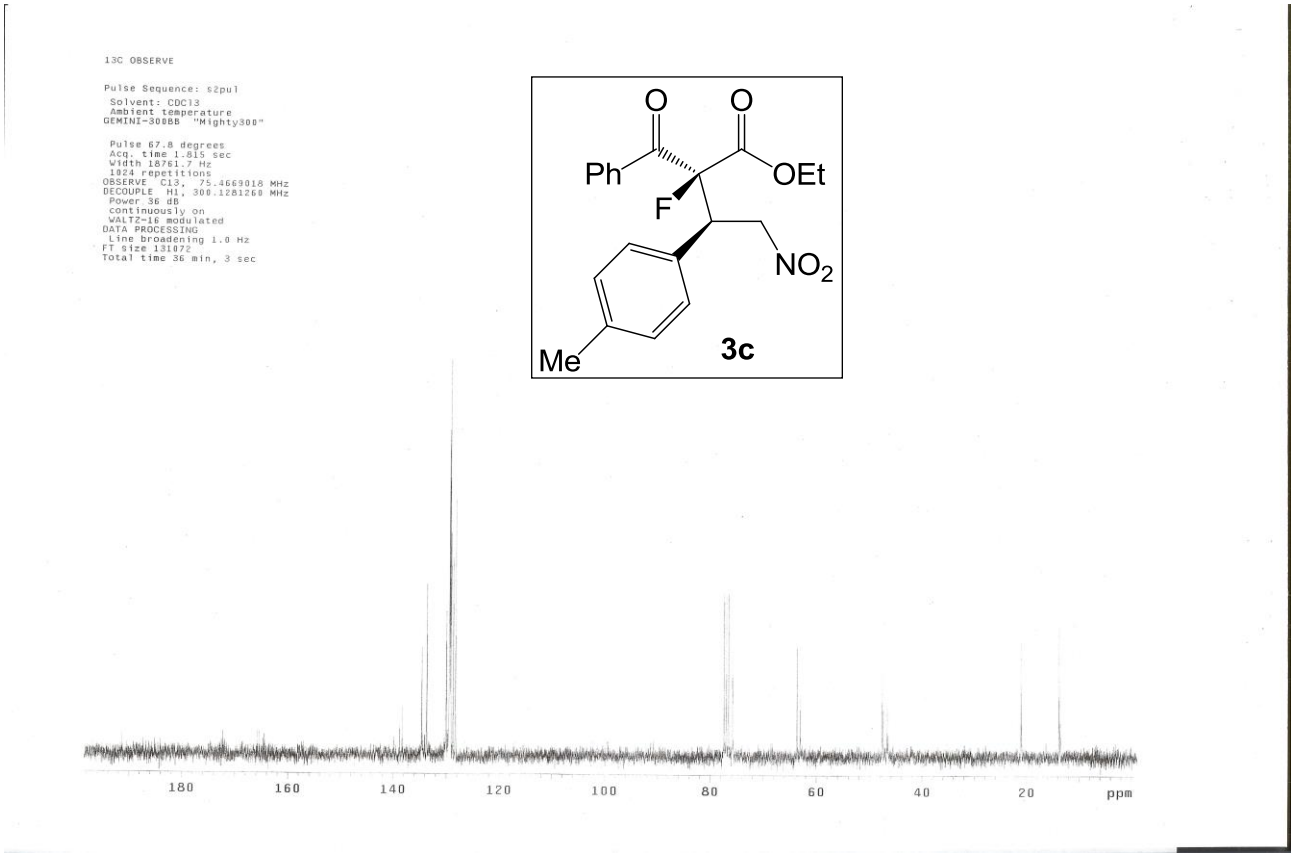

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "Mighty300"
Relax. delay 1.000 sec
Pulse 43.4 degrees
Acq. time 1.998 sec
Width 4500.5 Hz
16 repetitions
OBSERVE H1, 300.1266232 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 49 sec
O
O
Ph
OEt
F
Ph
NO2
3a
8
7
6
5
4
3
2
1
ppm

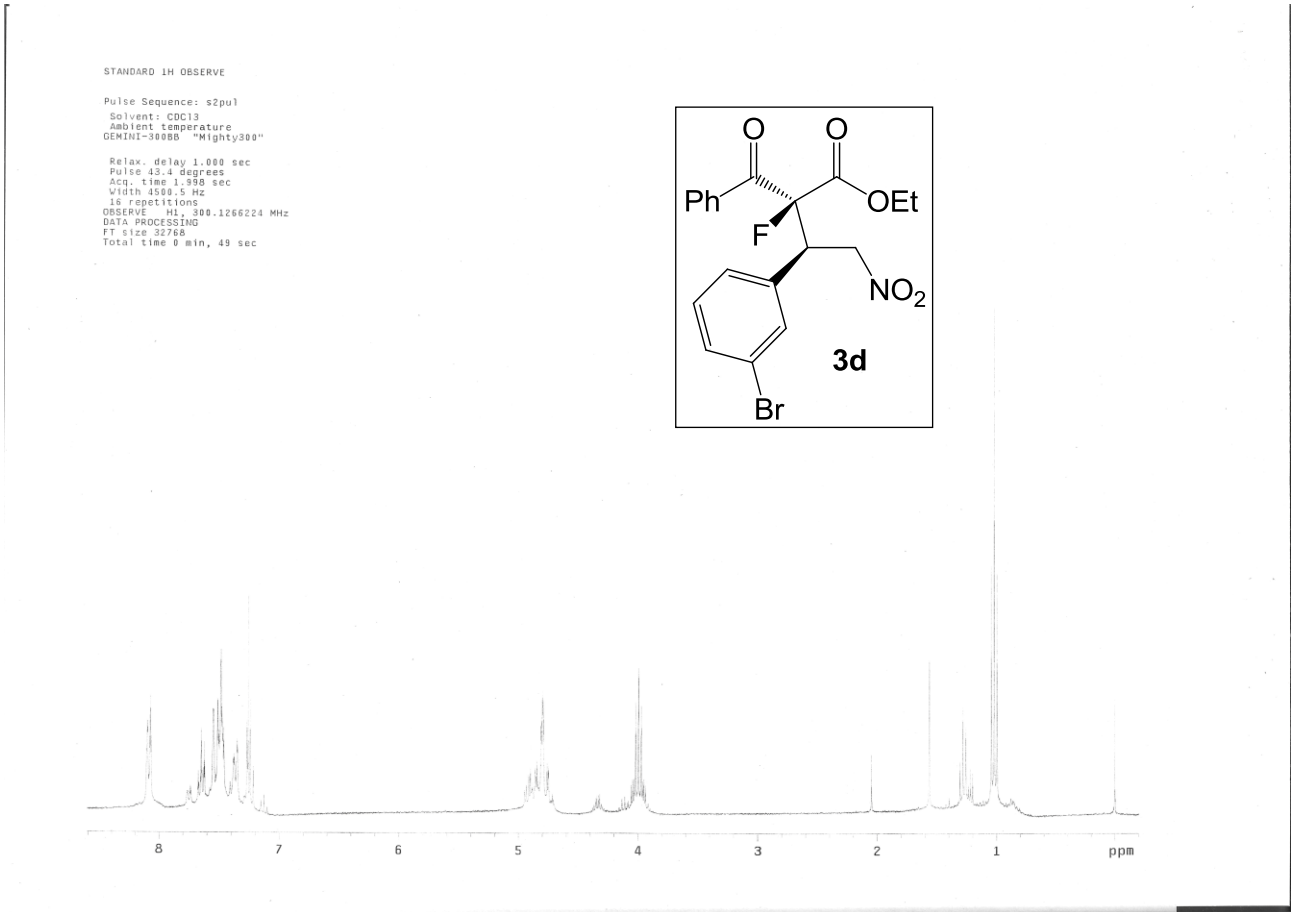

13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "Mighty300"
Pulse 67.8 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
2768 repetitions
OBSERVE C13, 75.4669024 MHz
DECOUPLE H1, 300.1281260 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 hr, 45 min, 40 sec
O
O
Ph
OEt
F
Ph
NO2
3a
180
160
140
120
100
80
60
40
20
ppm

