Supporting Information for

Oxidative Cross-Esterification of Dithiolanes with Alcohols through a Cross-Dehydrogenative Coupling (CDC) / Deprotection Sequence

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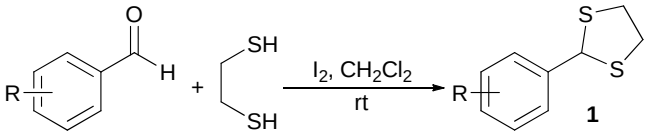
1. General Information

Unless otherwise noted, materials were purchased from commercial suppliers and used without further purification. All the solvents and alcohols were treated according to general methods, (*S*)-tert-butyl 2-(hydroxymethyl) pyrrolidine-1-carboxylate was synthesized and CuI was activated according to the known methods. Flash column chromatography was performed using 200-300 mesh silica gel or 200-300 mesh neutral Al_2O_3. ^1H NMR spectra were recorded on Varian Mercury 400/600 (400/600 MHz) spectrophotometers. Chemical shifts (δ) are reported in ppm from the solvent resonance as the internal standard ($CDCl_3$: 7.26 ppm). Datas are reported as follows: chemical shift, multiplicity (s = single, d = doublet, t = triplet, dd = doublet of doublets, m = multiplet), coupling constants (Hz) and integration. ^{13}C NMR spectra were recorded on Varian Mercury 400/600 (100/150MHz) with complete proton decoupling spectrophotometers ($CDCl_3$: 77.0 ppm). Mass spectra were measured on a Finnigan Trace MS spectrometer (EI) or a Bruker Daltonics Inc. ApexII FT-ICR MS spectrometer (EI). Infrared spectra were measured on Nicolet Avatar 360. UV were measured on Scinco S-3100. Enantiomeric ratios were determined by HPLC on Agilent 1100 series with chiralpak column with hexane and i-PrOH as eluants. Optical rotations were measured with JASCO P-1020 polarimeter.

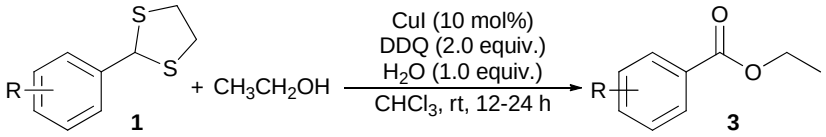
2 Preparation of Substrates

Substrates **1** were prepared according to the known procedures.[1]



3. General Procedure and Spectral Data of Products

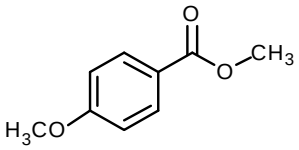
3.1 General Procedure



To a solution of substrates **1** (0.5 mmol) in 5 mL dried $CHCl_3$, C_2H_5OH (30 μL, 0.5 mmol) and 10 mol% of CuI (9.52 mg, 0.05 mmol) were added (a 25 mL flask equipped with rubber stopper). The mixture was stirred for 10 min at rt, then DDQ (227 mg, 1.0 mmol) was added into the reaction system. After an hour, H_2O (9 μL, 0.5 mmol) was added. The reaction was stirred at rt for 12-24 hours. Upon the completion of reaction monitored by TLC, the solvent was removed under reduced pressure and the crude product was purified by flash chromatography on silica gel or neutral aluminium oxide (silica/Al_2O_3: 200~300; eluant: petroleum ether/ethyl acetate) to provide pure products **3**.

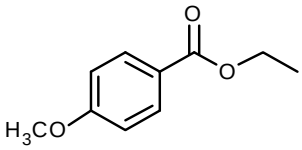
3.2 Spectral Data of Products (all compounds were known except compounds 4e and 5a)

methyl 4-methoxybenzoate (2)[2]



Yield: 92%, MeOH (20.5 μL), white solid. **^{1}H NMR** (600 MHz, $CDCl_3$) δ (ppm) 8.00 (d, J = 8.9 Hz, 2H), 6.92 (d, J = 8.9 Hz, 2H), 3.89 (s, 3H), 3.86 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm) 166.75, 163.21, 131.48, 122.47, 113.49, 55.30, 51.77. **MS**: m/z = 166.09. **UV**: λ_{max} = 264 nm.

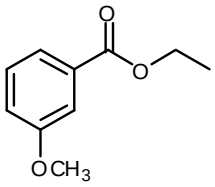
ethyl 4-methoxybenzoate (3a)[2]



Yield: 90% (table 2, entry 1), EtOH (30 μL) and 87% (table 2, entry 13), EtOH (585 μL), white solid. **^{1}H NMR** (600

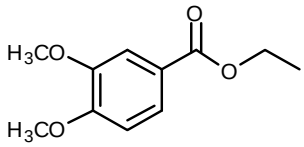
MHz, $CDCl_3$) δ (ppm) 8.00 (d, *J* = 8.8 Hz, 2H), 6.91 (d, *J* = 8.8 Hz, 2H), 4.34 (q, *J* = 7.1 Hz, 2H), 3.84 (s, 3H), 1.38 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm) 166.29, 163.11, 131.42, 122.77, 113.41, 60.55, 55.30, 14.30. **MS**: m/z = 180, **HRMS** (EI) calculated for $C_{10}H_{12}O_3$ $[M]^+$: 180.0786, found 180.0789.

ethyl 3-methoxybenzoate (3b)[3]



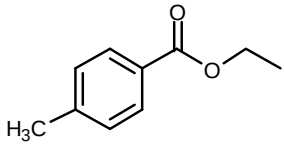
Yield: 74%, EtOH (30 μL), white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm) 7.64 (d, *J* = 7.6 Hz, 1H), 7.56 (s, 1H), 7.33 (t, *J* = 7.9 Hz, 1H), 7.09 (dd, *J* = 8.2, 2.6 Hz, 1H), 4.37 (q, *J* = 7.1 Hz, 2H), 3.84 (s, 3H), 1.39 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm) 166.37, 159.38, 131.65, 129.22, 121.79, 119.17, 113.84, 60.94, 55.28, 14.22. **MS**: m/z = 180.16. **UV**: λ_{max} = 252 nm, 299 nm.

ethyl 3,4-dimethoxybenzoate (3c)[4]



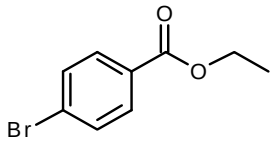
Yield: 89%, EtOH (30 μL), colorless oil, purified by neutral Al_2O_3. **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm) 7.59 (dd, *J* = 8.4, 1.8 Hz, 1H), 7.45 (d, *J* = 1.7 Hz, 1H), 6.78 (d, *J* = 8.4 Hz, 1H), 4.26 (q, *J* = 7.1 Hz, 2H), 3.84 (s, 3H), 3.83 (s, 3H), 1.29 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm) 166.15, 152.54, 148.24, 123.23, 122.69, 111.54, 109.88, 60.58, 55.72, 14.18. **MS**: m/z = 210.10. **UV**: λ_{max} = 264 nm, 295 nm.

ethyl 4-methylbenzoate (3d)[5]



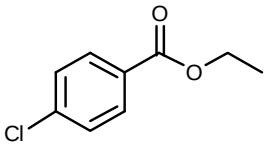
Yield: 71%, EtOH (30 μL), white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm) 7.93 (d, *J* = 8.1 Hz, 2H), 7.22 (d, *J* = 8.0 Hz, 2H), 4.35 (q, *J* = 7.1 Hz, 2H), 2.39 (s, 3H), 1.38 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm) 166.59, 143.30, 129.44, 128.91, 127.61, 60.65, 21.54, 14.25. **MS**: m/z = 164.16. **UV**: λ_{max} = 256 nm.

ethyl 4-bromobenzoate (3e)[2]



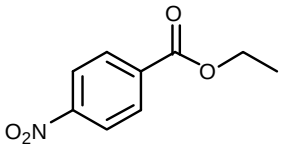
Yield: 81%, EtOH (30 μL), white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm) 8.0–7.81 (m, 2H), 7.57 (d, *J* = 8.5 Hz, 2H), 4.37 (q, *J* = 7.1 Hz, 2H), 1.39 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm) 165.74, 131.52, 130.96, 129.19, 127.79, 61.15, 14.20. **MS**: m/z = 229.99. **UV**: λ_{max} = 264 nm.

ethyl 4-chlorobenzoate (3f)[6]



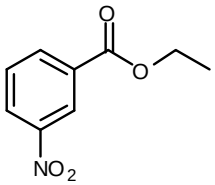
Yield: 80%, EtOH (30 µL), white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm) 8.21–7.75 (m, 2H), 7.52–7.29 (m, 2H), 4.37 (q, *J* = 7.1 Hz, 2H), 1.39 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm) 165.64, 139.13, 130.85, 128.83, 128.56, 61.13, 14.23. **MS**: m/z = 184.01. **UV**: λ_{max} = 263 nm.

ethyl 4-nitrobenzoate (3g)[2]



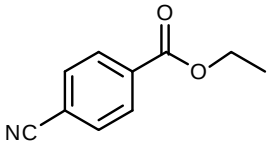
Yield: 92%, EtOH (30 µL), white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm) 8.45–8.26 (m, 2H), 8.26–8.12 (m, 2H), 4.44 (q, *J* = 7.2 Hz, 2H), 1.44 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm) 164.61, 150.36, 135.76, 130.59, 123.42, 61.90, 14.17. **MS**: m/z = 195.15. **UV**: λ_{max} = 265 nm, 304 nm.

ethyl 3-nitrobenzoate (3h)[5]



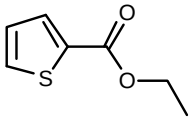
Yield: 84%, EtOH (30 µL), white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm) 8.87 (d, *J* = 1.7 Hz, 1H), 8.54–8.25 (m, 2H), 7.67 (t, *J* = 8.0 Hz, 1H), 4.45 (q, *J* = 7.1 Hz, 2H), 1.44 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) 164.42, 148.19, 135.24, 132.17, 129.54, 127.25, 124.50, 61.91, 14.24. **MS**: m/z = 195.11. **UV**: λ_{max} = 265 nm, 296 nm.

ethyl 4-cyanobenzoate (3i)[7]



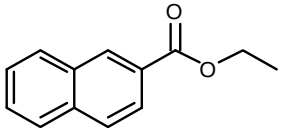
Yield: 87%, EtOH (30 µL), white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm) 8.15 (dd, *J* = 8.3, 1.7 Hz, 2H), 7.76 (dd, *J* = 8.2, 1.7 Hz, 2H), 4.43 (q, *J* = 7.1 Hz, 2H), 1.43 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm) 164.78, 134.11, 132.05, 129.91, 117.89, 116.09, 61.67, 14.10. **MS**: m/z = 175.17. **UV**: λ_{max} = 263 nm.

ethyl thiophene-2-carboxylate (3j)[2]



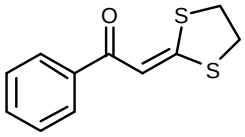
Yield: 62%, EtOH (30 µL), white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm) 7.80 (d, *J* = 2.9 Hz, 1H), 7.66–7.43 (m, 1H), 7.22–6.97 (m, 1H), 4.36 (q, *J* = 7.1 Hz, 2H), 1.38 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm) 162.24, 134.00, 133.21, 132.14, 127.64, 61.11, 14.30. **MS**: m/z = 156.13. **UV**: λ_{max} = 264 nm.

ethyl 2-naphthoate (3k)[8]



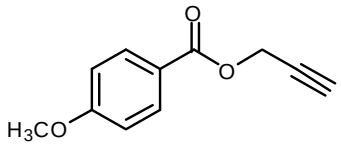
Yield: 78%, EtOH (30 μL), colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm) 8.60 (s, 1H), 8.06 (dd, *J* = 8.6, 1.6 Hz, 1H), 7.93 (d, *J* = 7.9 Hz, 1H), 7.85 (d, *J* = 8.8 Hz, 2H), 7.64–7.43 (m, 2H), 4.43 (q, *J* = 7.1 Hz, 2H), 1.43 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm) 166.69, 135.36, 132.38, 130.84, 129.24, 128.06, 127.99, 127.65, 127.60, 126.50, 125.15, 61.03, 14.33. **MS**: m/z = 200.10. **UV**: λ_{max} = 265 nm, 300 nm.

2-benzoylmethylidene-1,3-dithiolane (3l)[9]



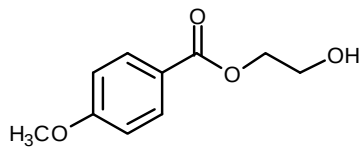
Yield: 65%, EtOH (145 μL), pale yellow solid, purified by neutral Al_2O_3. **^{1}H NMR** (600 MHz, $CDCl_3$) δ (ppm) 7.95 (d, *J* = 7.5 Hz, 2H), 7.51 (t, *J* = 7.3 Hz, 1H), 7.45 (t, *J* = 7.5 Hz, 2H), 7.37 (s, 1H), 3.49 (t, *J* = 6.1 Hz, 2H), 3.40 (t, *J* = 6.3 Hz, 2H); **^{13}C NMR** (150 MHz, $CDCl_3$) δ (ppm) 185.87, 168.15, 138.05, 131.92, 128.38, 127.69, 108.13, 38.79, 35.31. ν_{max}/cm^{-1} (KBr) 1609 (C=O), **MS**: m/z = 222.04.

Prop-2-ynyl p-methoxybenzoate (4a)[2]



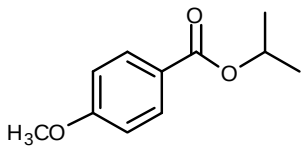
Yield: 41%, propargyl alcohol (150 μL), colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm) 8.11–7.95 (m, 2H), 6.99–6.86 (m, 2H), 4.90 (d, *J* = 2.4 Hz, 2H), 3.86 (s, 3H), 2.52 (t, *J* = 2.4 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm) 165.46, 163.58, 131.83, 121.65, 113.62, 77.92, 74.77, 55.40, 52.12. **MS**: m/z = 190.16. **UV**: λ_{max} = 264 nm.

2-hydroxyethyl 4-methoxybenzoate (4b)[10]



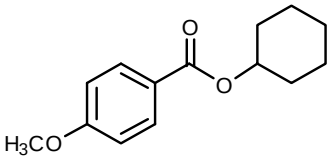
Yield: 76%, ethylene glycol (32 μL), pale yellow oil, purified by neutral Al_2O_3. **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm) 8.01 (d, *J* = 8.8 Hz, 2H), 6.91 (d, *J* = 8.8 Hz, 2H), 4.51–4.34 (m, 2H), 3.94 (dd, *J* = 8.9, 5.0 Hz, 2H), 3.85 (s, 3H), 2.66 (t, *J* = 5.5 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm) 166.72, 163.40, 131.66, 122.05, 113.55, 66.35, 61.30, 55.37. **MS**: m/z = 196.16. **UV**: λ_{max} = 264 nm.

isopropyl 4-methoxybenzoate (4c)[2]



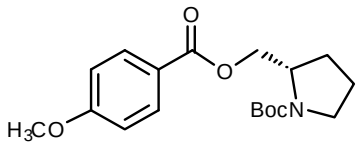
Yield: 41%, isopropanol (39 μL), colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm) 8.00 (d, *J* = 8.8 Hz, 2H), 6.91 (d, *J* = 8.8 Hz, 2H), 5.23 (dt, *J* = 12.5, 6.2 Hz, 1H), 3.86 (s, 3H), 1.36 (s, 3H), 1.35 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm) 165.80, 163.06, 131.41, 123.23, 113.38, 67.85, 55.32, 21.93. **MS**: m/z = 194.19. **UV**: λ_{max} = 264 nm.

cyclohexyl 4-methoxybenzoate (4d)[11]

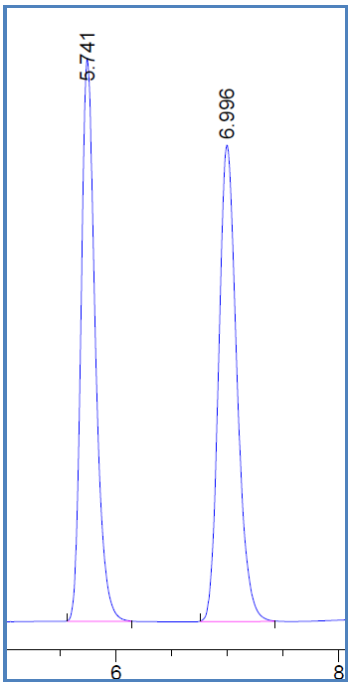


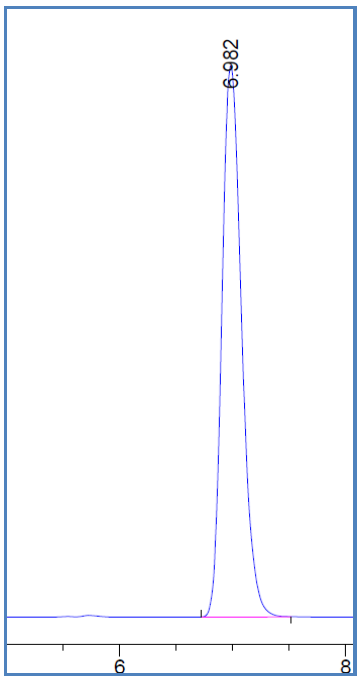
Yield: 50%, cyclohexanol (100.2 mg), white solid. **^{1}H NMR** (600 MHz, $CDCl_3$) δ (ppm) 8.00 (d, *J* = 8.5 Hz, 2H), 6.91 (d, *J* = 8.3 Hz, 2H), 5.04–4.95 (m, 1H), 3.85 (s, 3H), 1.92 (s, 2H), 1.78 (d, *J* = 12.5 Hz, 2H), 1.63–1.52 (m, 3H), 1.49–1.39 (m, 2H), 1.38–1.28 (m, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm) 165.66, 163.04, 131.42, 123.33, 113.38, 72.56, 55.32, 31.61, 25.43, 23.62. **MS**: m/z = 234.13. **UV**: λ_{max} = 264 nm.

(*S*)-tert-butyl 2-(((4-methoxybenzoyl)oxy)methyl)pyrrolidine-1-carboxylate (4e)



Yield: 76%, N-Boc-*L*-Prolinol (100.7 mg), colorless oil, $[\alpha]_D^{27}$ -62.4 (*c* 1.0, MeOH) for 100% *ee*, purified by neutral Al_2O_3. **^{1}H NMR** (600 MHz, $CDCl_3$) δ (ppm) 7.99 (d, *J* = 8.1 Hz, 2H), 6.92 (d, *J* = 5.9 Hz, 2H), 4.37 (s, 1H), 4.22 (m, 2H), 3.86 (s, 3H), 3.52–3.31 (m, 2H), 1.96 (m, 4H), 1.47 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm) 165.83, 163.18, 154.21, 131.38, 122.18, 113.38, 79.44, 79.08, 64.72, 64.51, 55.49, 55.15, 46.48, 46.25, 28.63, 28.22, 27.68, 23.62, 22.85; **MS**: m/z = 335, **HRMS** (EI) calculated for $C_{18}H_{25}NO_5$ $[M]^+$: 335.1733, found 335.1737; The enantiomeric excess was determined by chiral HPLC (Chiralpak AD column: hexane/2-propanol = 80/20, 1 mL/min, 254 nm, t_{major} = 6.98 min).





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2	6.996	BB	0.1750	5135.89697	451.13449	51.8954

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	6.982	BB	0.1897	2.32823e4	1917.65588	100.0000

Reference:

(1) H. Firouzabadi, N. Iranpoor and H. Hazarkhani, *J. Org. Chem.*, 2001, **66**, 7527.

(2) X.-F. Wu and C. Darcel, *Eur. J. Org. Chem.*, 2009, 1144.

(3) B. Karimi and J. Rajabi, *Synthesis*, 2003, 2373.

(4) Shigekazu and Yamazaki, *Chem. Lett.*, 1995, 127.

(5) Q.-H. Xu, W.-Y. Liu, B.-H. Chen and Y.-X. Ma, *Synth. Commun.*, 2001, **31**, 2113.

(6) P. J. Siler, S. T. Chill and R. C. Mebane, *Synth. Commun.*, 2011, **41**, 1247.

(7) Chidambaram and Ramakrishnan, *Tetrahedron Lett.*, 2004, **45**, 1441.

(8) J. Mo and P. H. Lee, *Org. Lett.*, 2010, **12**, 2570.

(9) K. Tanemura, H. Dohya, M. Imamura, T. Suzuki and T. Horaguchi, *J. Chem. Soc., Perkin*

Trans. 1, 1996, 453.

(10) T. Yasukawa, H. Miyamura and S. Kobayashi, *Chem. Asian J.*, 2011, **6**, 621.

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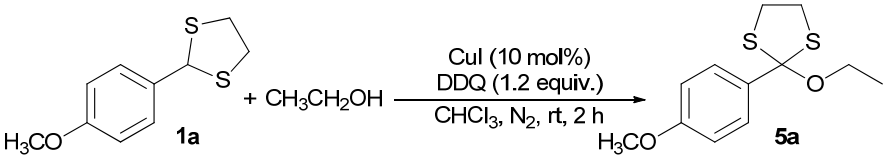
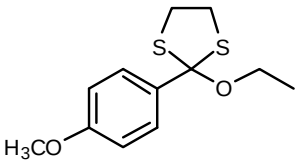
4. Mechanism Study

4.1 Procedure and Spectral Data of Intermediate 5a



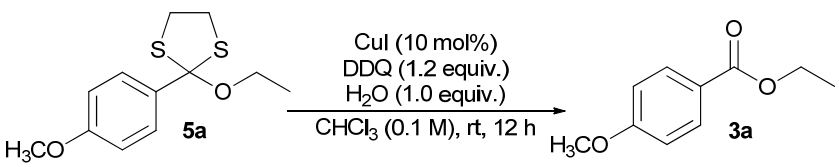
To a solution of substrate **1a** (106.2 mg, 0.5 mmol) in 5 mL dried $CHCl_3$, C_2H_5OH (145 μL, 2.5 mmol) and 10 mol% of CuI (9.52 mg, 0.05 mmol) were added under nitrogen atmosphere. The mixture was stirred for 10 min at rt, then DDQ (136.2 mg, 0.6 mmol) was added into the reaction system. Upon the completion of reaction monitored by TLC (basic $KMnO_4$ and heating), the mixture was filtered through a plug of neutral aluminium oxide (DCM as eluant) as soon as quickly, solvent was removed under reduced pressure and crude product was purified by flash chromatography on neutral aluminium oxide (Al_2O_3: 200~300; eluant: petroleum ether/ethyl acetate) to provide pure product **5a** in 77% yield.

2-ethoxy-2-PMP-1, 3-dithiolane (5a)



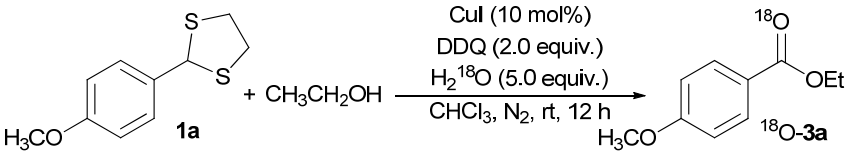
Yield: 77%, colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm) 7.75–7.58 (m, 2H), 6.90–6.78 (m, 2H), 3.79 (s, 3H), 3.66 (q, J = 7.0 Hz, 2H), 3.58–3.45 (m, 4H), 1.27 (t, J = 7.0 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm) 159.24, 133.64, 127.71, 113.20, 109.84, 60.85, 55.17, 40.75, 14.90. **MS**: m/z = 256, **HRMS** (EI) calculated for $C_{12}H_{16}O_2S_2$ $[M]^+$: 256.0592, found 256.0595. **UV**: λ_{max} = 264 nm.