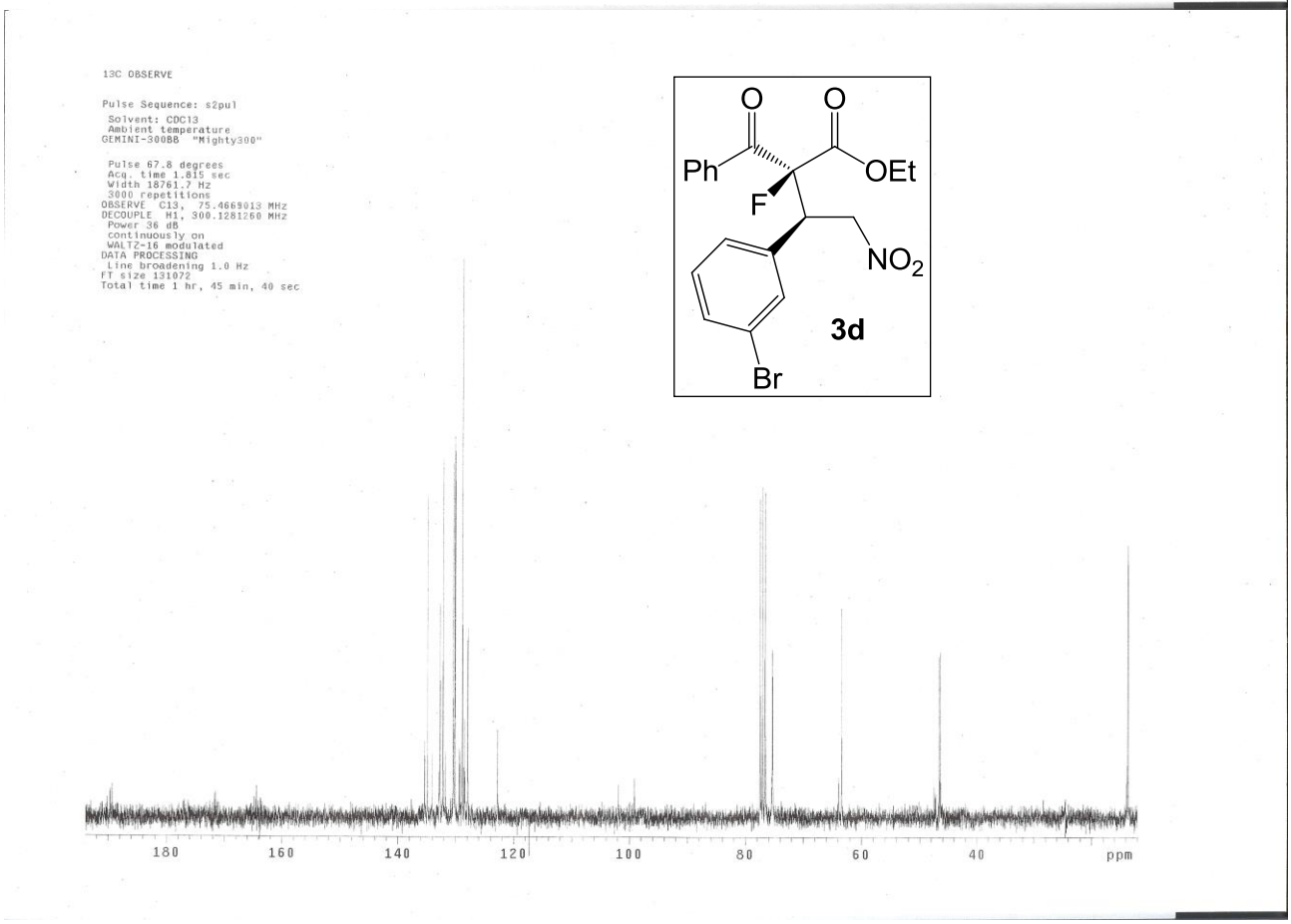


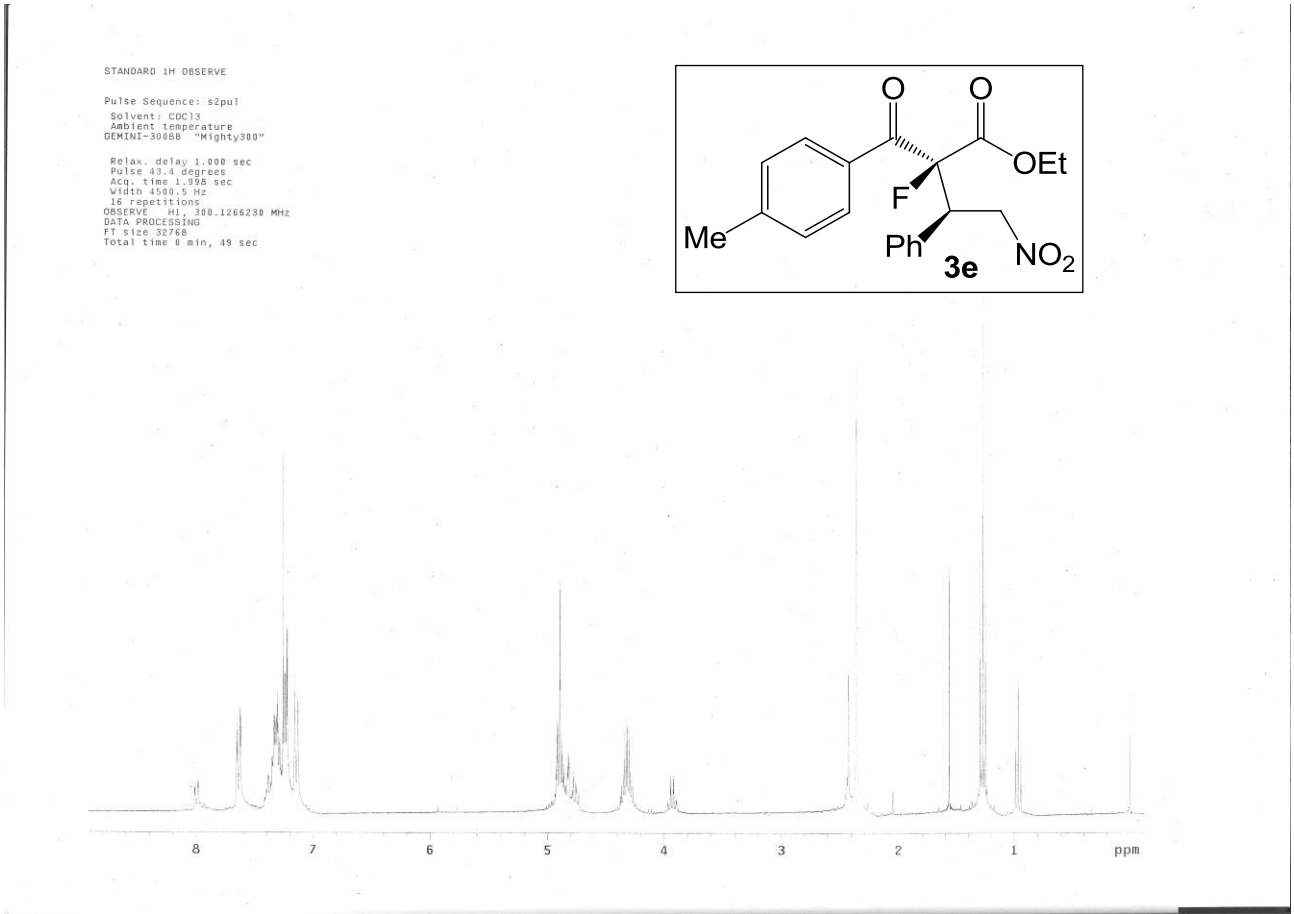


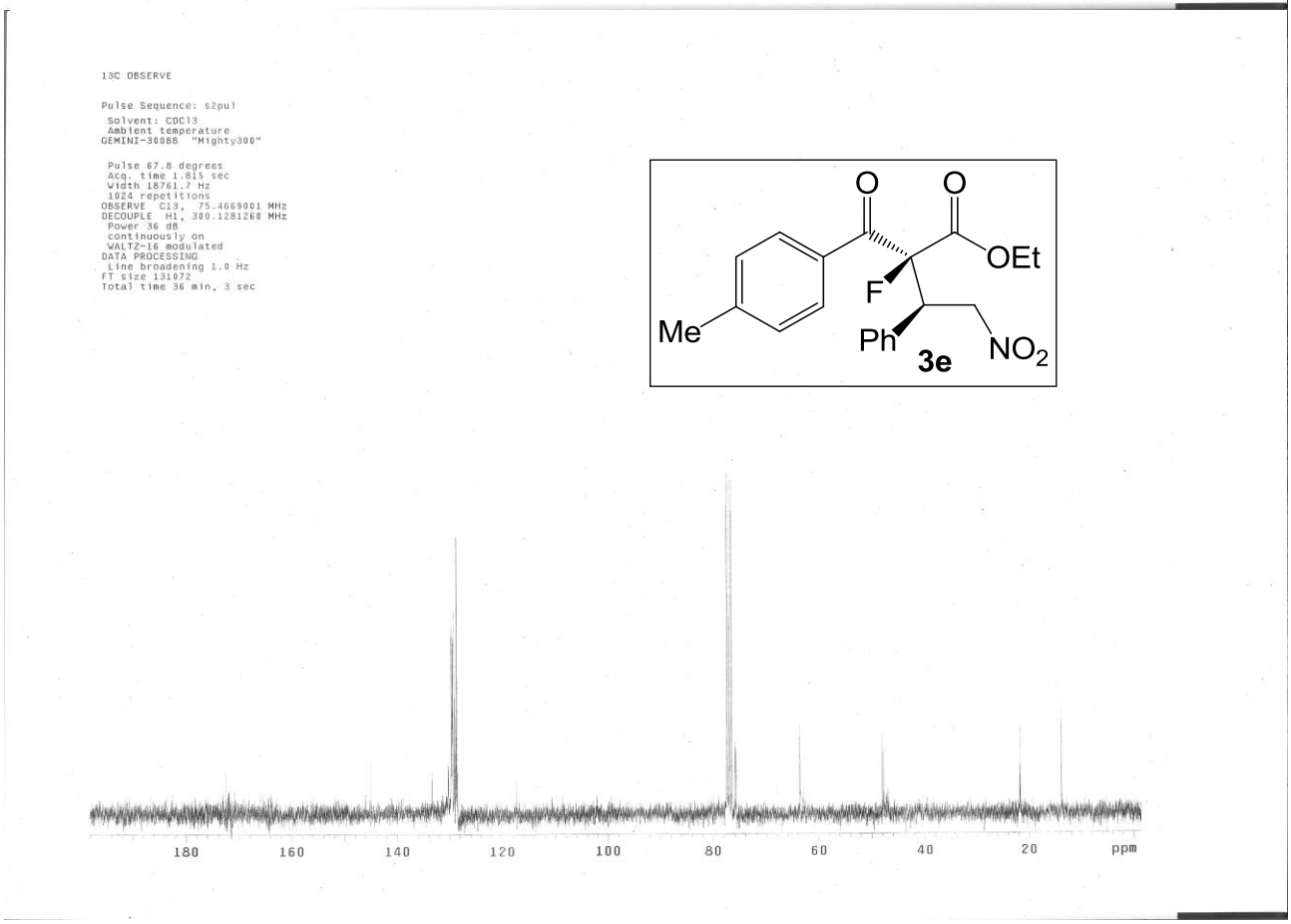


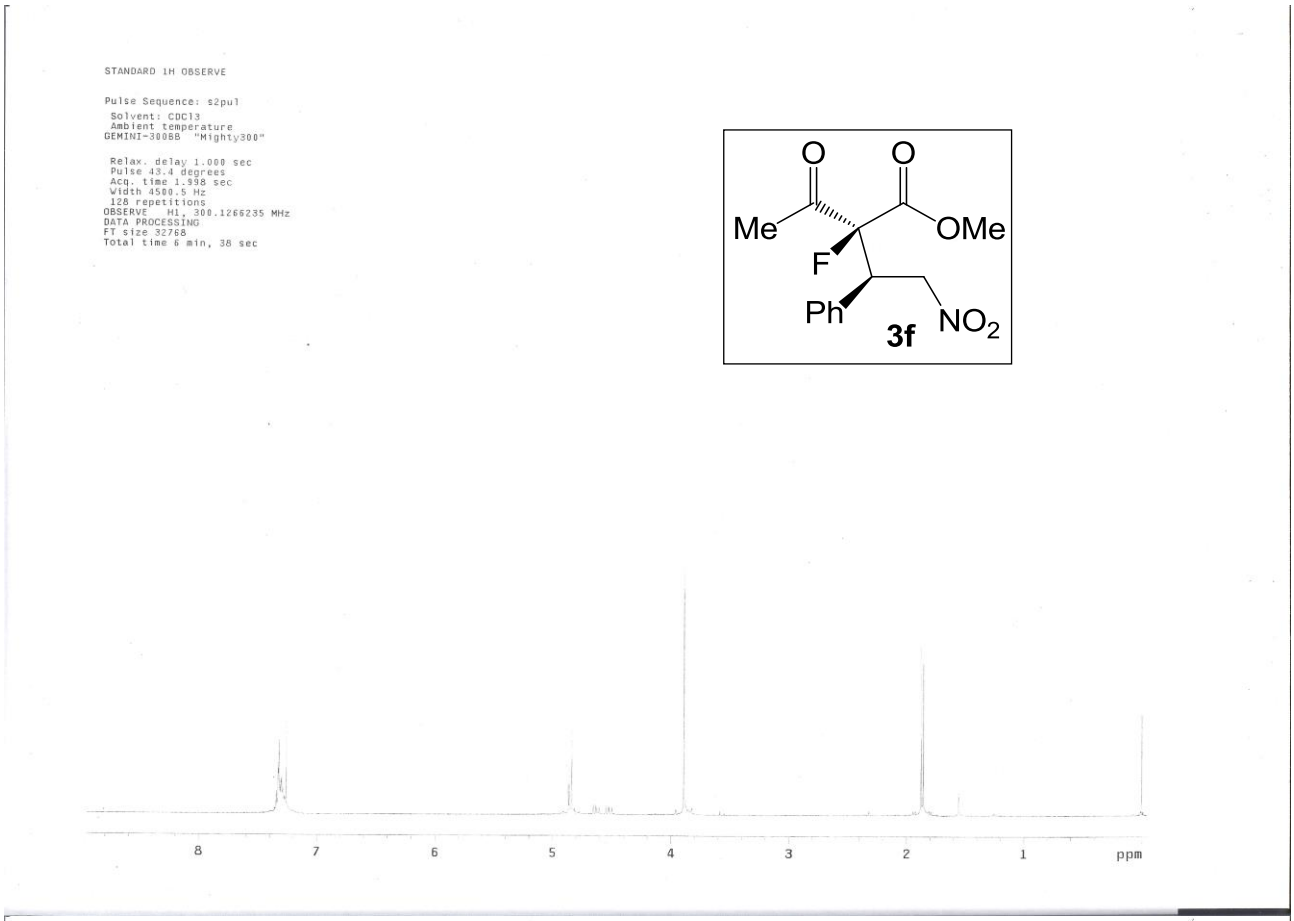


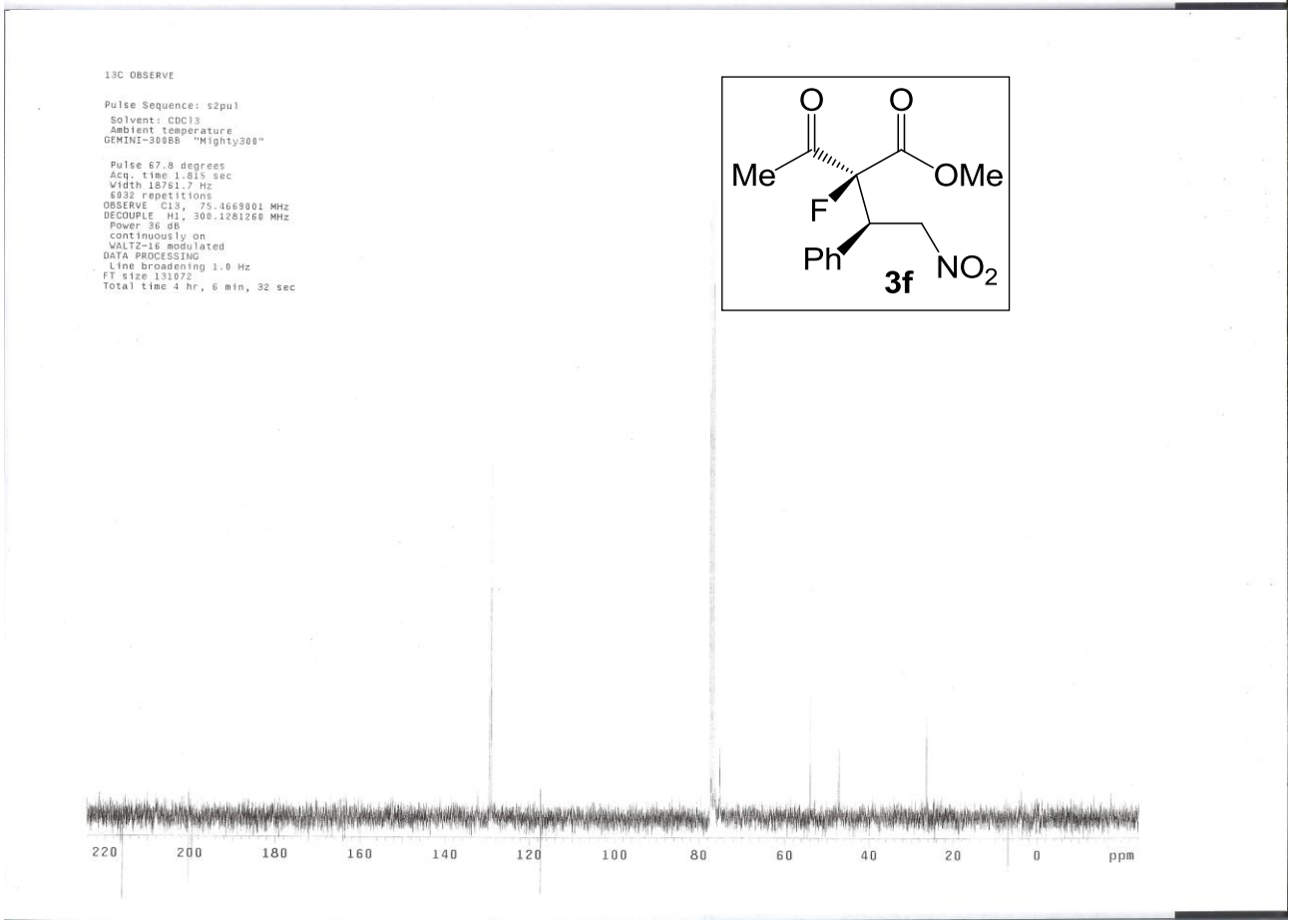


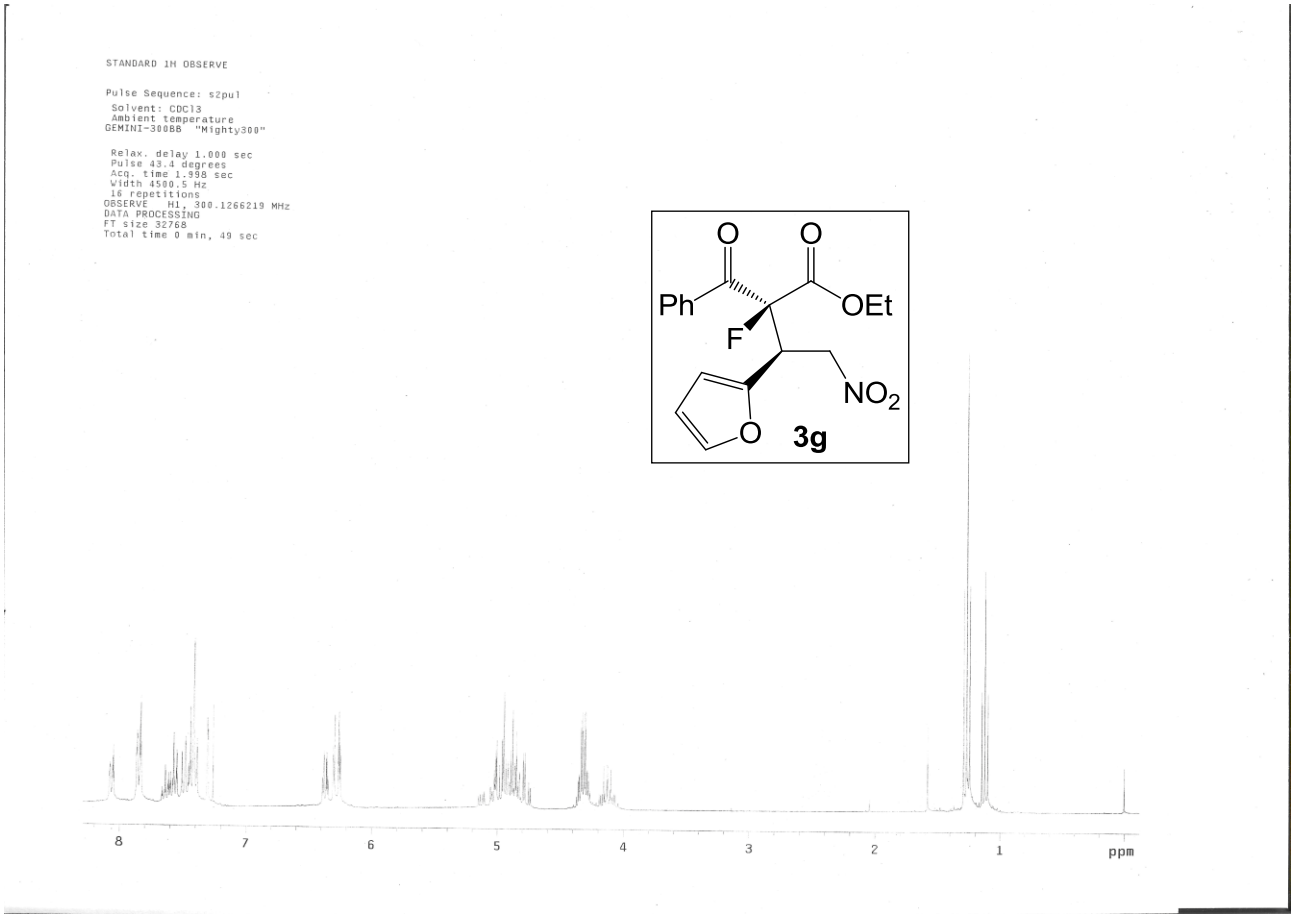


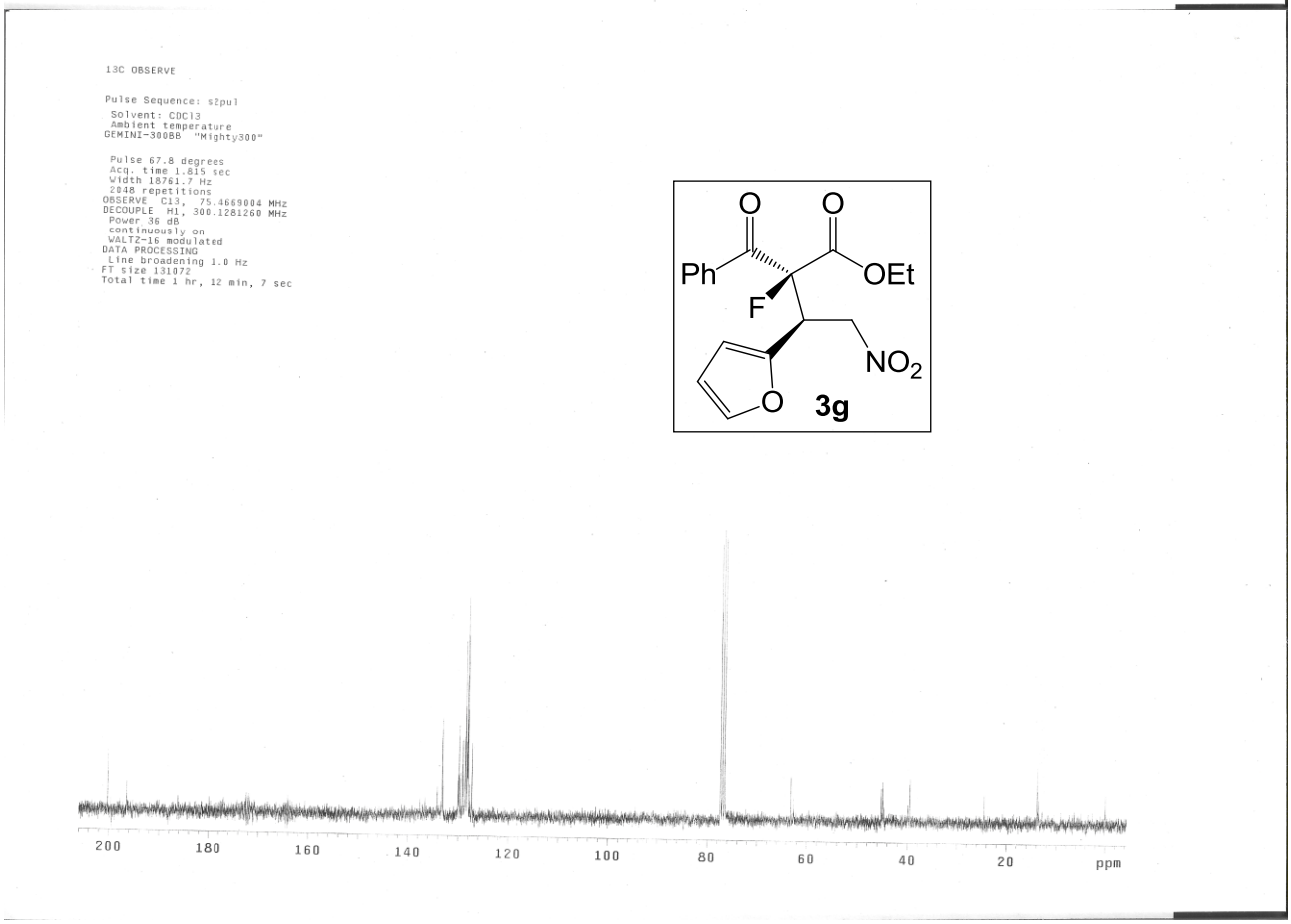