4.2 Procedure for Ester 3a from Intermediate 5a

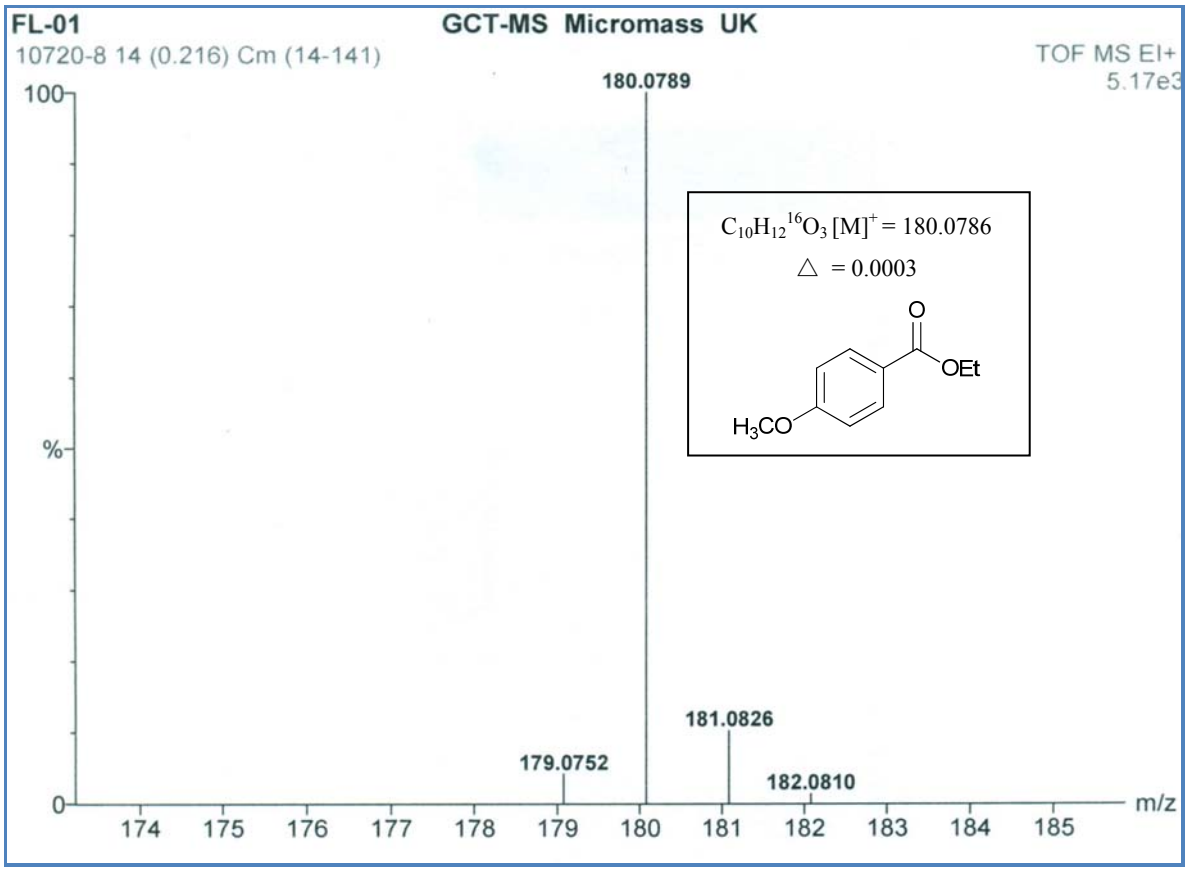


To a solution of substrate **5a** (128.2 mg, 0.5 mmol) in 5 mL dried $CHCl_3$, 10 mol% of CuI (9.52 mg, 0.05 mmol) and DDQ (136.2 mg, 0.6 mmol) were added, then H_2O (9 μL, 0.5 mmol) was added into the reaction system immediately, the reaction was stirred at rt for 12 hours. Upon the completion of reaction monitored by TLC, the solvent was removed under reduced pressure and the crude product was purified by flash chromatography on silica gel (silica: 200~300; eluant: petroleum ether/ethyl acetate) to provide pure product **3a** in 92% yield.

4.3 $H_2{}^{18}O$ Labeling Experiment



To a solution of substrate **1a** (106.2 mg, 0.5 mmol) in 5 mL dried $CHCl_3$, C_2H_5OH (145 μL, 2.5 mmol) and 10 mol% of CuI (9.52 mg, 0.05 mmol) were added under nitrogen atmosphere. The mixture was stirred for 10 min at rt, then DDQ (227 mg, 1.0 mmol) was added into the reaction system and 98% $H_2{}^{18}O$ (50 μL, 2.5 mmol) was added immediately, the reaction was stirred at rt for 12 hours. Upon the completion of reaction monitored by TLC, the solvent was removed under reduced pressure and the crude product was purified by flash chromatography on silica gel (silica: 200~300; eluant: petroleum ether/ethyl acetate) to provide the product ^{18}O-**3a** in 87% yield. **MS**: m/z = 182, **HRMS** (EI) calculated for $C_{10}H_{12}{}^{16}O_2{}^{18}O$ $[M]^+$: 182.0829, found 182.0831.

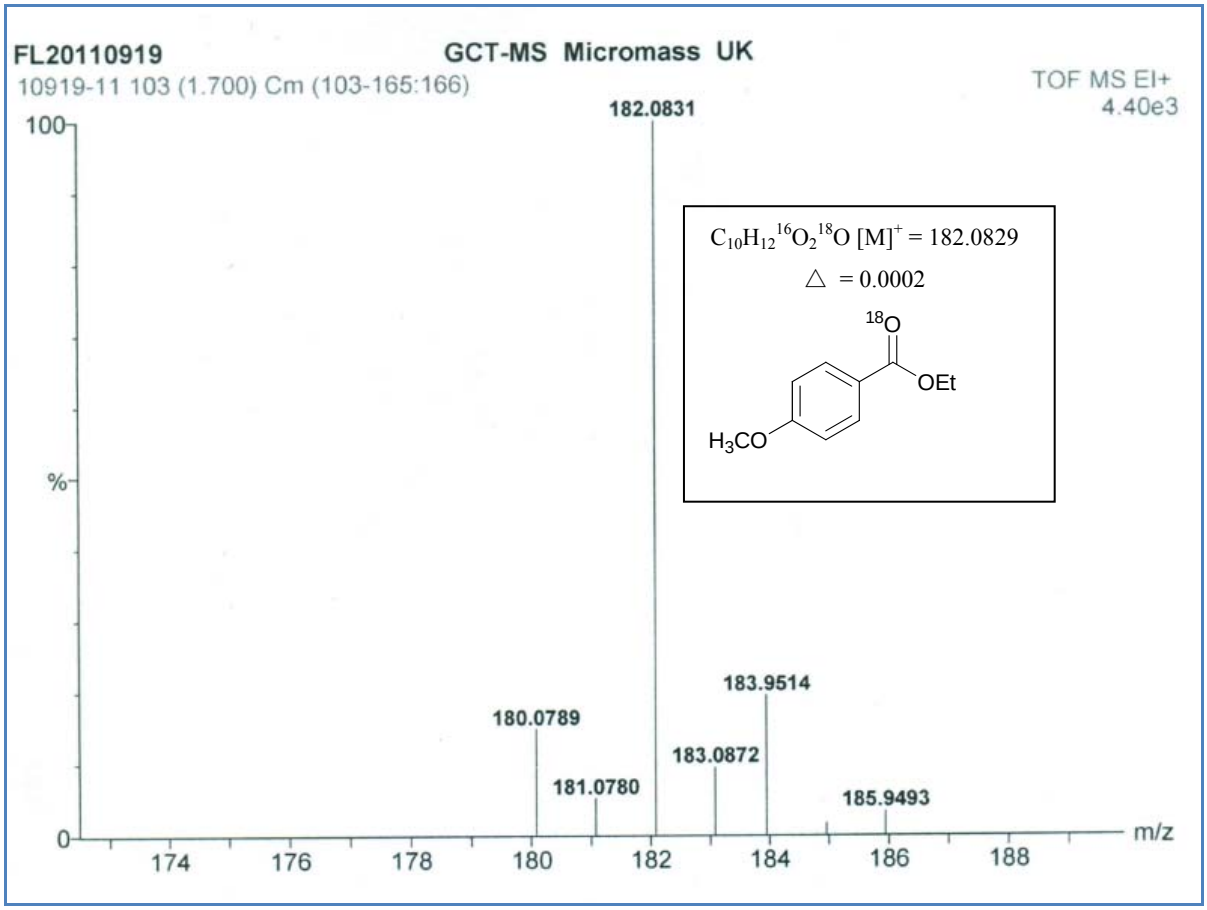


Elemental Composition Report

Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions
78 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Minimum:	80.00				-1.5		
Maximum:	100.00		200.0	10.0	50.0		
Mass	RA	Calc. Mass	mDa	PPM	DBE	Score	Formula
180.0789	100.00	180.0786	0.3	1.4	5.0	1	C10 H12 16O3



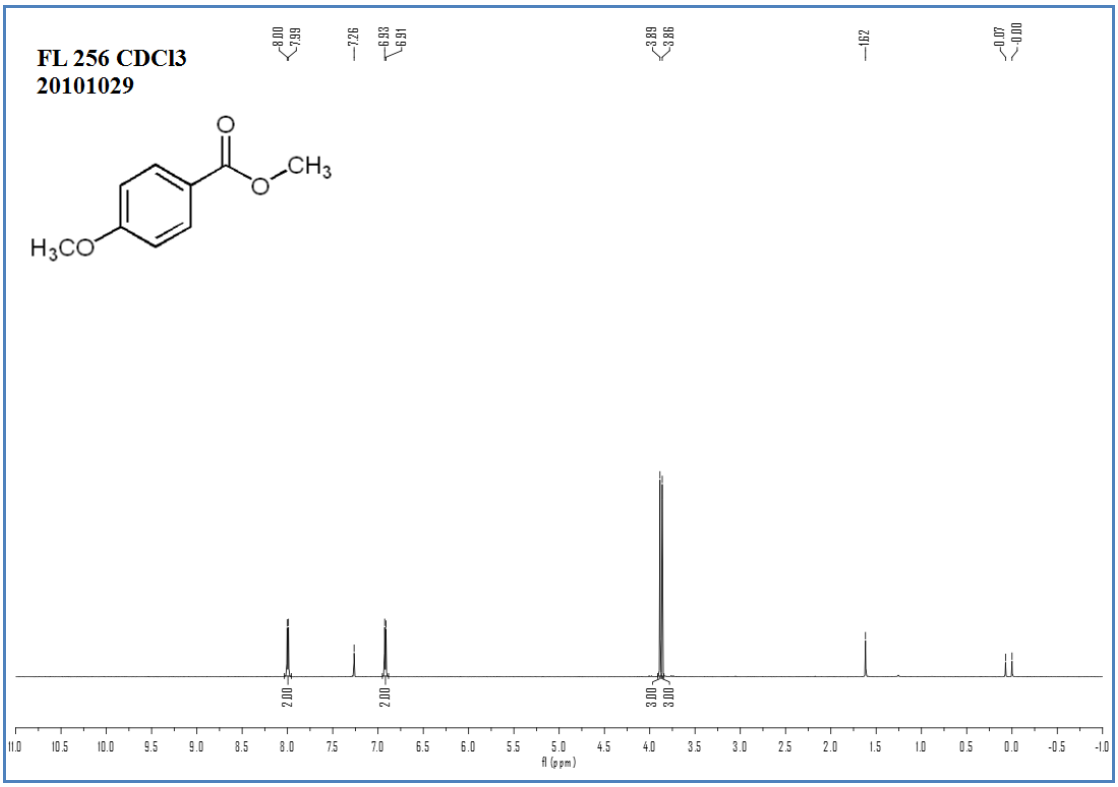
Elemental Composition Report

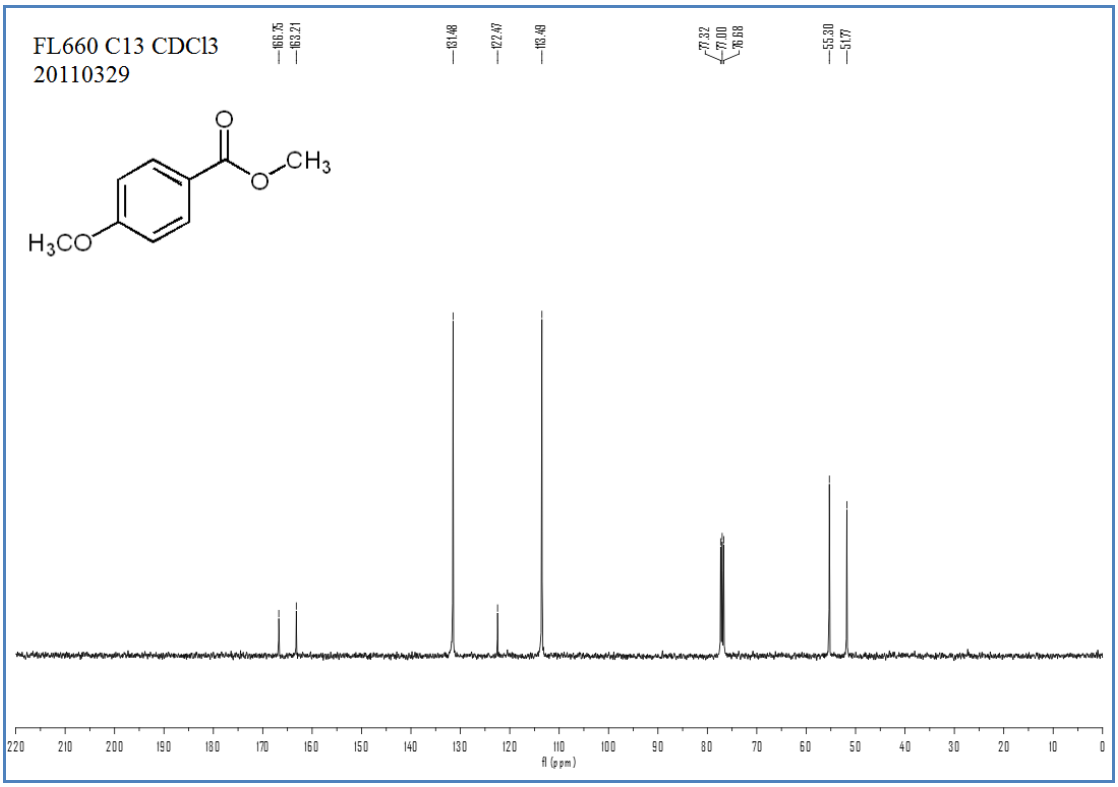
Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

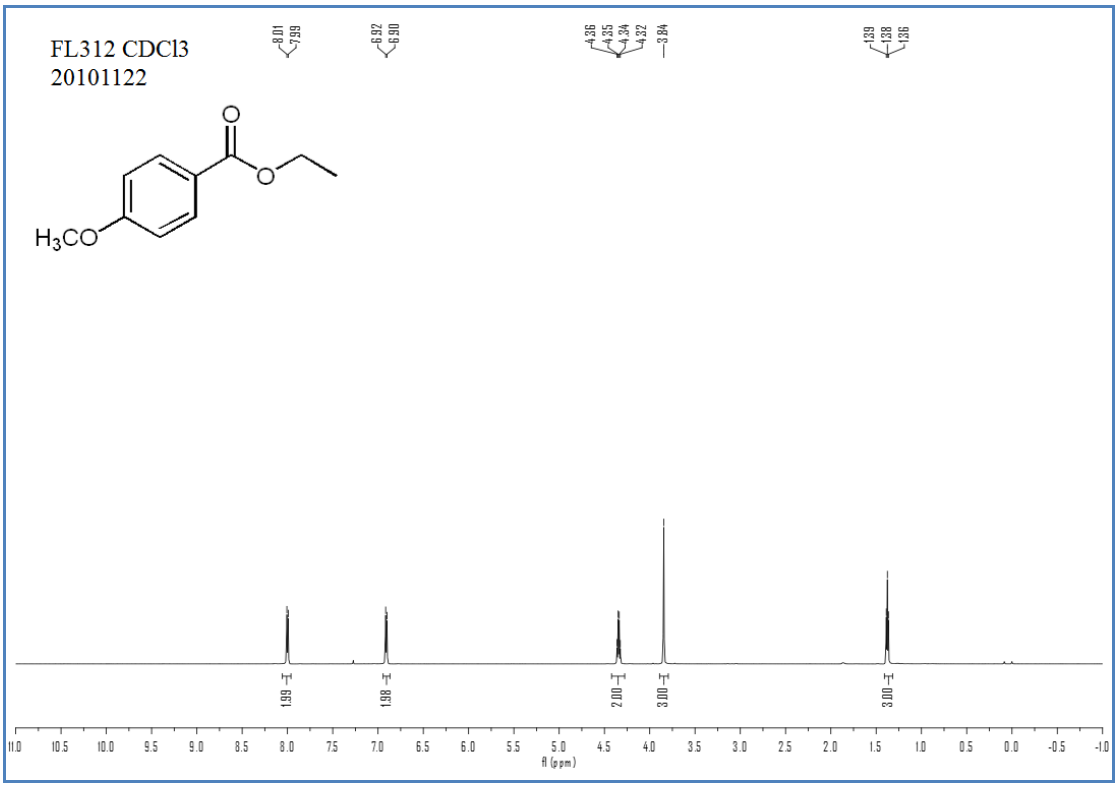
Monoisotopic Mass, Odd and Even Electron Ions
94 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Minimum:	80.00				-1.5		
Maximum:	100.00		200.0	10.0	50.0		
Mass	RA	Calc. Mass	mDa	PPM	DBE	Score	Formula
182.0831	100.00	182.0829	0.2	1.2	5.0	1	C10 H12 16O2 18O