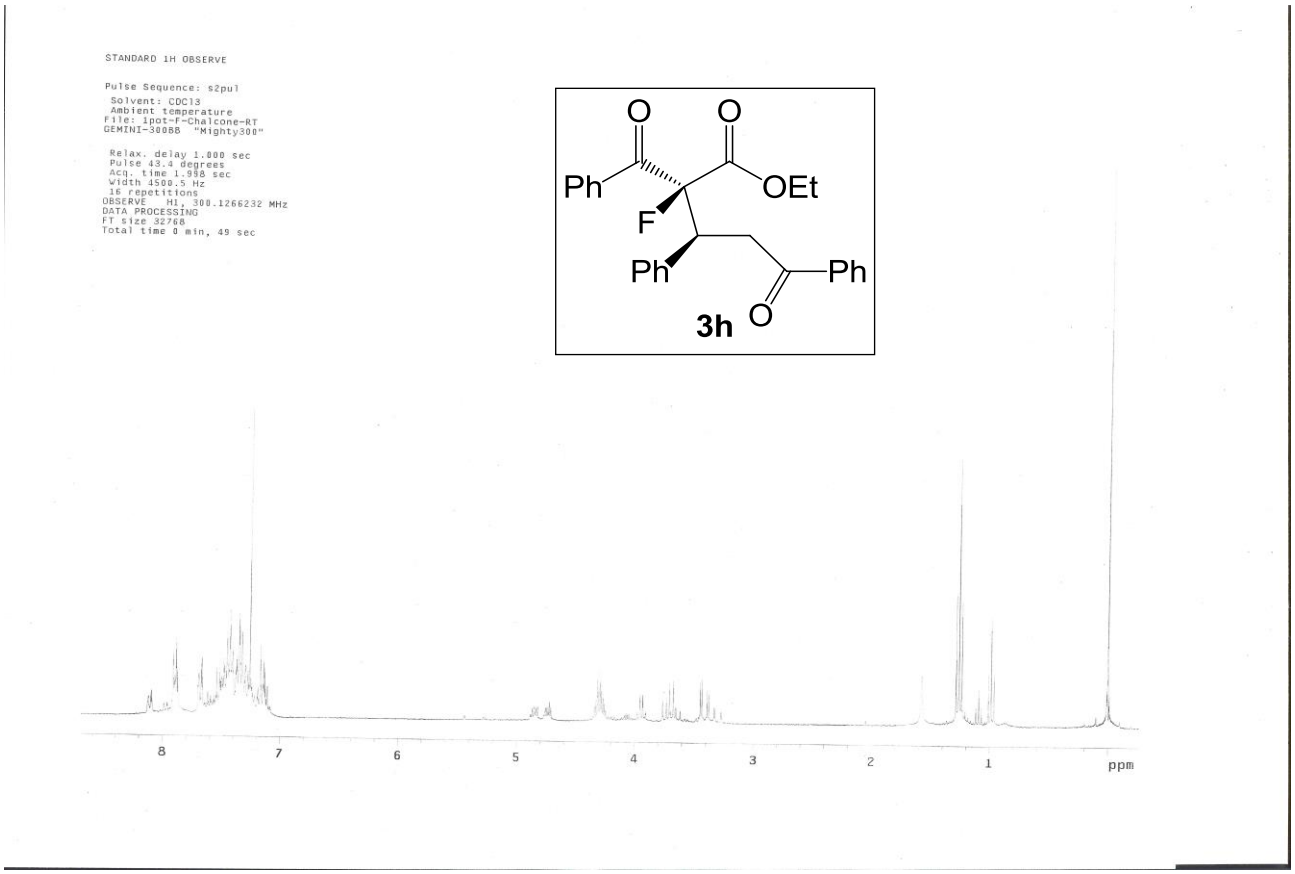


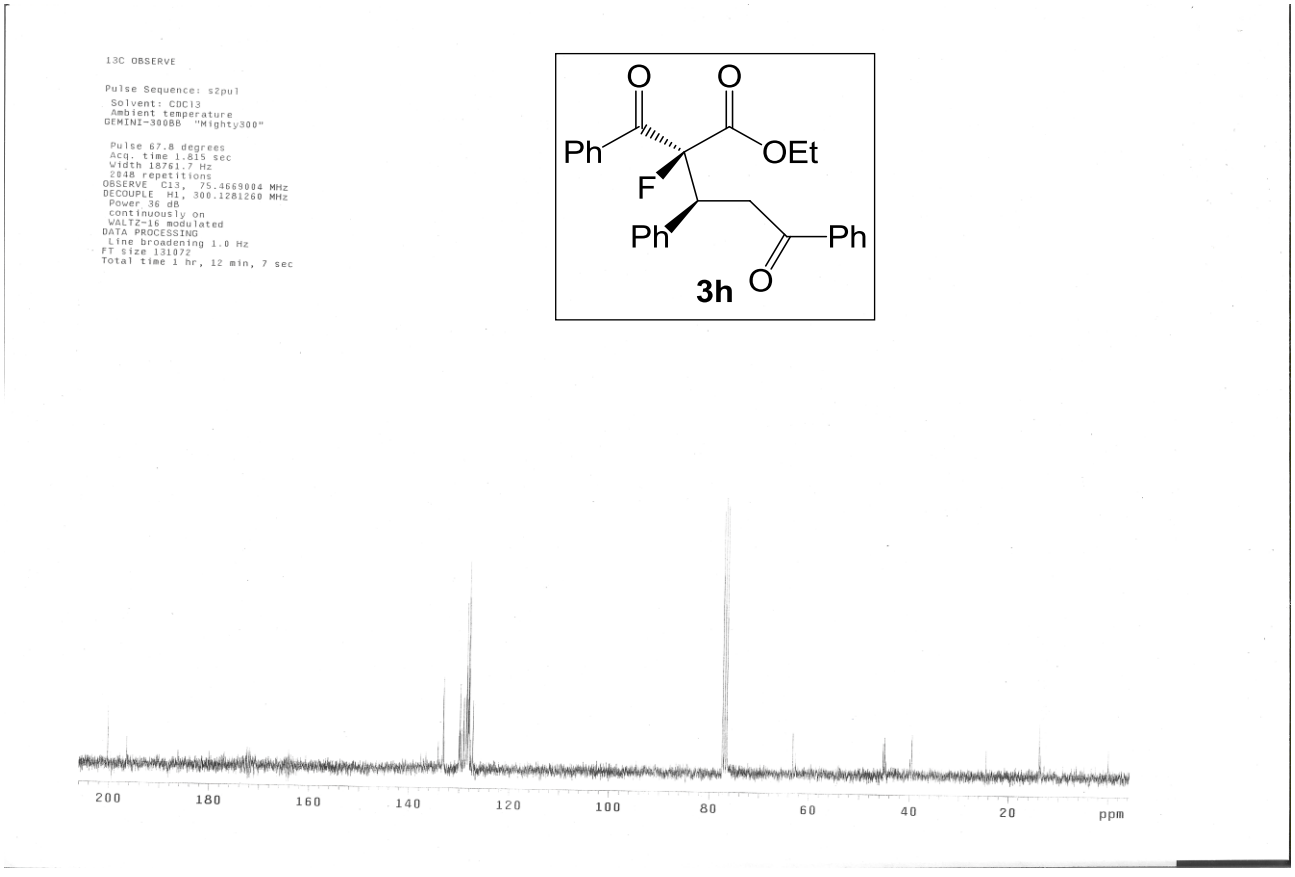


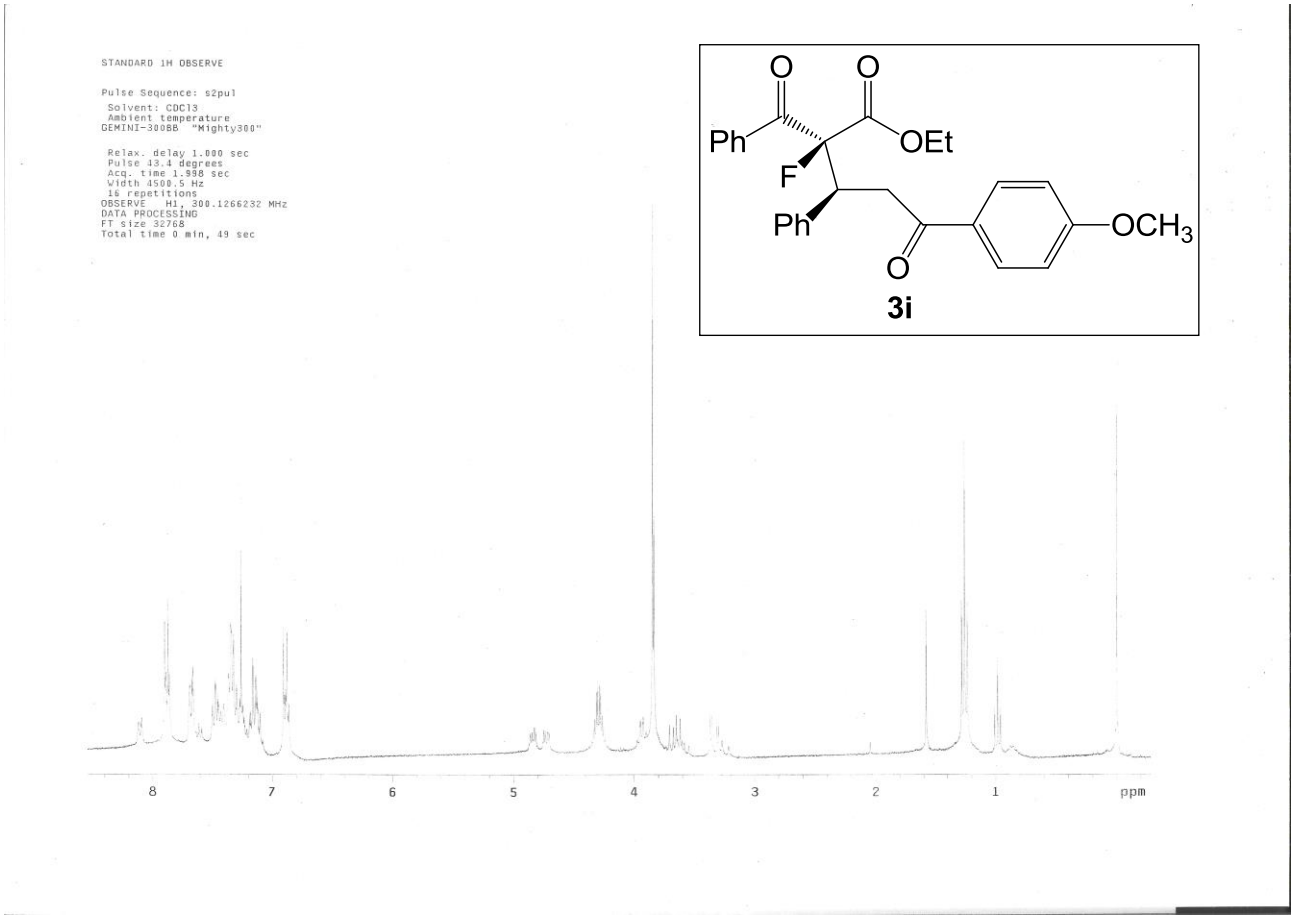


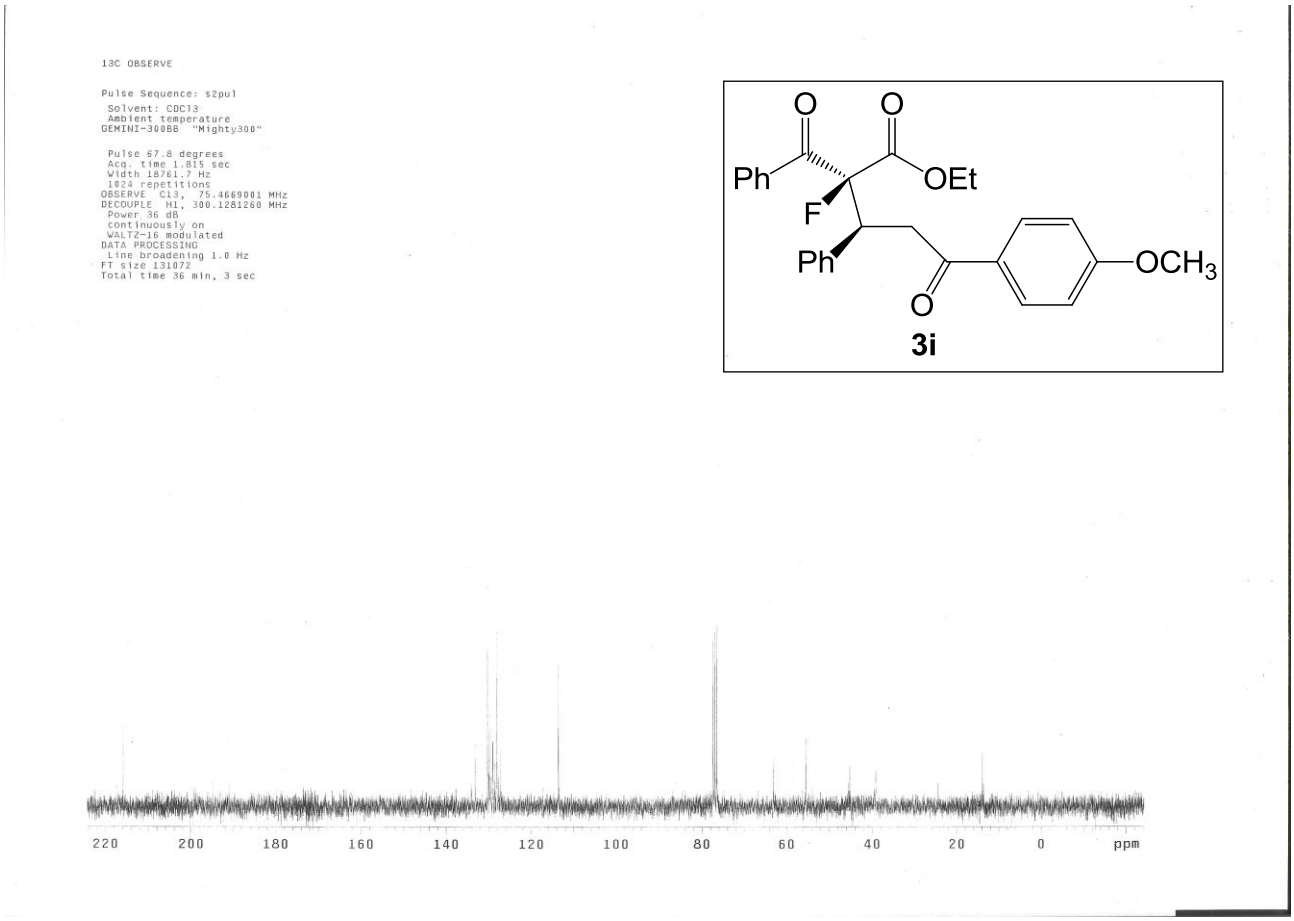


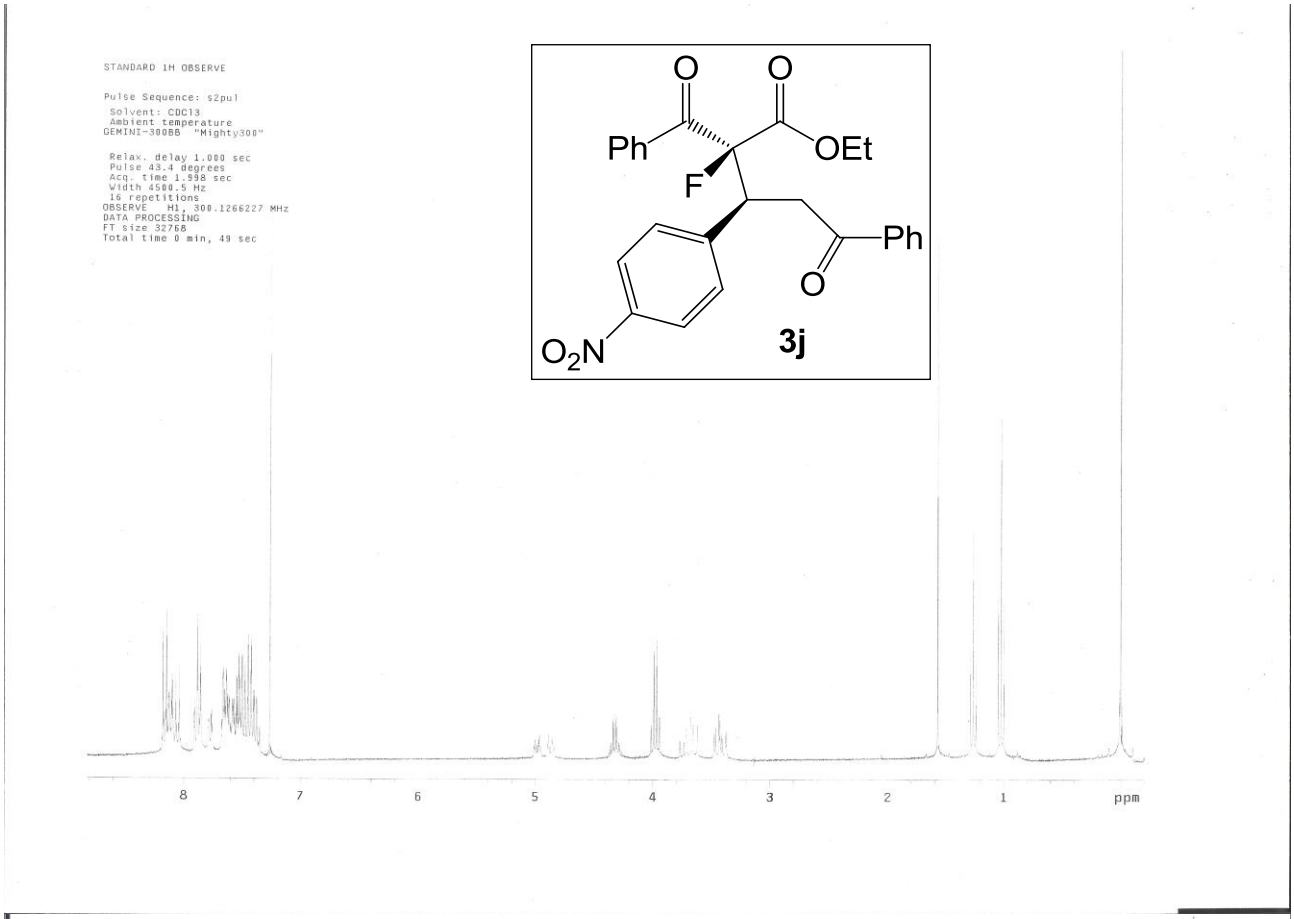


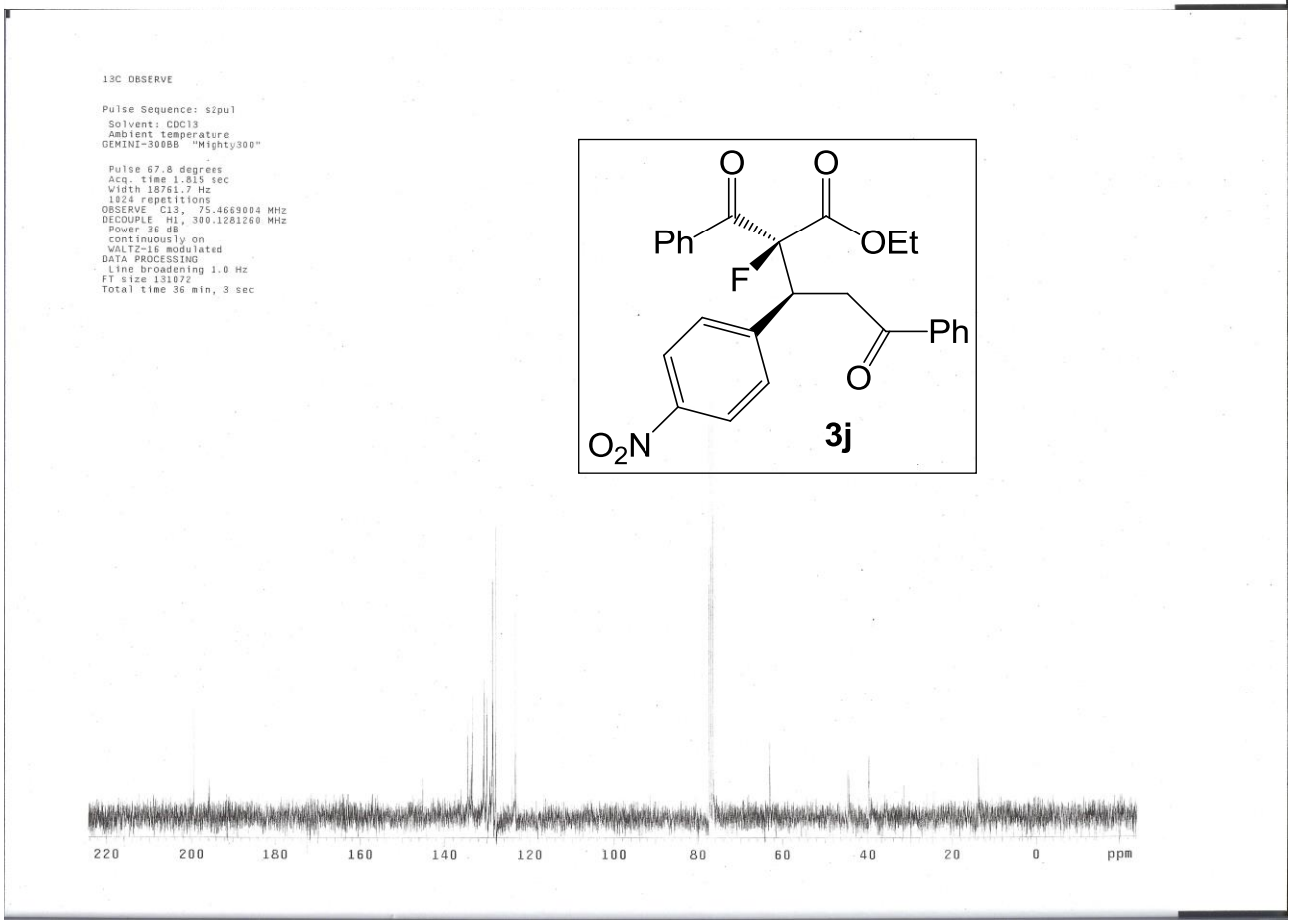


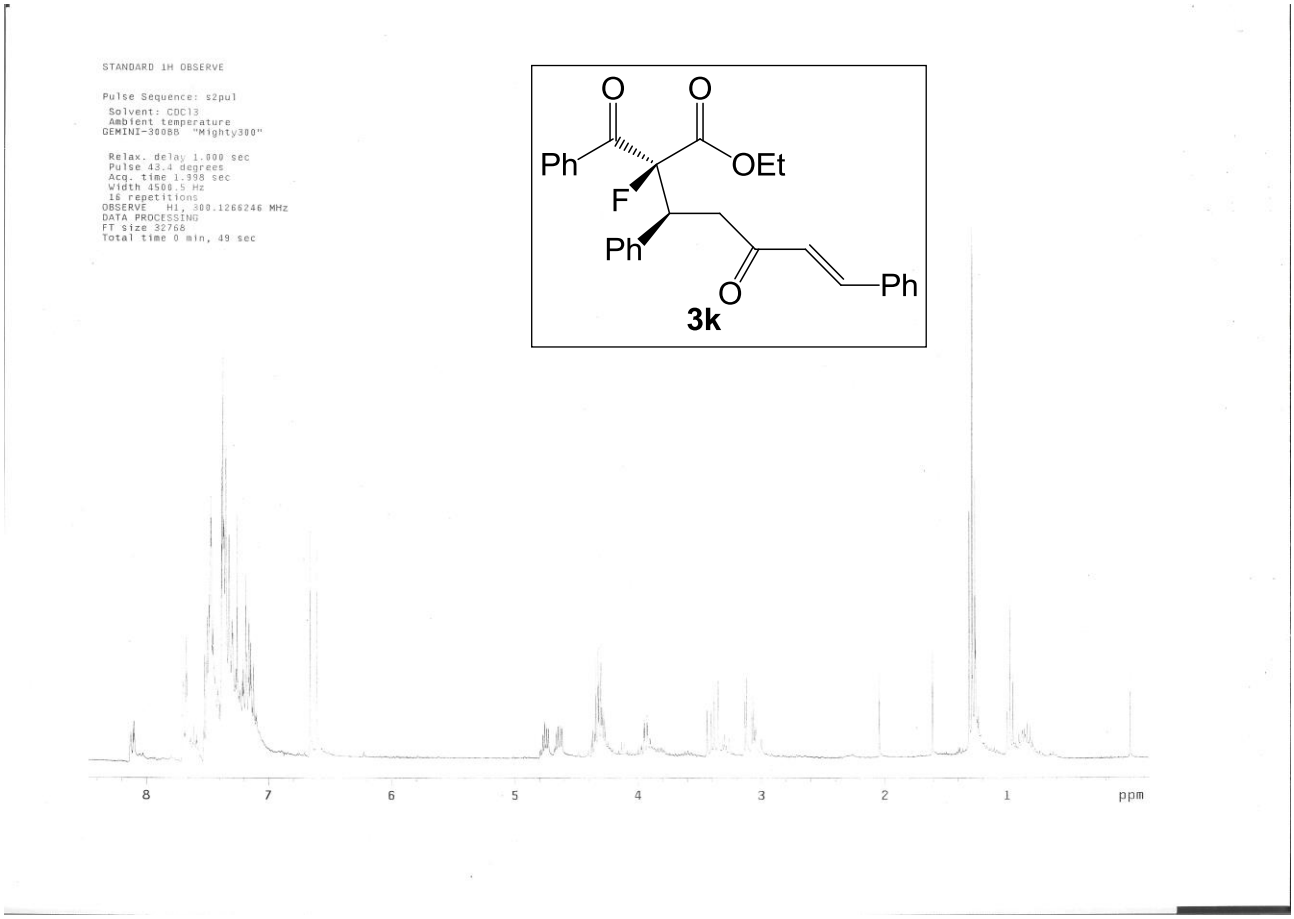


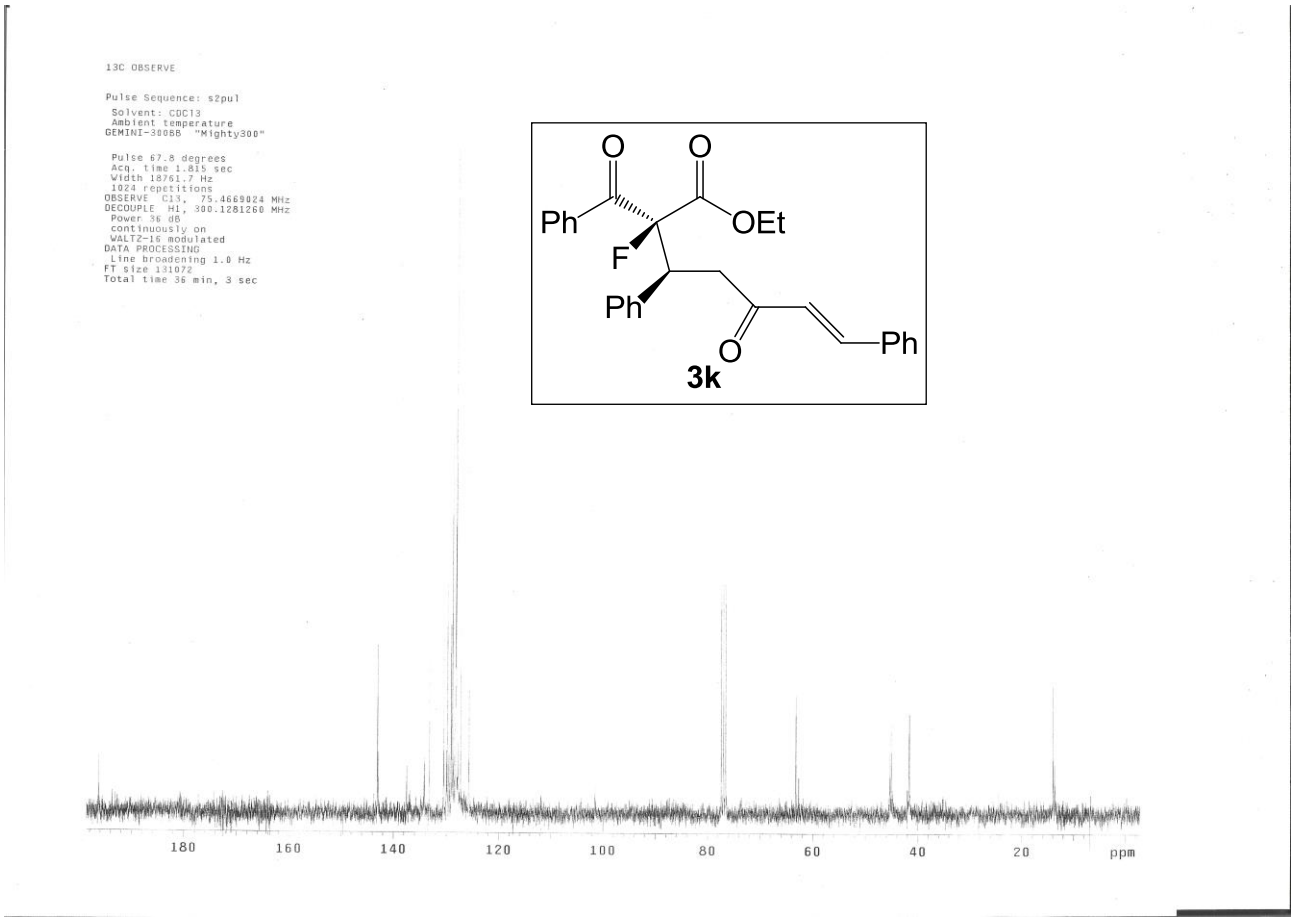


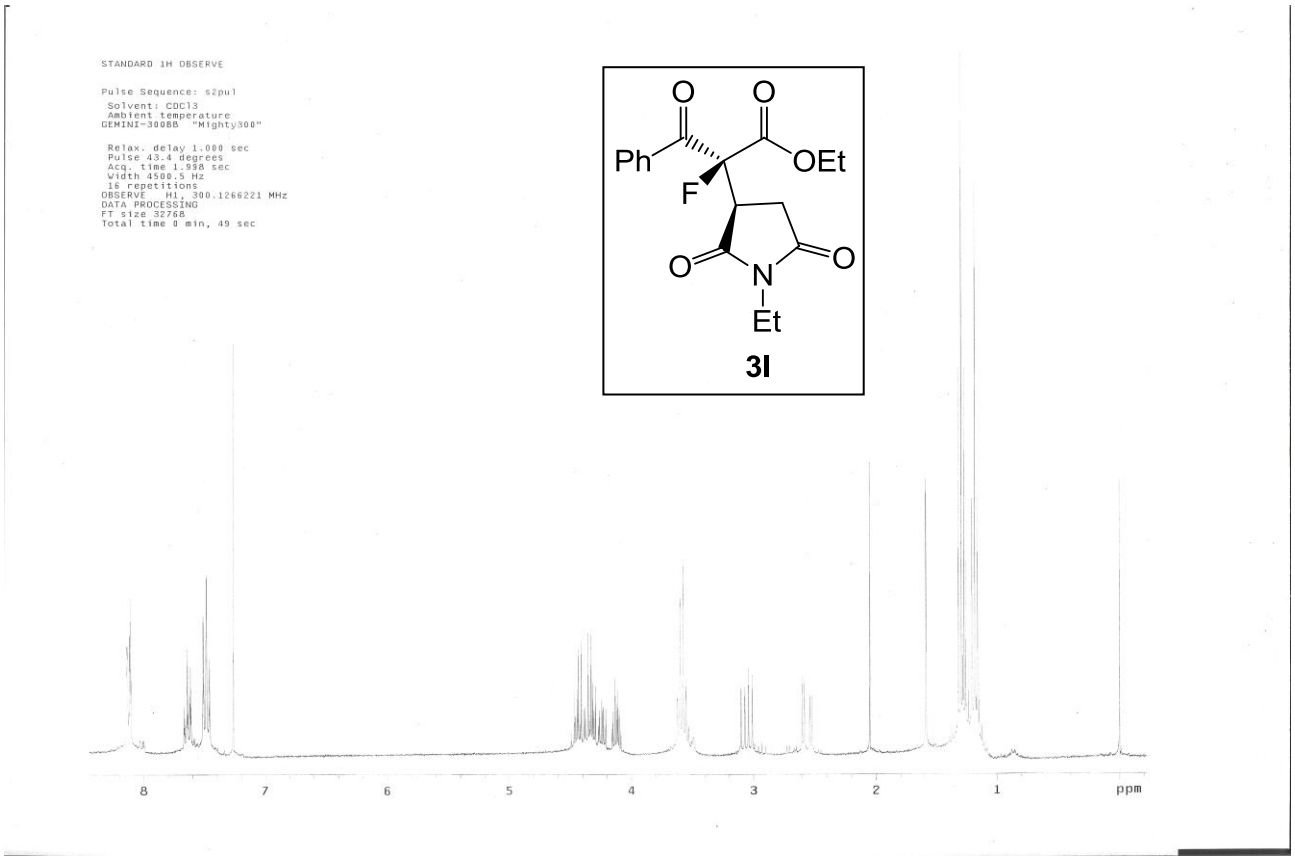


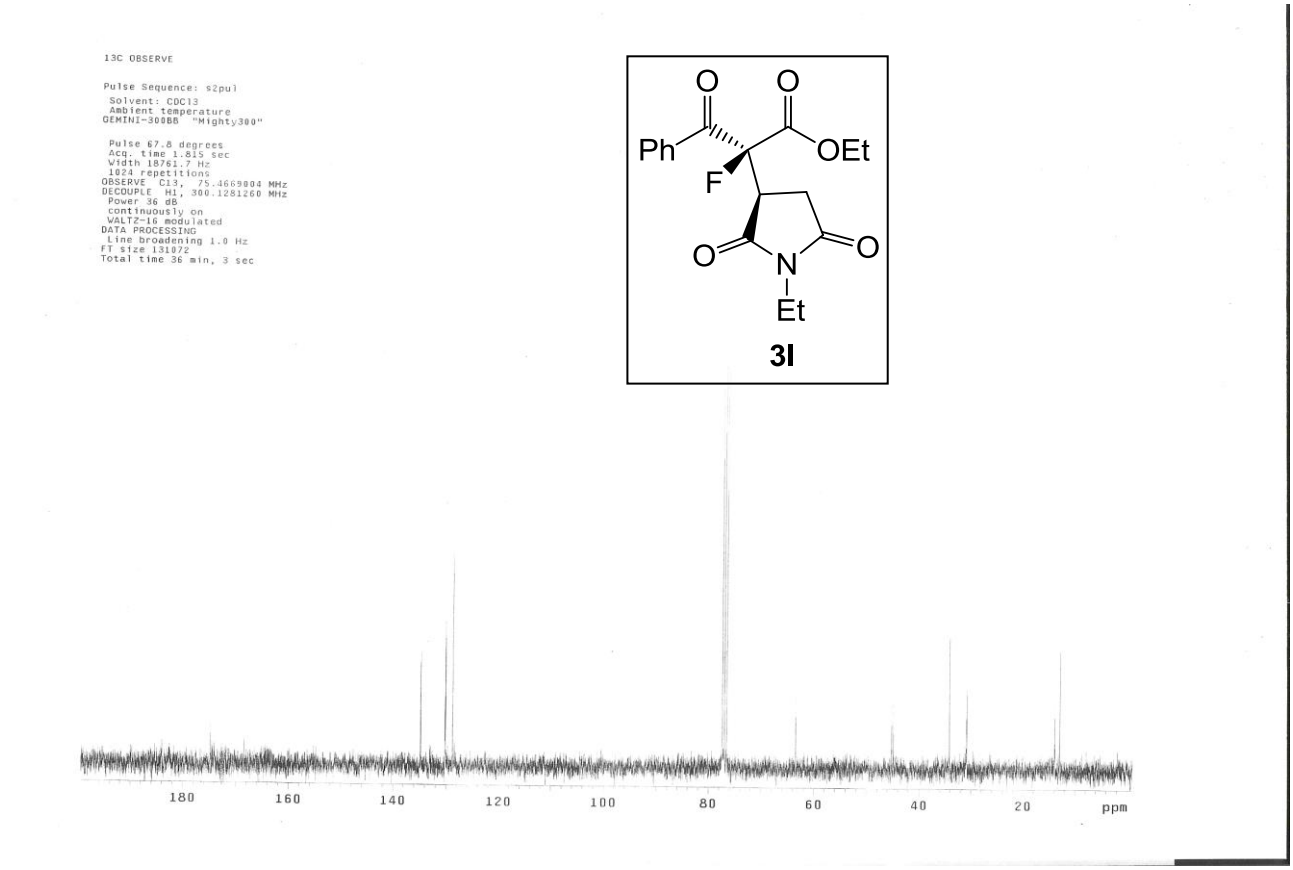


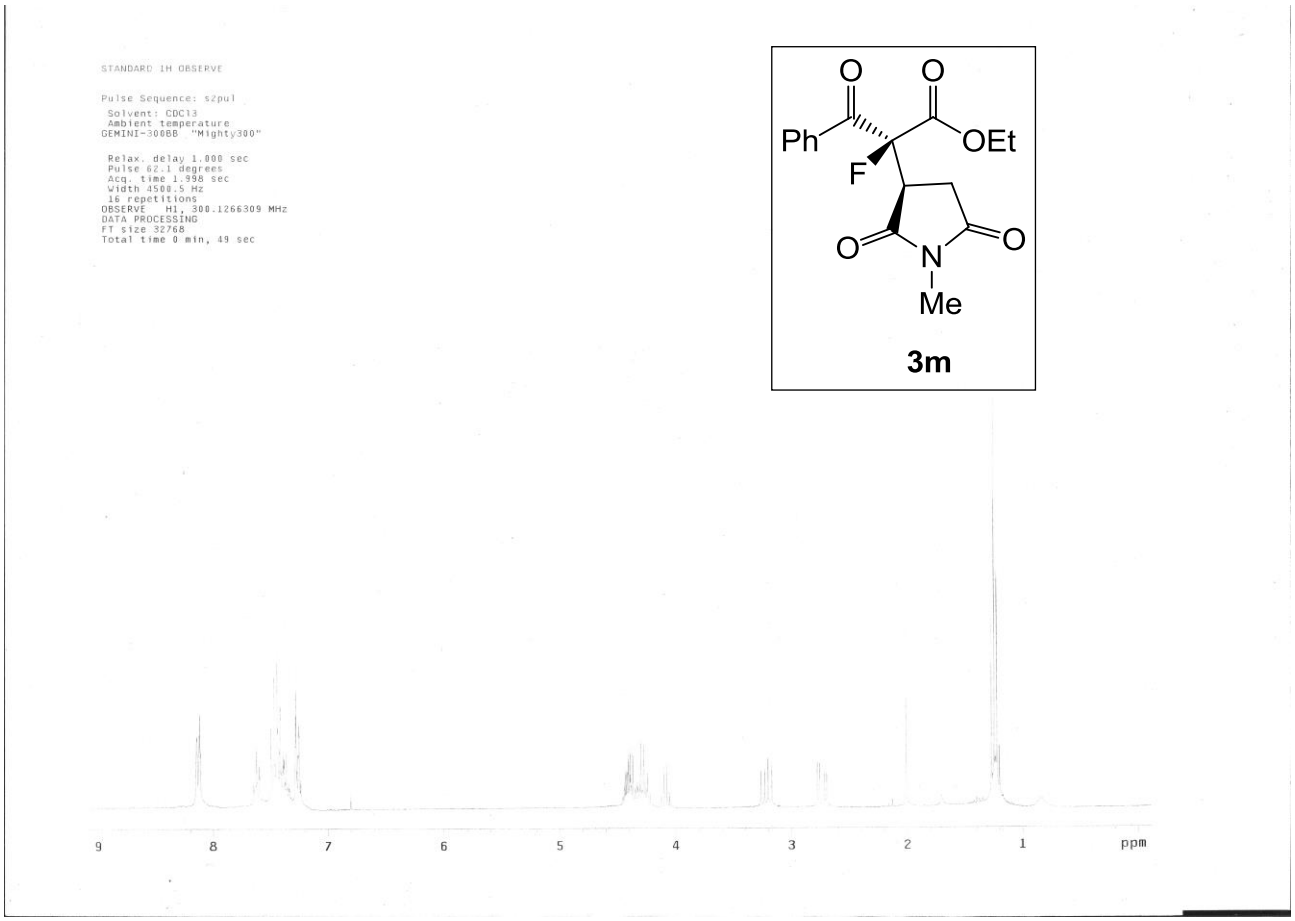


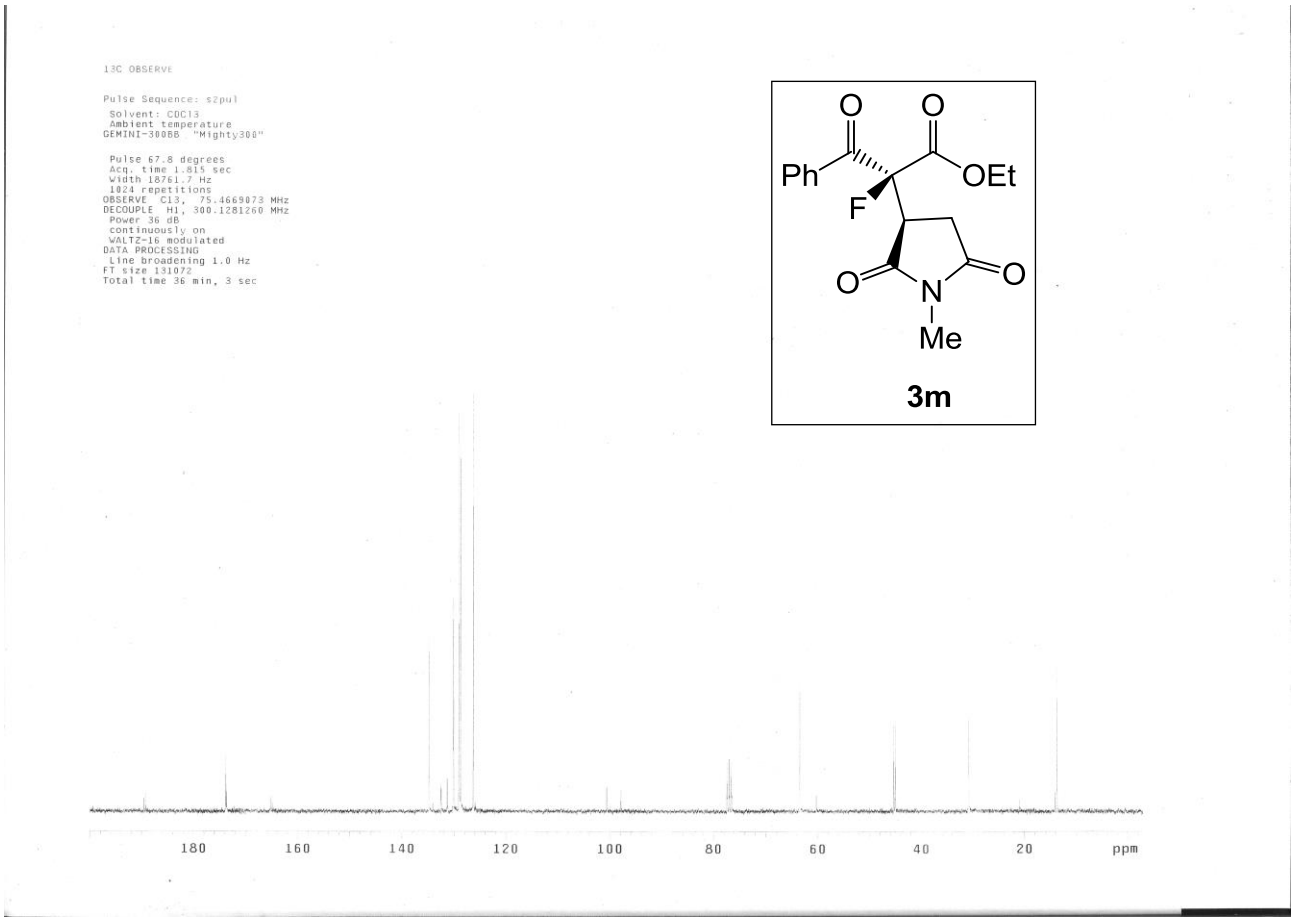


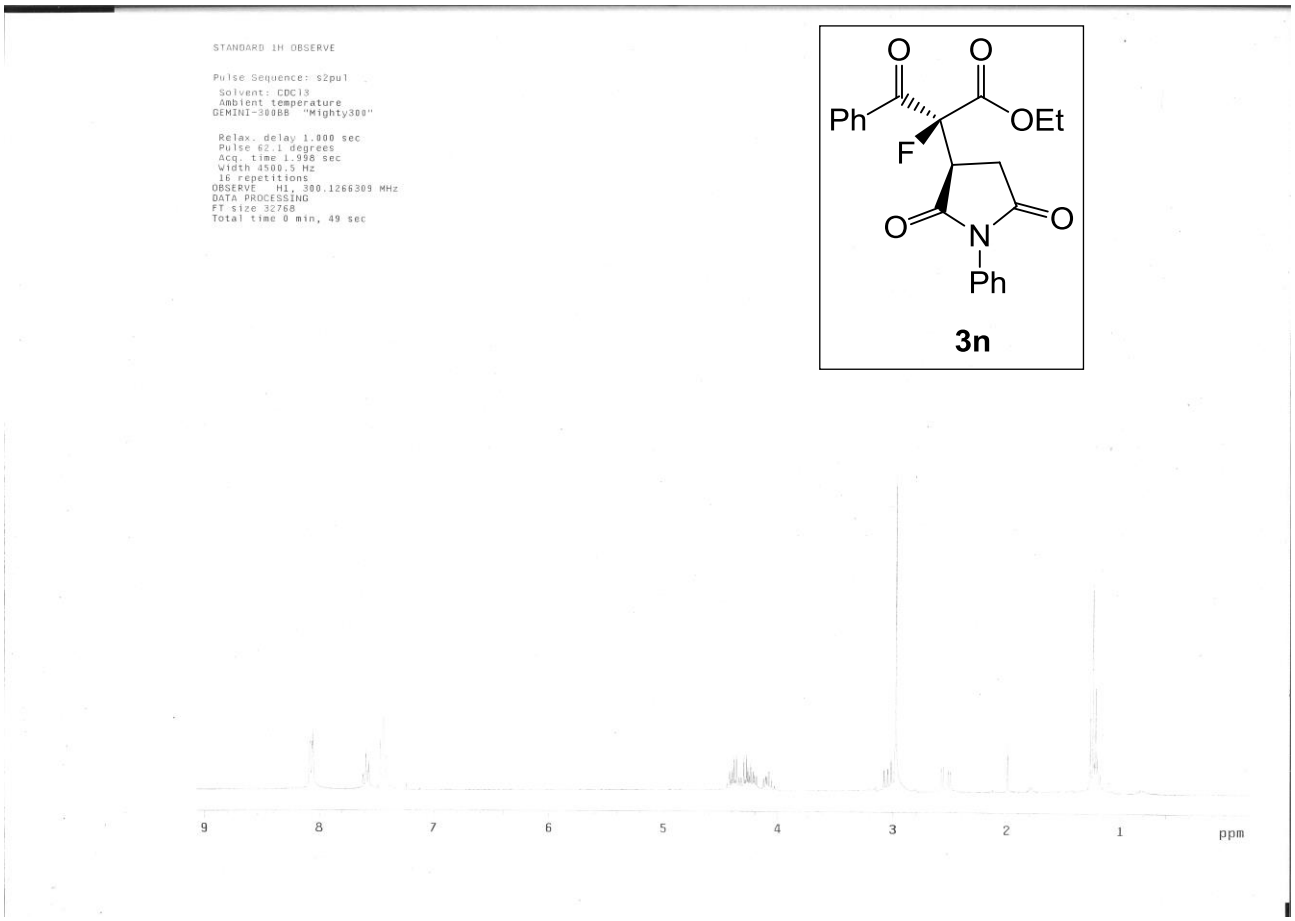


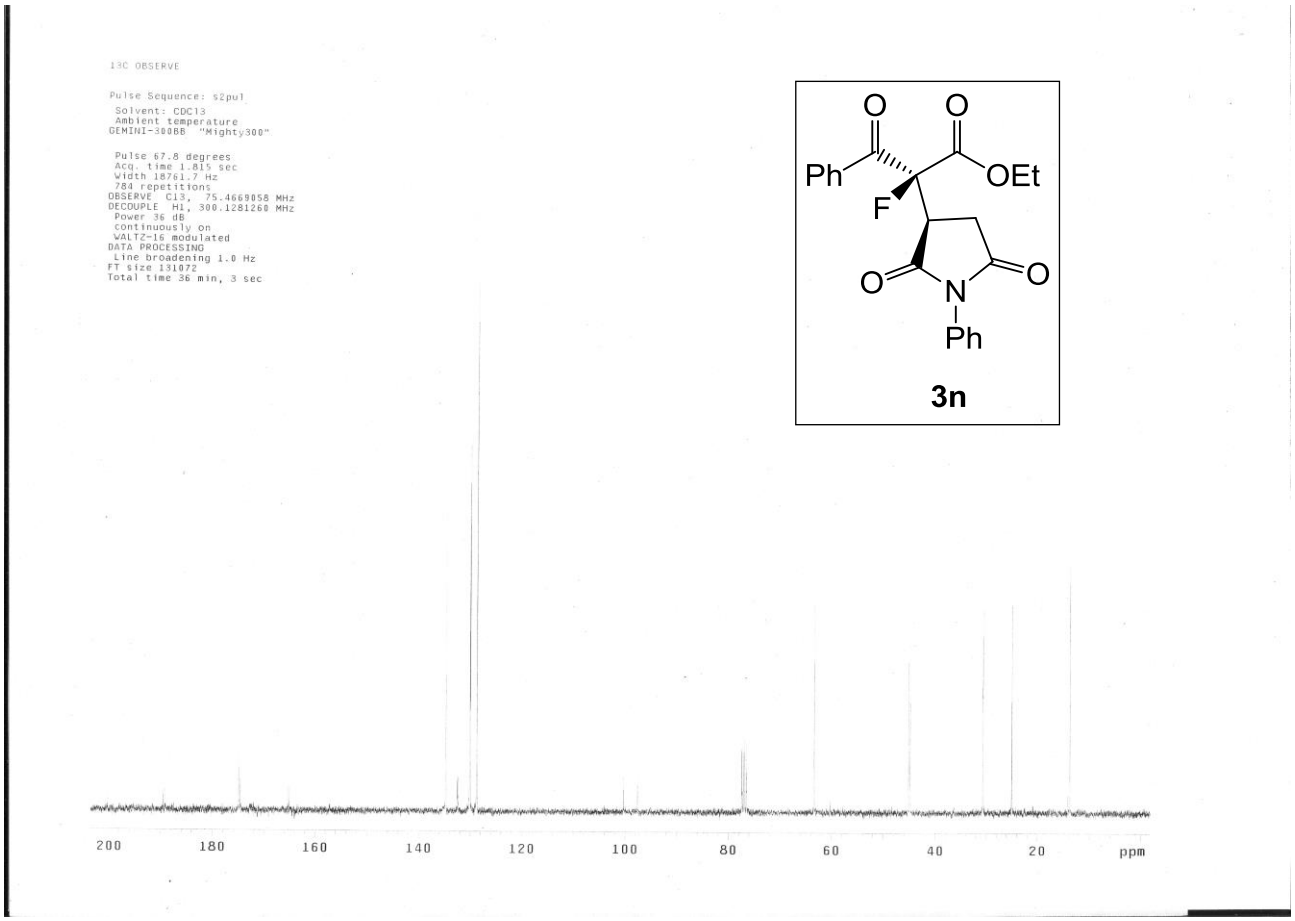

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "Mighty300"