5. Copies of ^{1}H NMR and ^{13}C NMR spectrums



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6.90
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4.35
4.34
4.32
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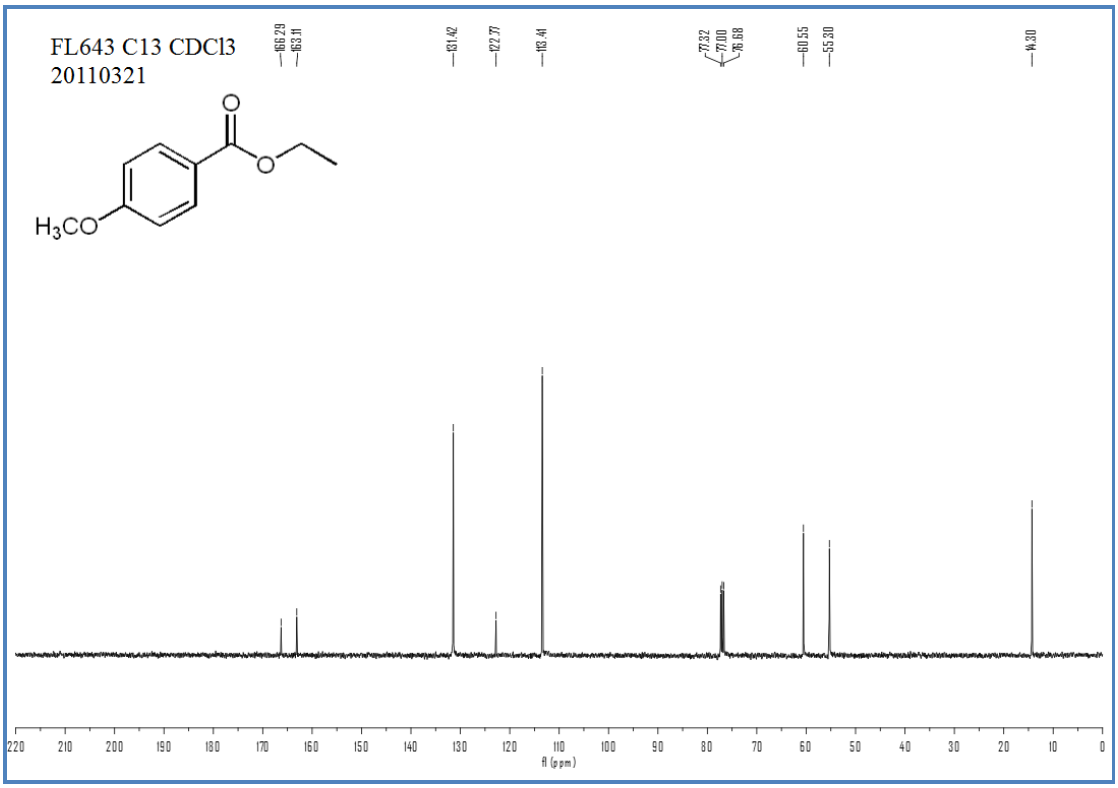
1.98
2.00
3.00
3.00
f1 (ppm)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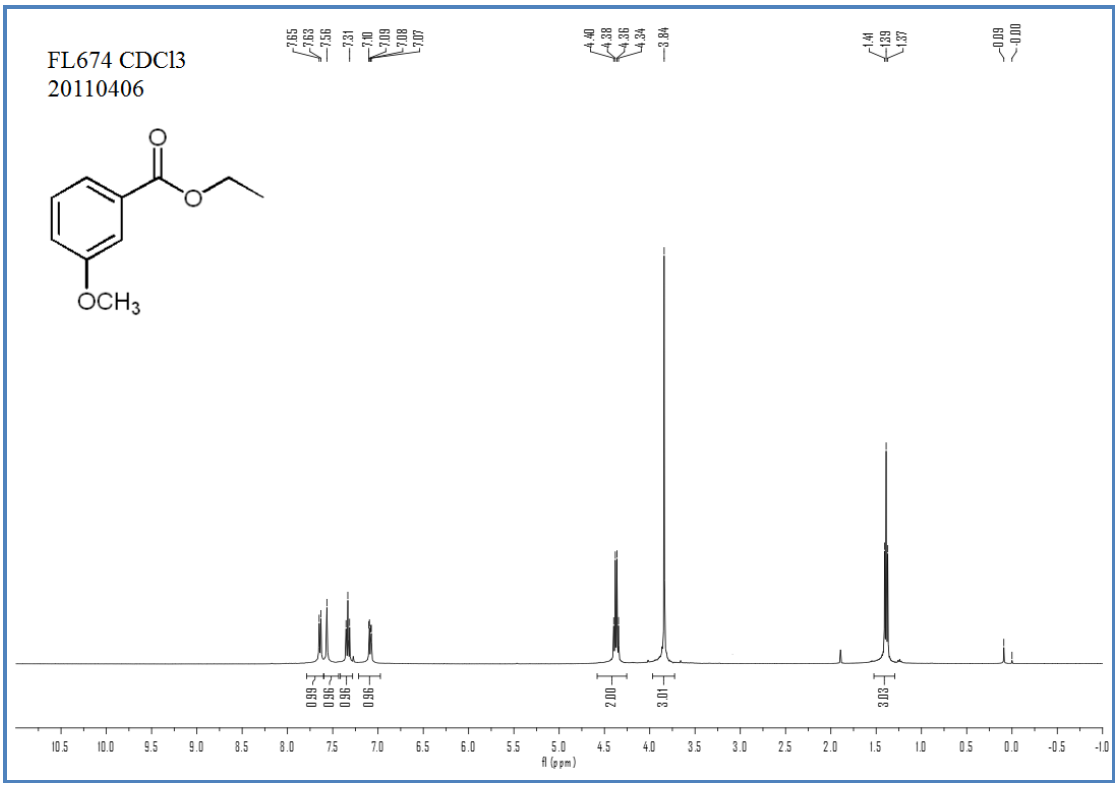
60.55
55.30
14.30
H3CO
f1 (ppm)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OCH3
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7.63
7.56
7.31
7.10
7.09
7.08
7.07
4.40
4.38
4.36
4.34
3.84
1.41
1.39
1.37
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0.00
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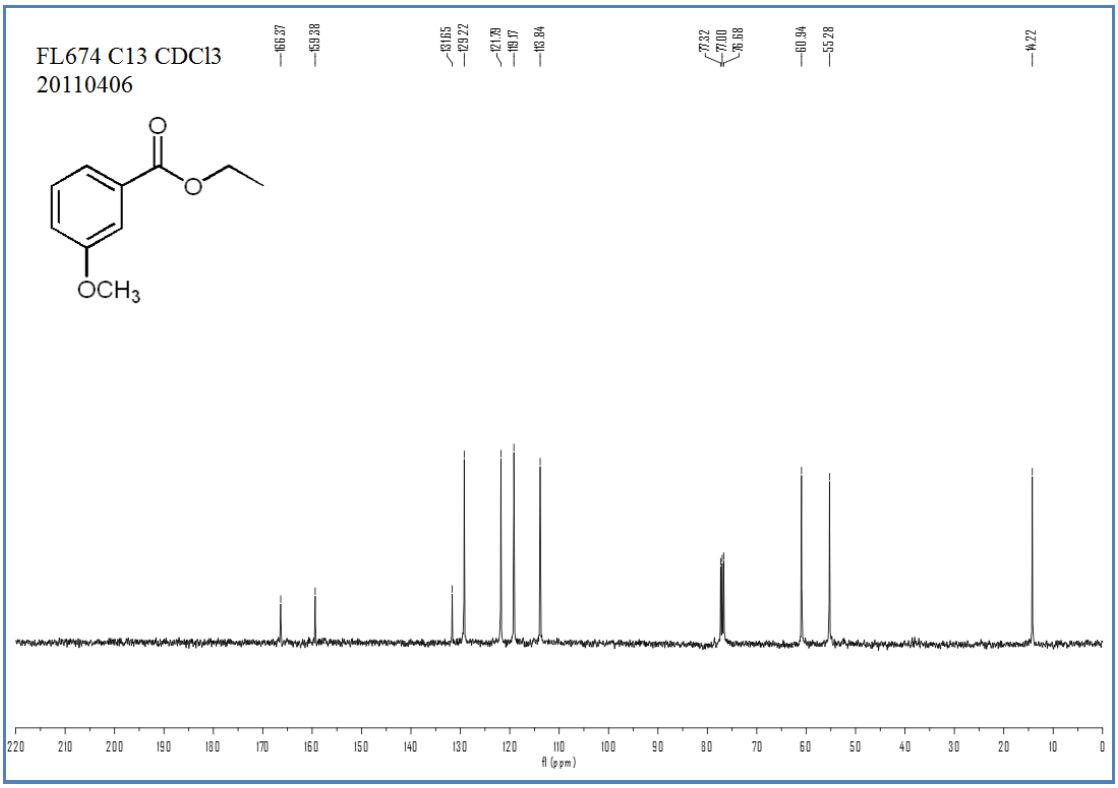
0.96
0.96
0.96
2.00
3.01
3.03
f1 (ppm)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OCH3
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113.84
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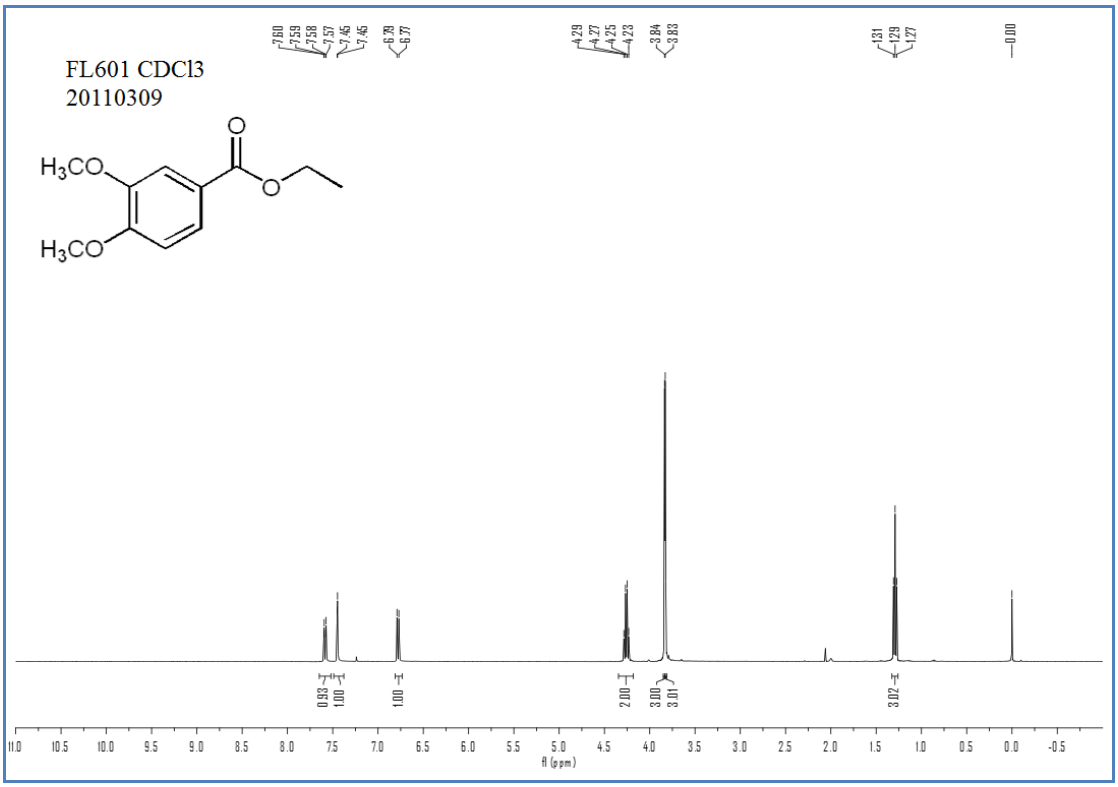
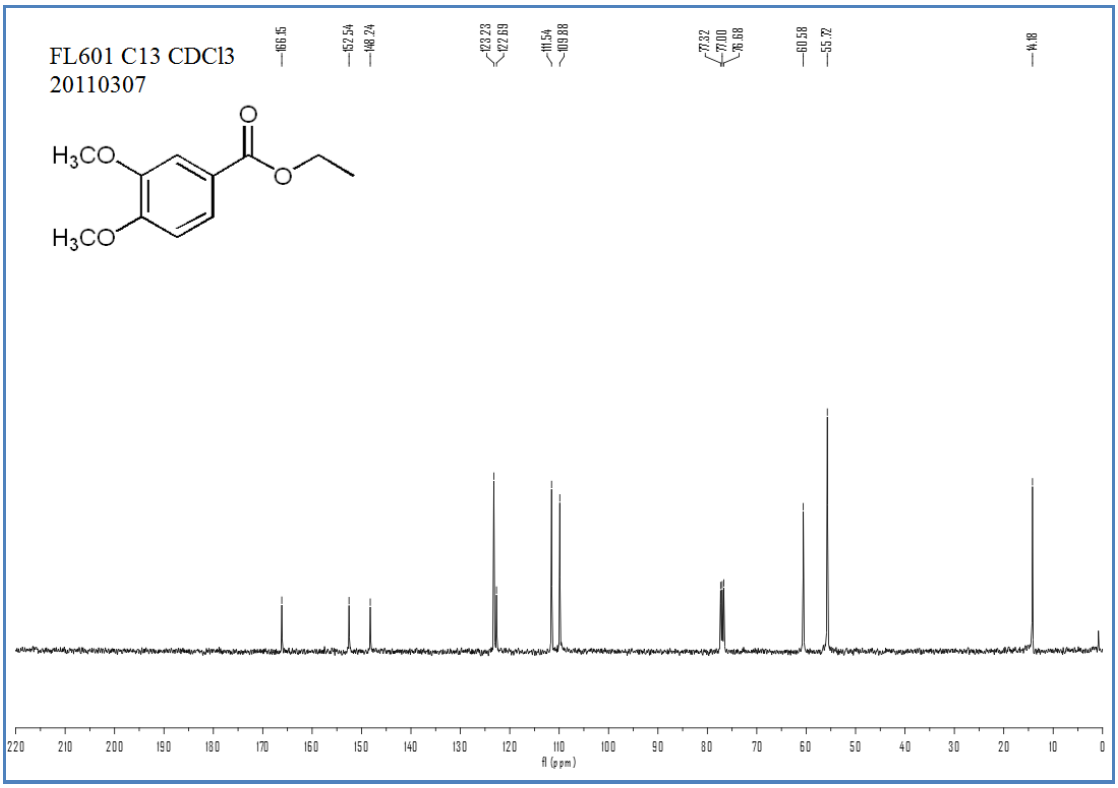
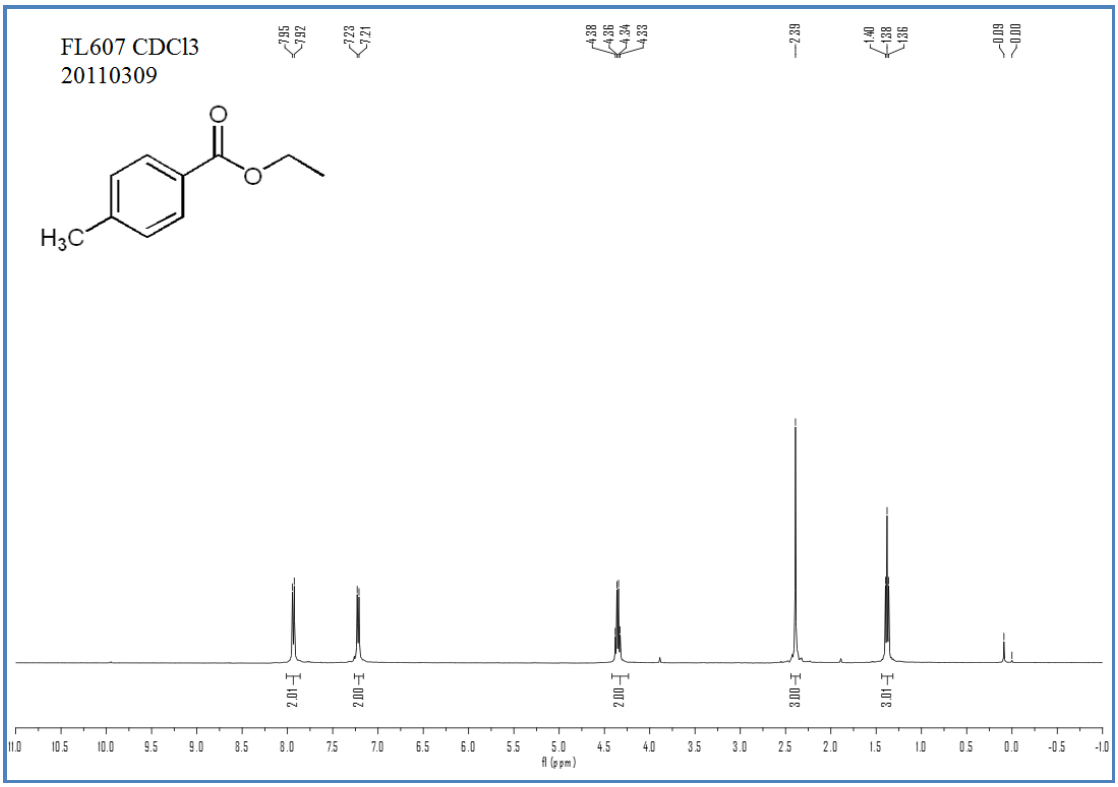
76.68
60.94
55.28
14.22
f1 (ppm)