Relax. delay 1.000 sec
Pulse 43.4 degrees
Acq. time 1.998 sec
Width 4500.5 Hz
16 repetitions
OBSERVE H1, 300.1266221 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 49 sec
Ph
O
O
OEt
F
O
N
O
Et
3l
8
7
6
5
4
3
2
1
ppm

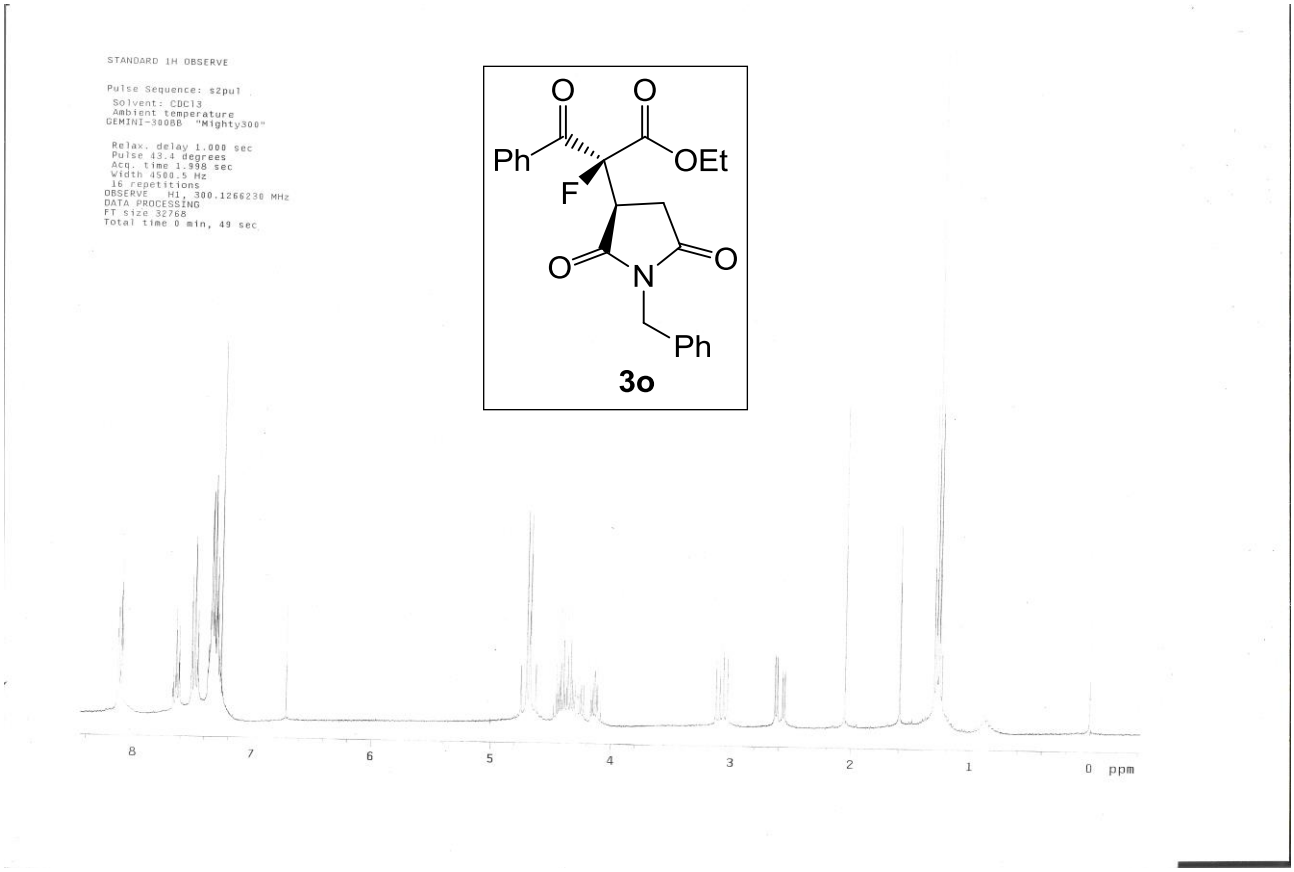

13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "Mighty300"
Pulse 67.8 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
1024 repetitions
OBSERVE C13, 75.4669804 MHz
DECOUPLE H1, 300.1281260 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 36 min, 3 sec
Ph
O
O
OEt
F
O
N
O
Et
3l
180
160
140
120
100
80
60
40
20
ppm