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7.23
7.21
4.38
4.36
4.34
4.33
2.39
1.40
1.38
1.36
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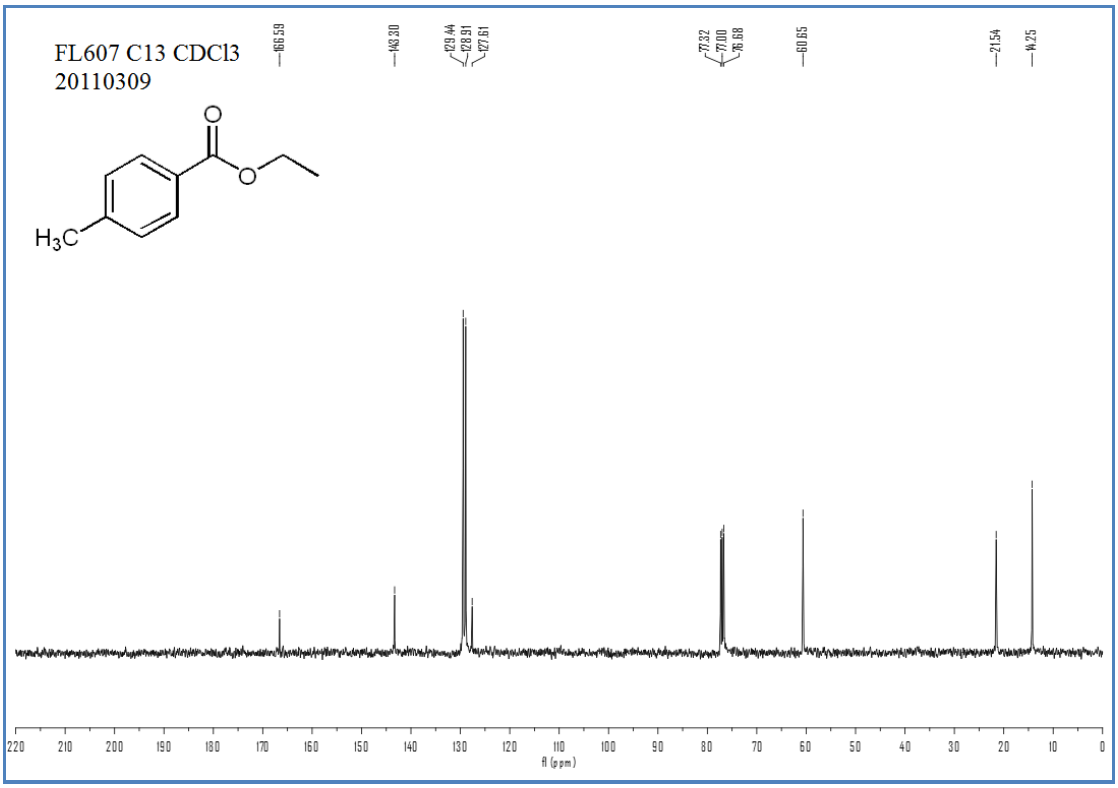
0.010
H3C
O
O
2.01
2.00
2.00
3.00
3.01
f1 (ppm)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14.25
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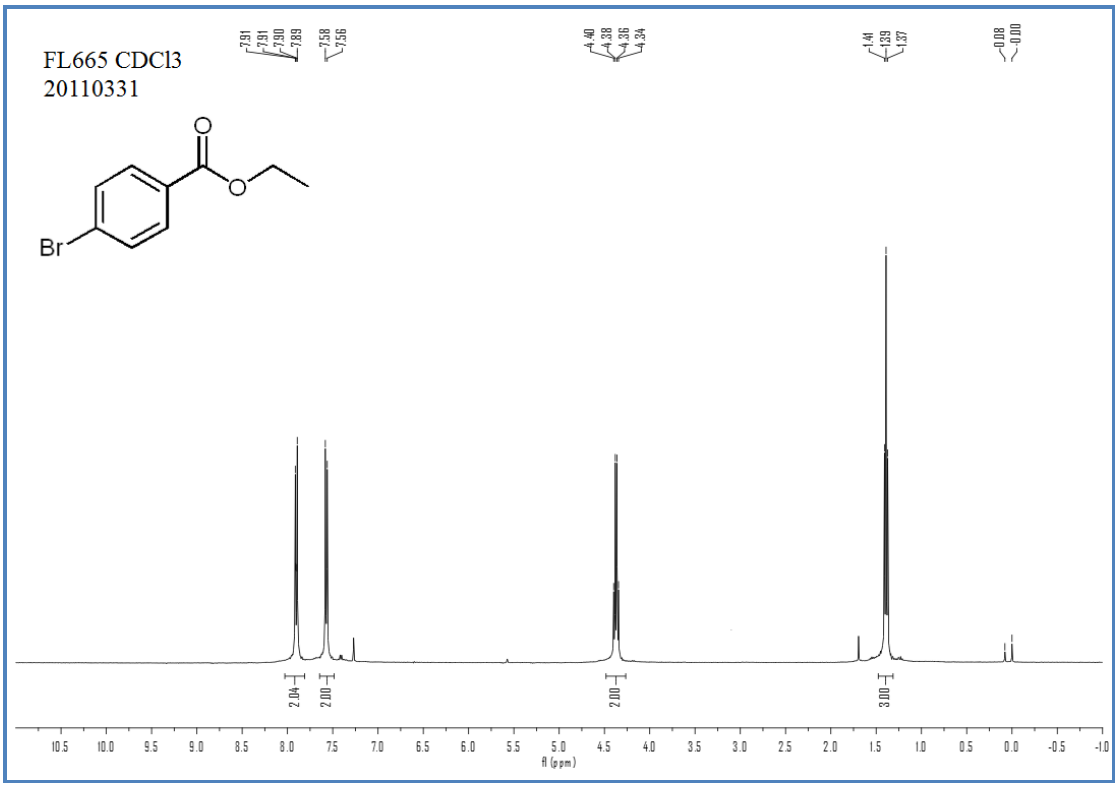
O
O
f1 (ppm)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7.56
4.40
4.38
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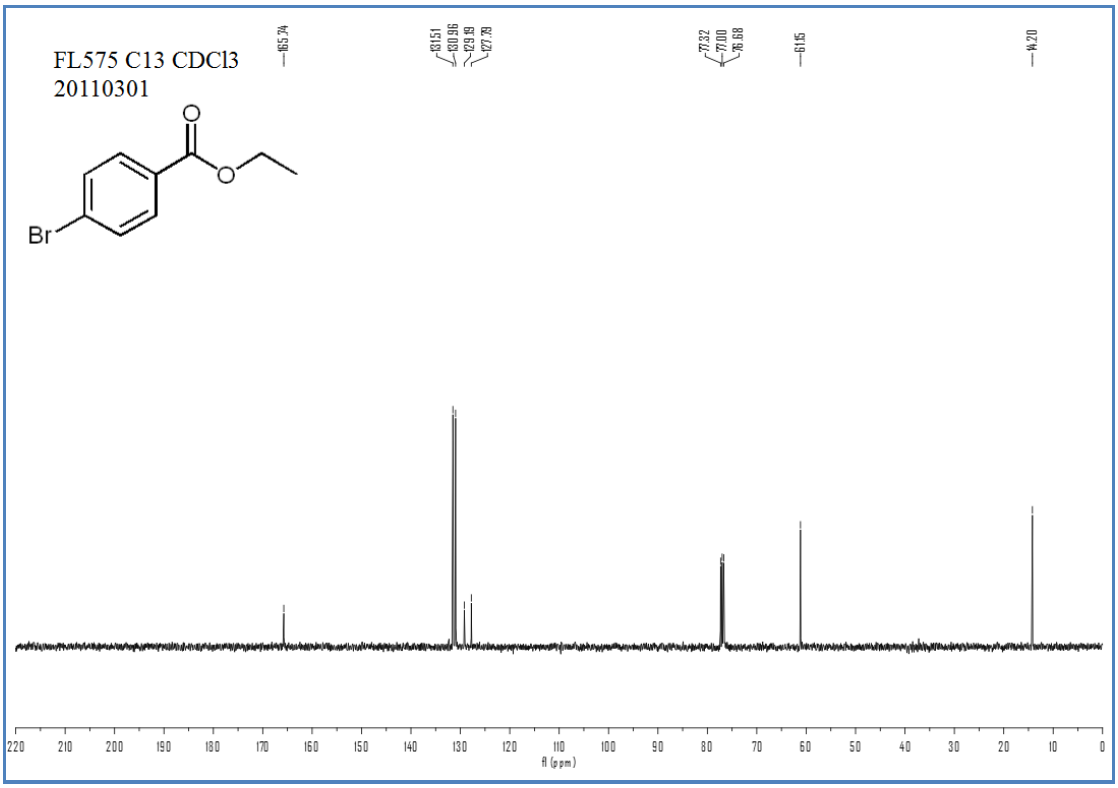
1.39
1.37
0.08
0.00
2.04
2.00
2.00
3.00
f1 (ppm)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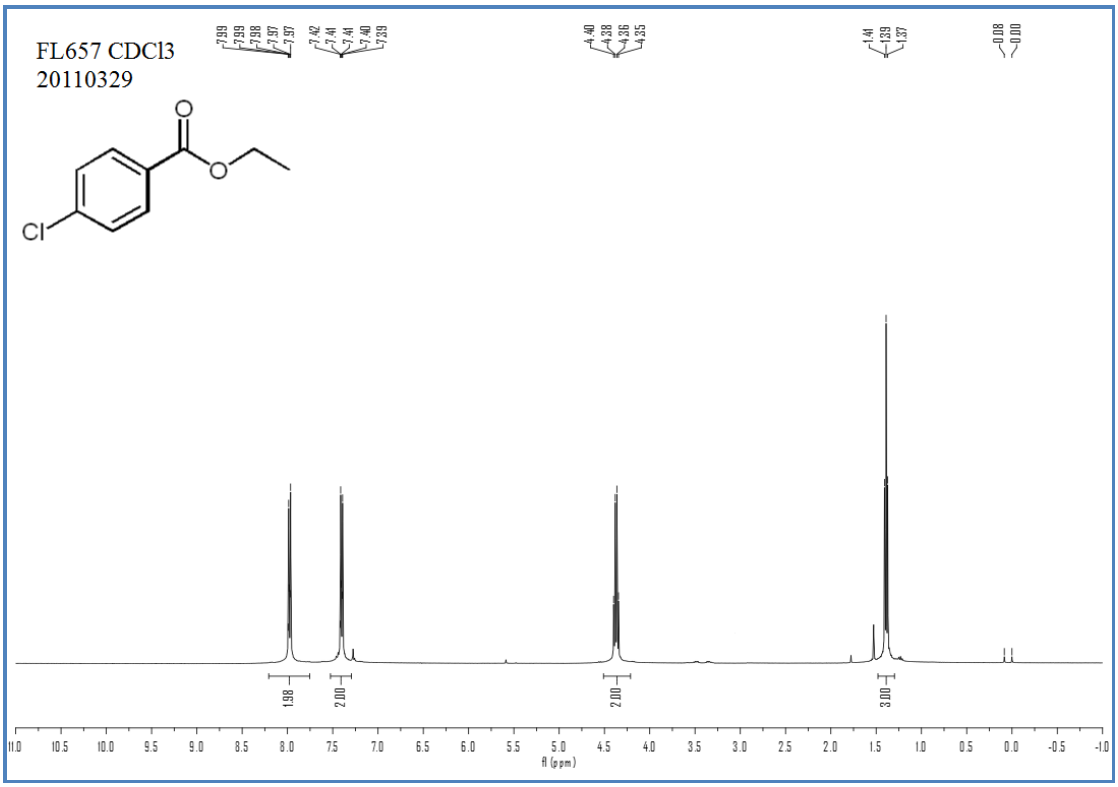
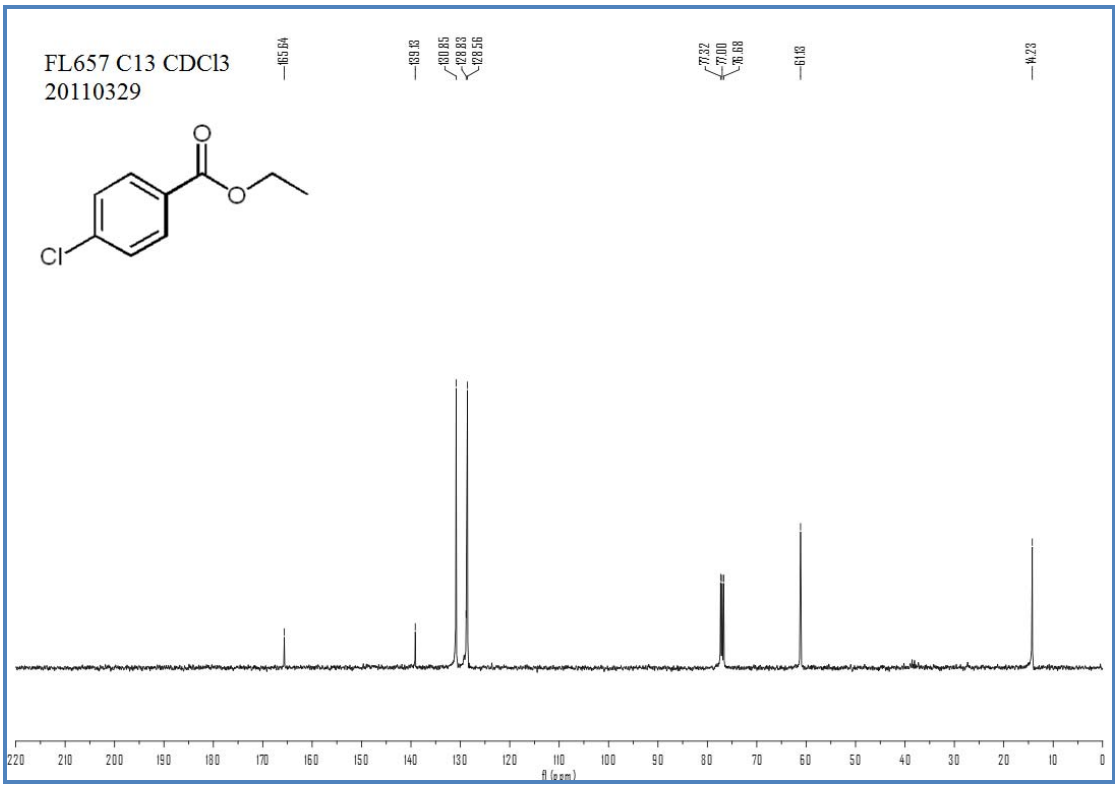
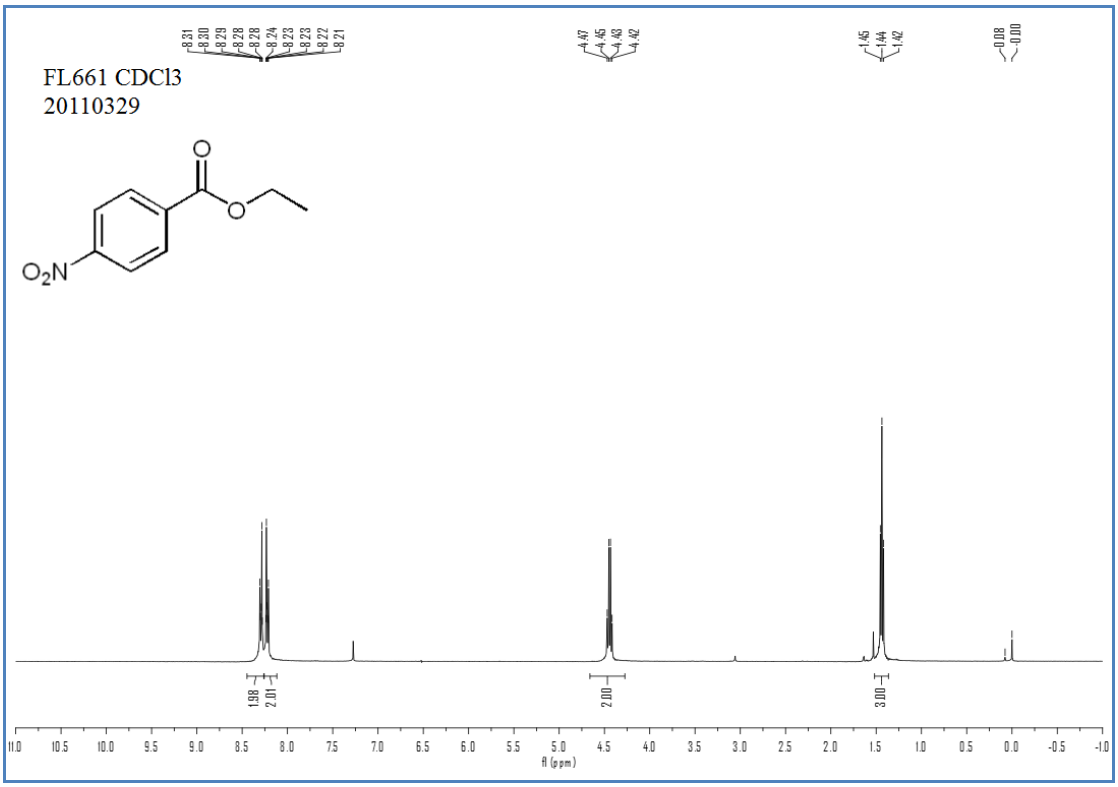
14.20
f1 (ppm)



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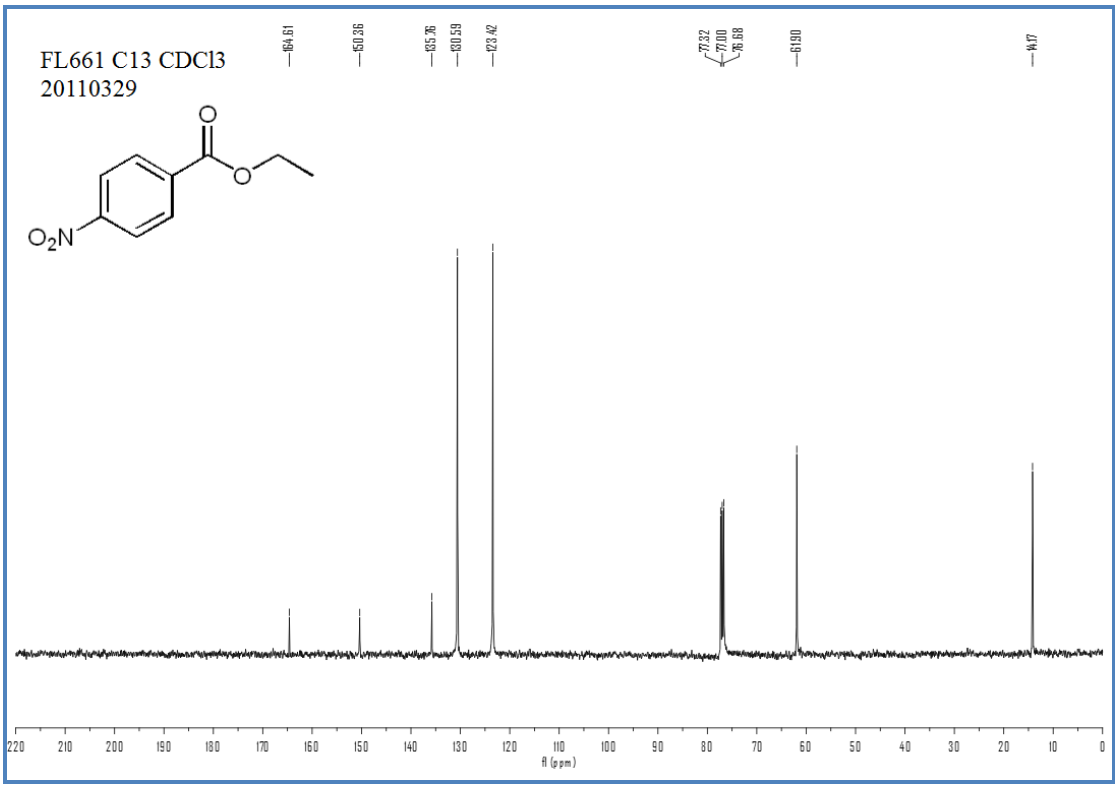
8.29
8.28
8.28
8.24
8.23
8.23
8.22
8.21
4.47
4.45
4.43
4.42
1.45
1.44
1.42
0.08
0.00
1.98
2.01
2.00
3.00
f1 (ppm)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77.00
76.68
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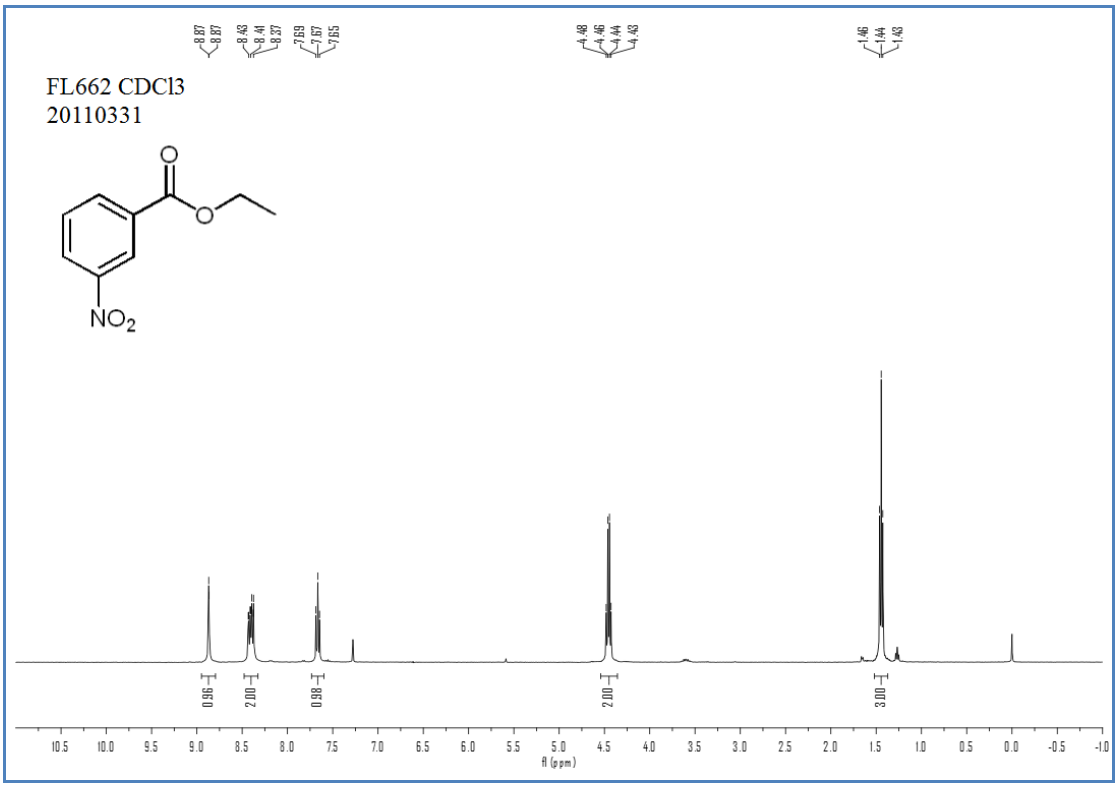
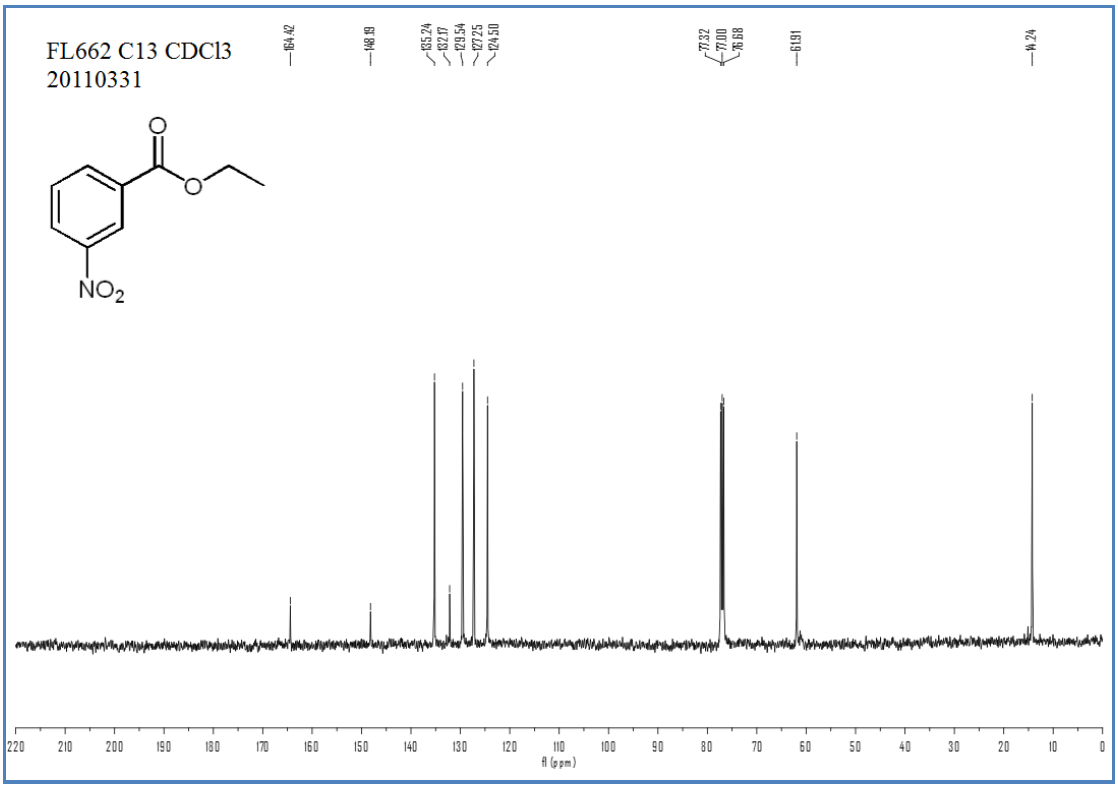
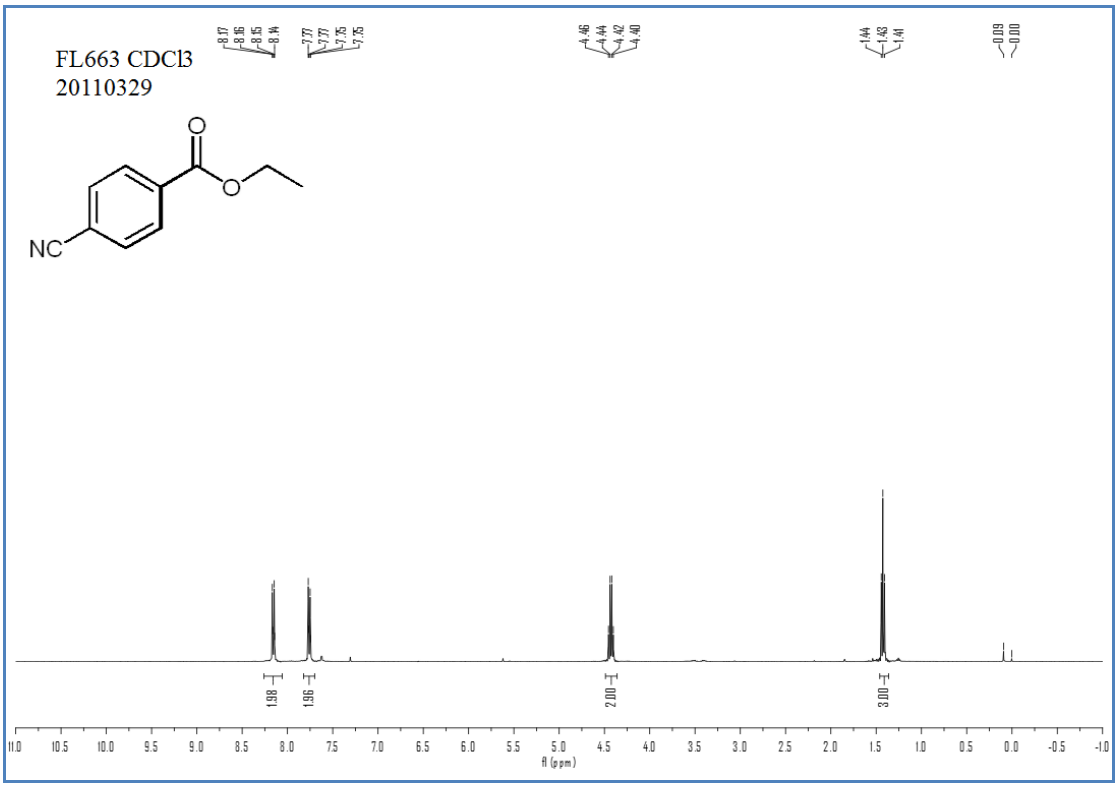
14.17
f1 (ppm)