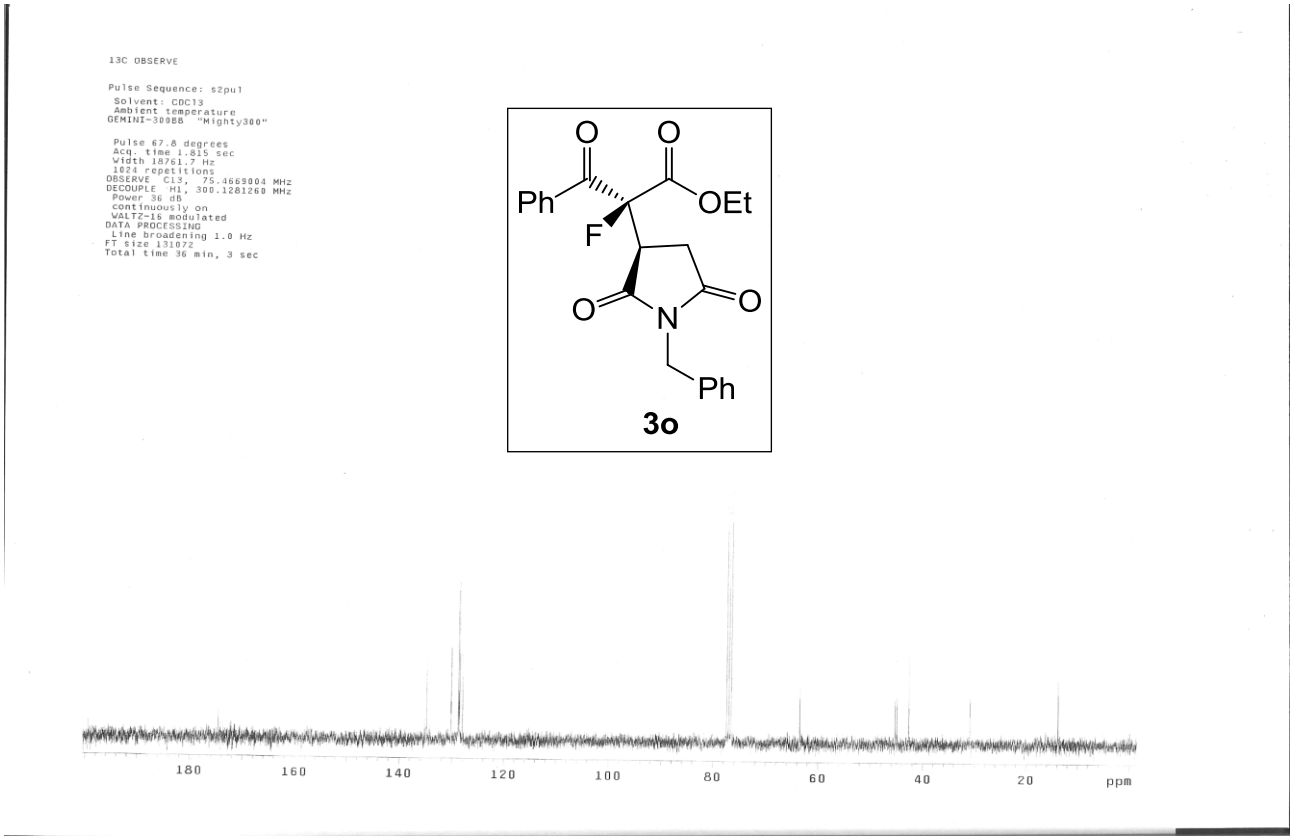


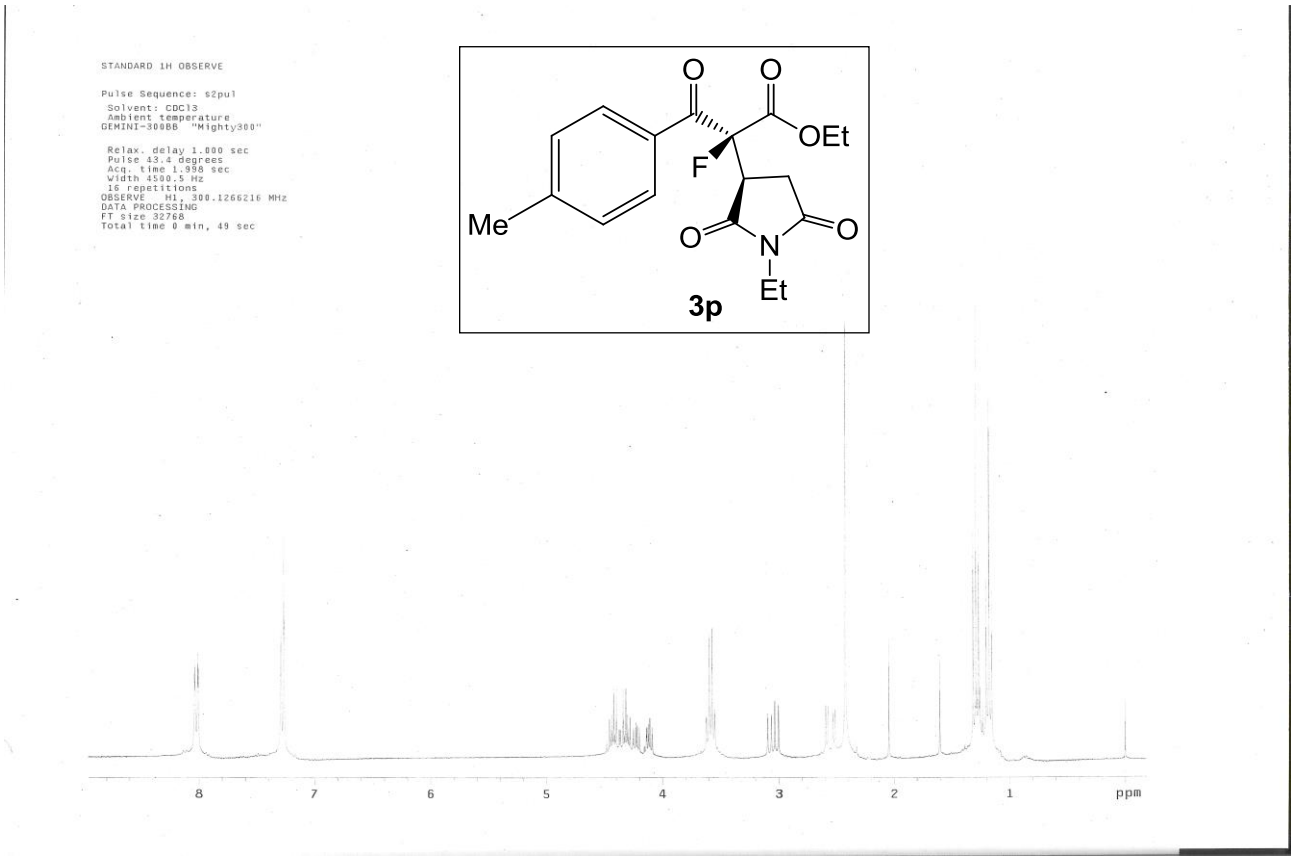


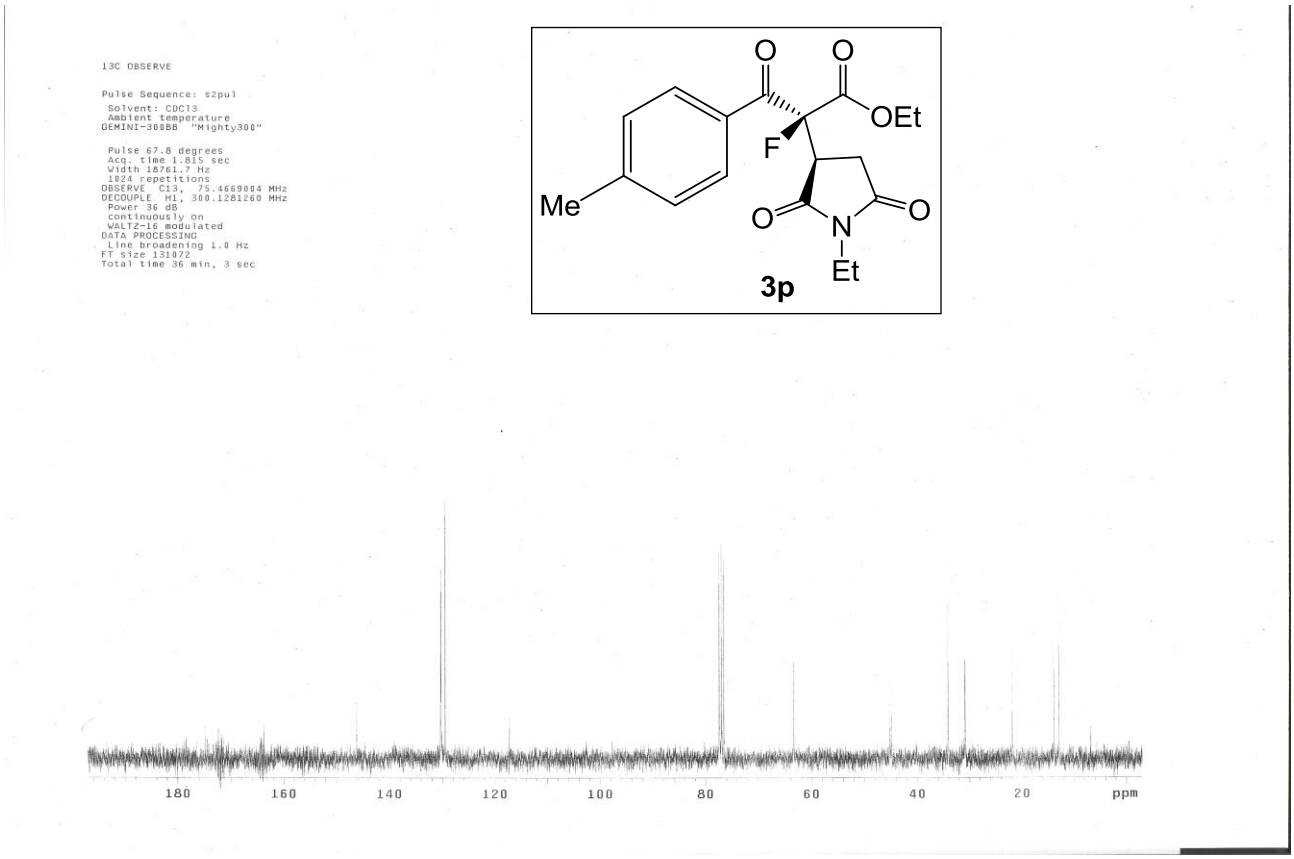


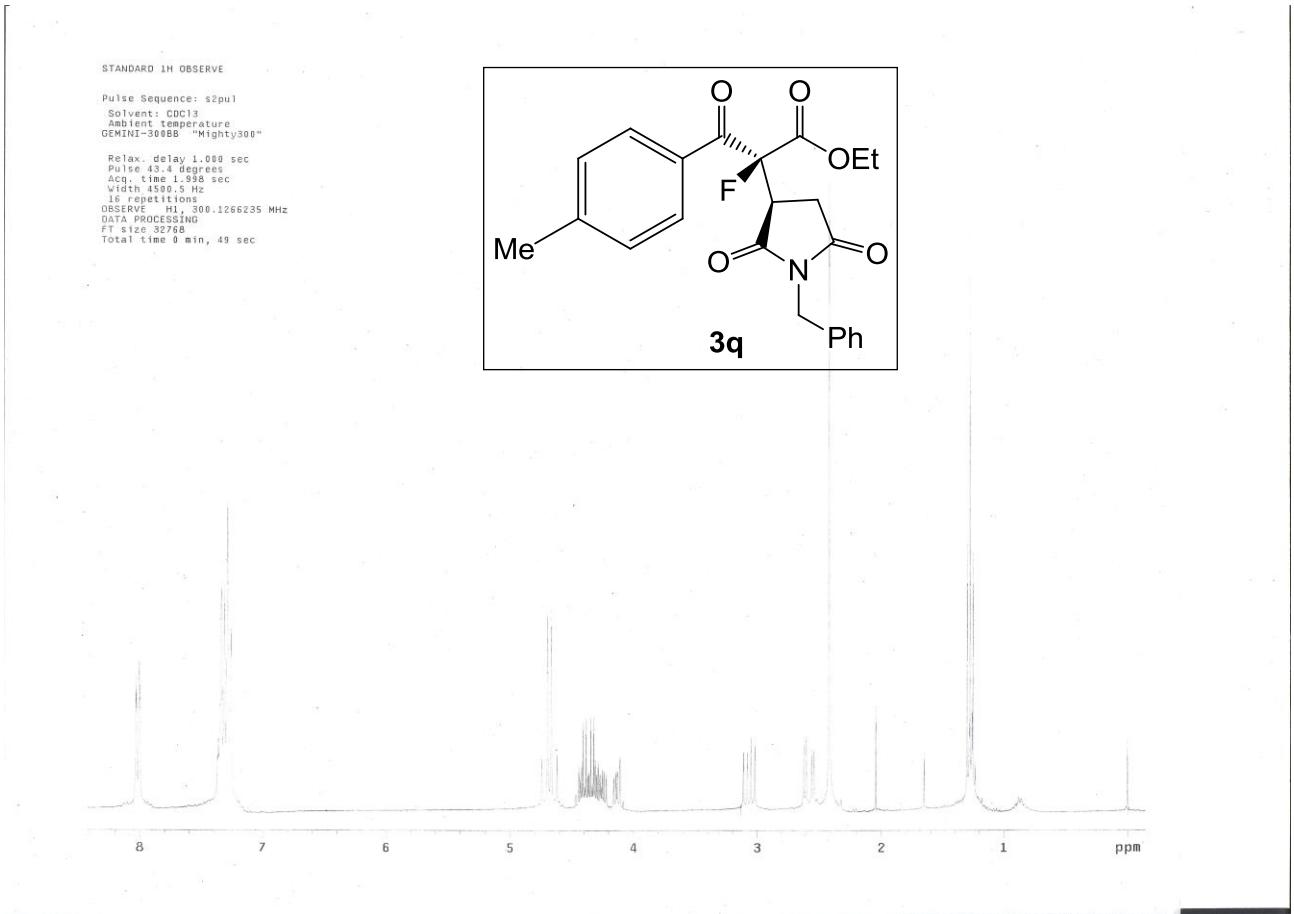


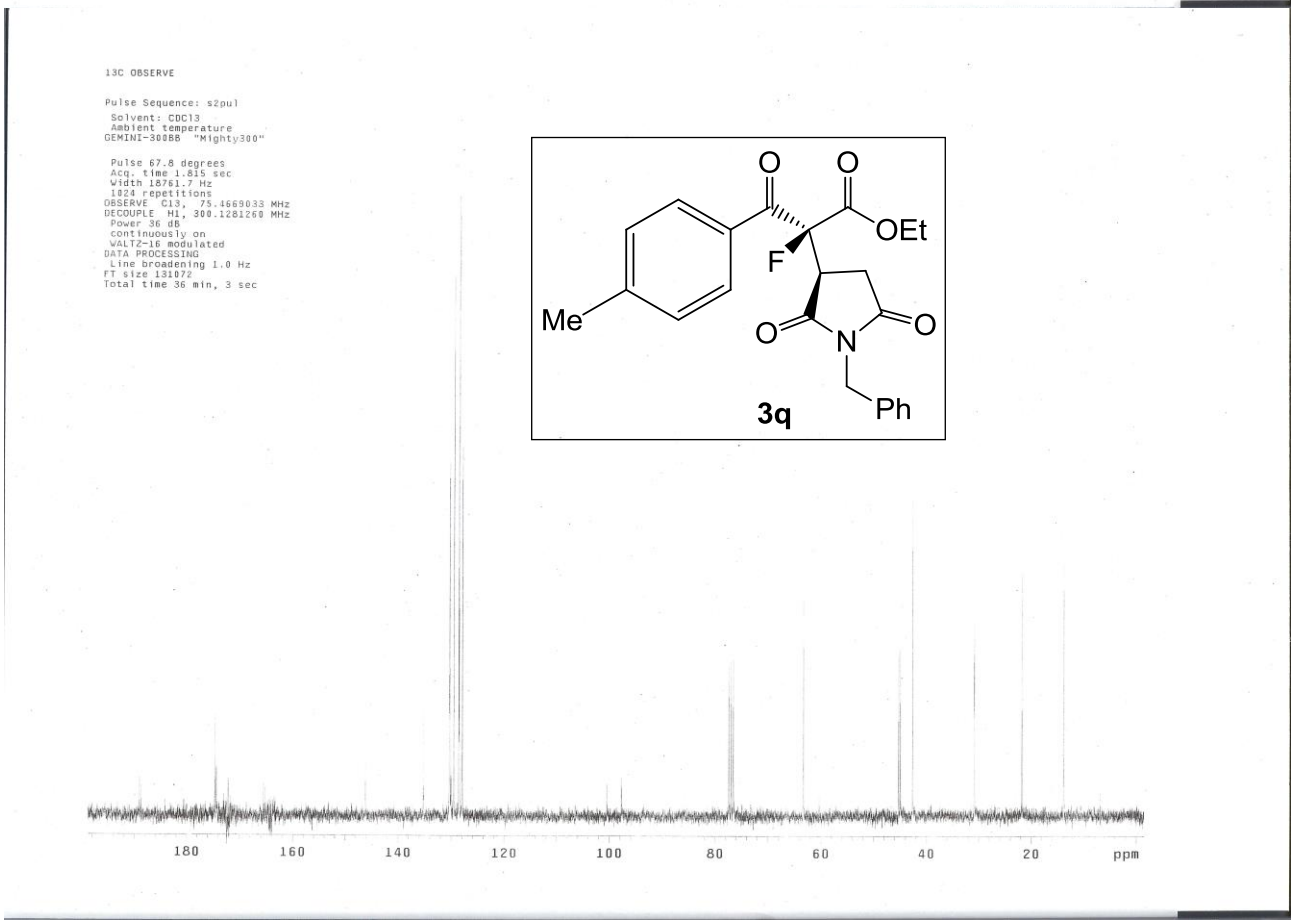

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "Mighty300"
Relax. delay 1.000 sec
Pulse 43.4 degrees
Acq. time 1.998 sec
Width 4500.5 Hz
16 repetitions
OBSERVE H1, 300.1266230 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 49 sec
Ph
O
O
OEt
F
O
N
O
Ph
3o
8
7
6
5
4
3
2
1
0 ppm


13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "Mighty300"
Pulse 67.8 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
1024 repetitions
OBSERVE C13, 75.4669004 MHz
DECOUPLE H1, 300.1281260 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 36 min, 3 sec
Ph
O
O
OEt
F
O
N
O
Ph
3o
180
160
140
120
100
80
60
40
20
ppm

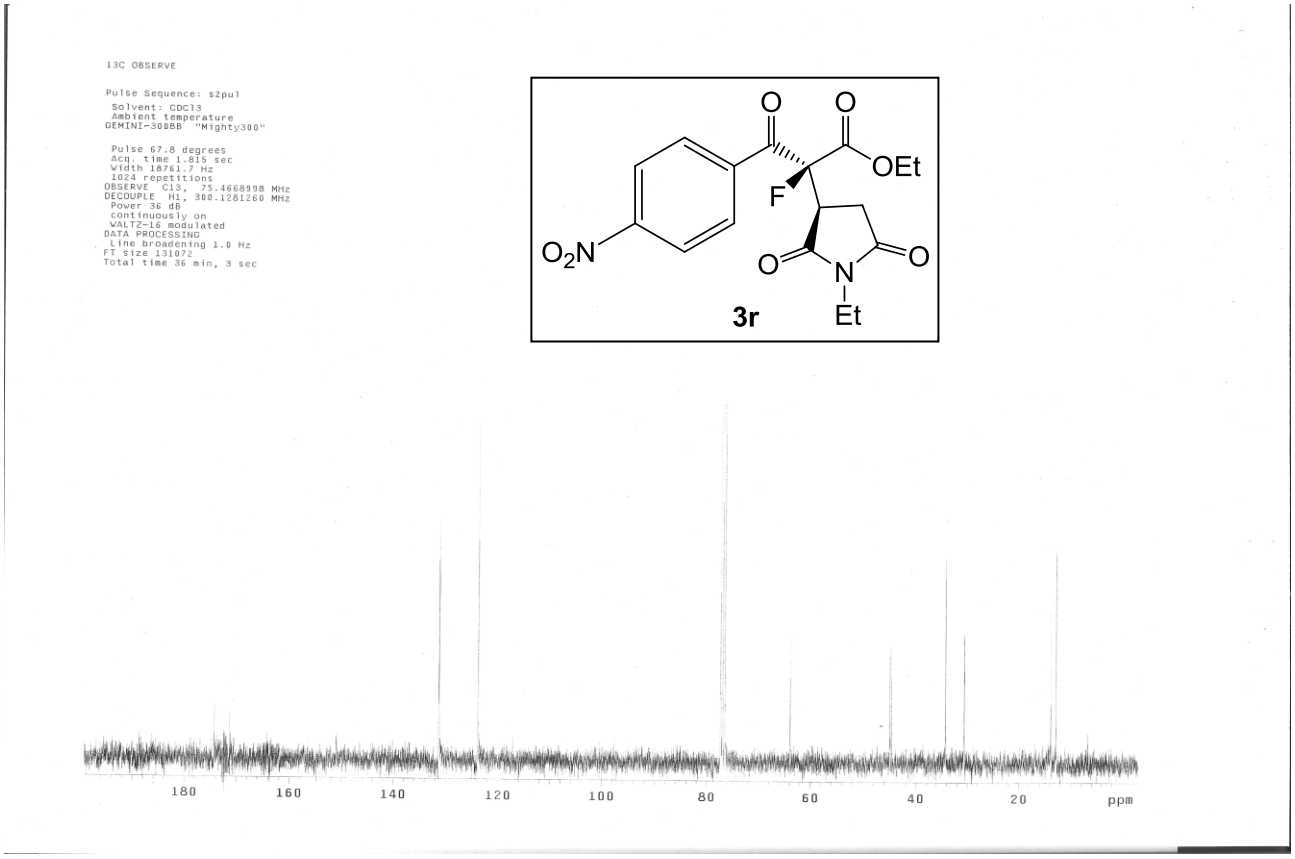


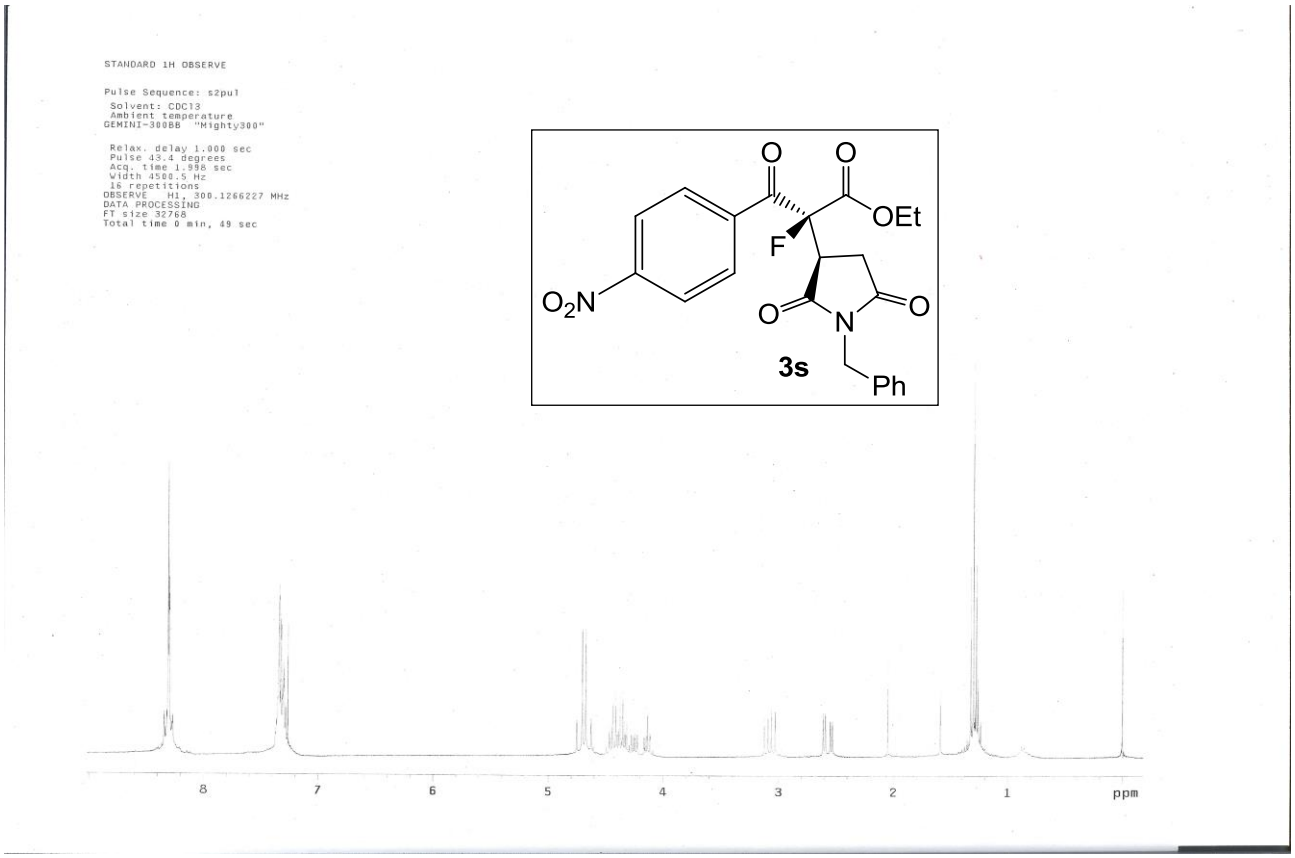


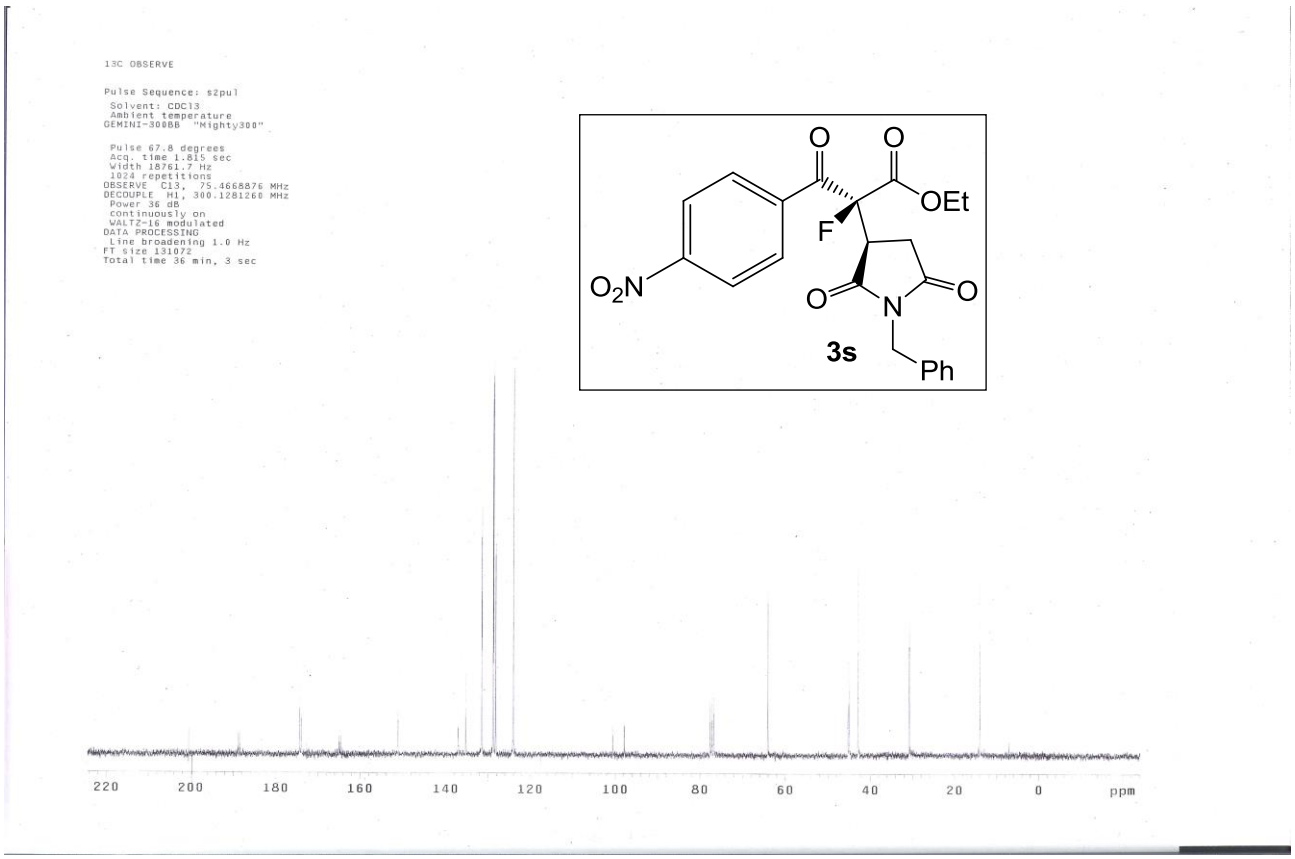





STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "Mighty300"
Relax. delay 1.000 sec
Pulse 43.4 degrees
Acq. time 1.998 sec
Width 4500.5 Hz
16 repetitions
OBSERVE H1, 300.1266208 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 49 sec
O
O
OEt
F
O2N
O
N
O
Et
3r
8
7
6
5
4
3
2
1
ppm


13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "Mighty300"
Pulse 67.8 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
1024 repetitions
OBSERVE C13, 75.4668998 MHz
DECOUPLE H1, 300.1281260 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 36 min, 3 sec
O
O
OEt
F
O2N
O
N
O
Et
3r
180
160
140
120
100
80
60
40
20
ppm


STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "Nighty300"
Relax. delay 1.000 sec
Pulse 45.4 degrees
Acq. time 1.956 sec
Width 4508.5 Hz
16 repetitions
OBSERVE H1, 300.1266227 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 49 sec
O
O
OEt
F
O2N
O
N
O
3s
Ph
8
7
6
5
4
3
2
1
ppm


13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "Nighty300"
Pulse 67.8 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
1024 repetitions
OBSERVE C13, 75.4668876 MHz
DECOUPLE H1, 300.1281260 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 36 min, 3 sec
O
O
OEt
F
O2N
O
N
O
3s
Ph
220
200
180
160
140
120
100
80
60
40
20
0
ppm