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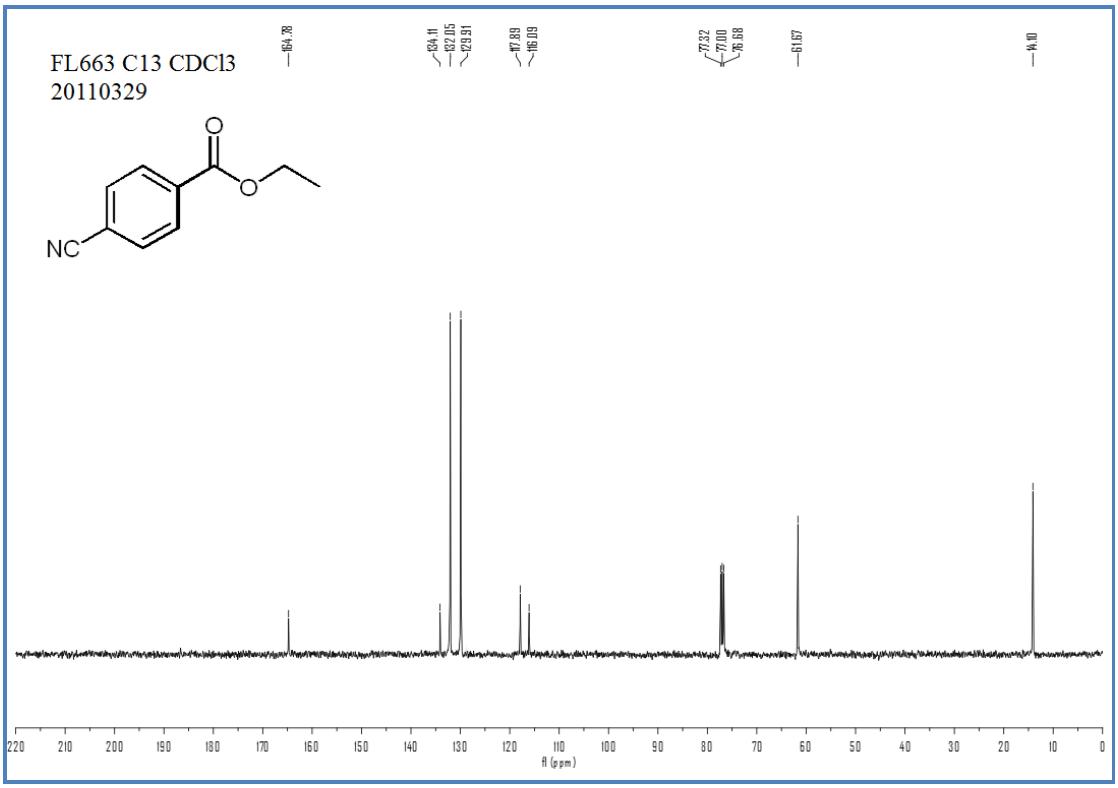
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20110329
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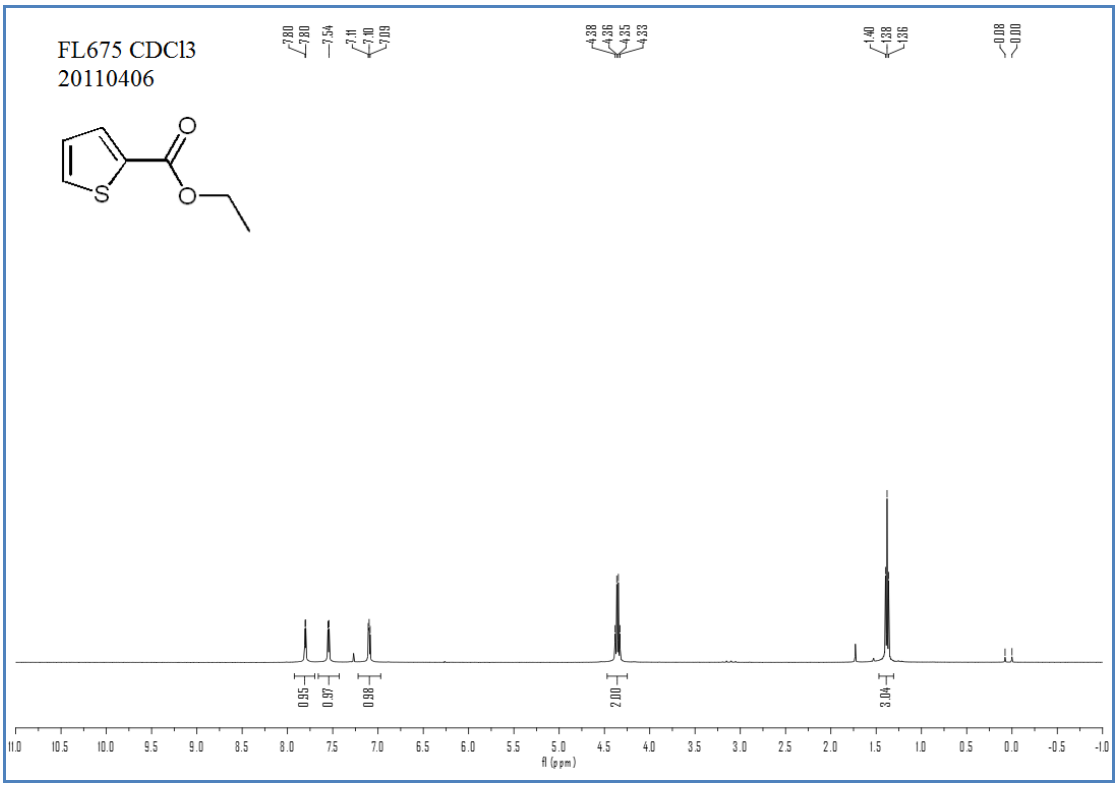
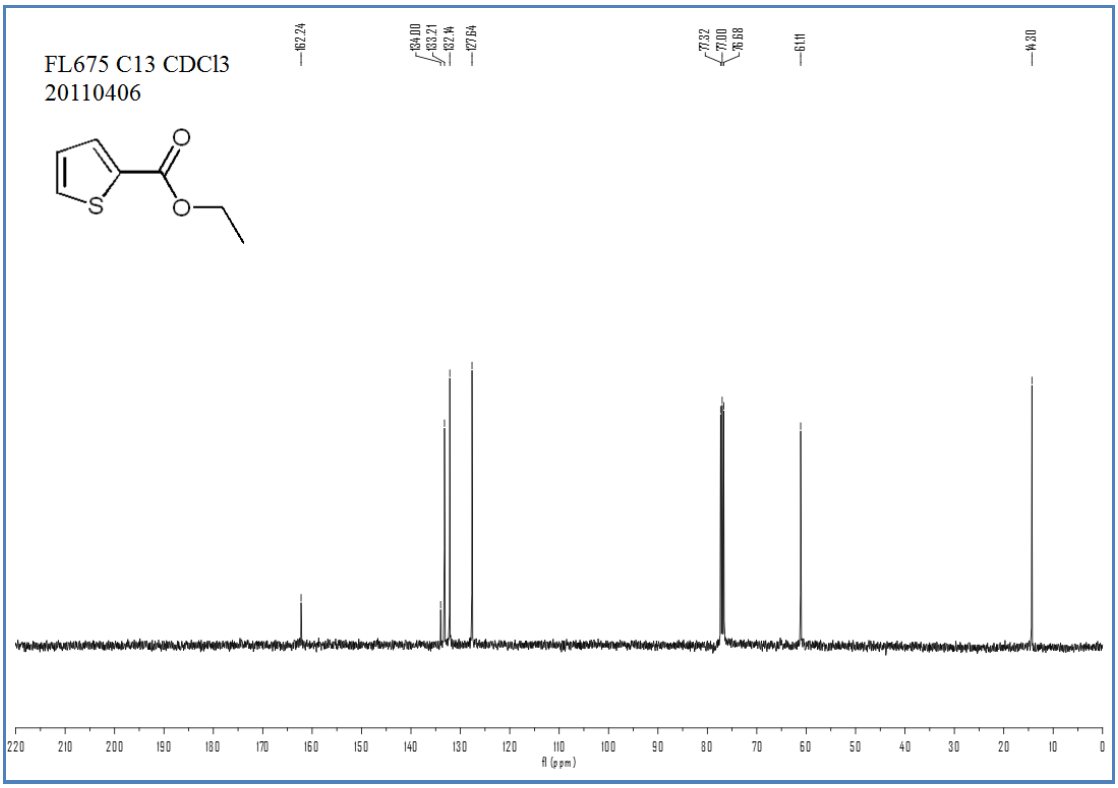
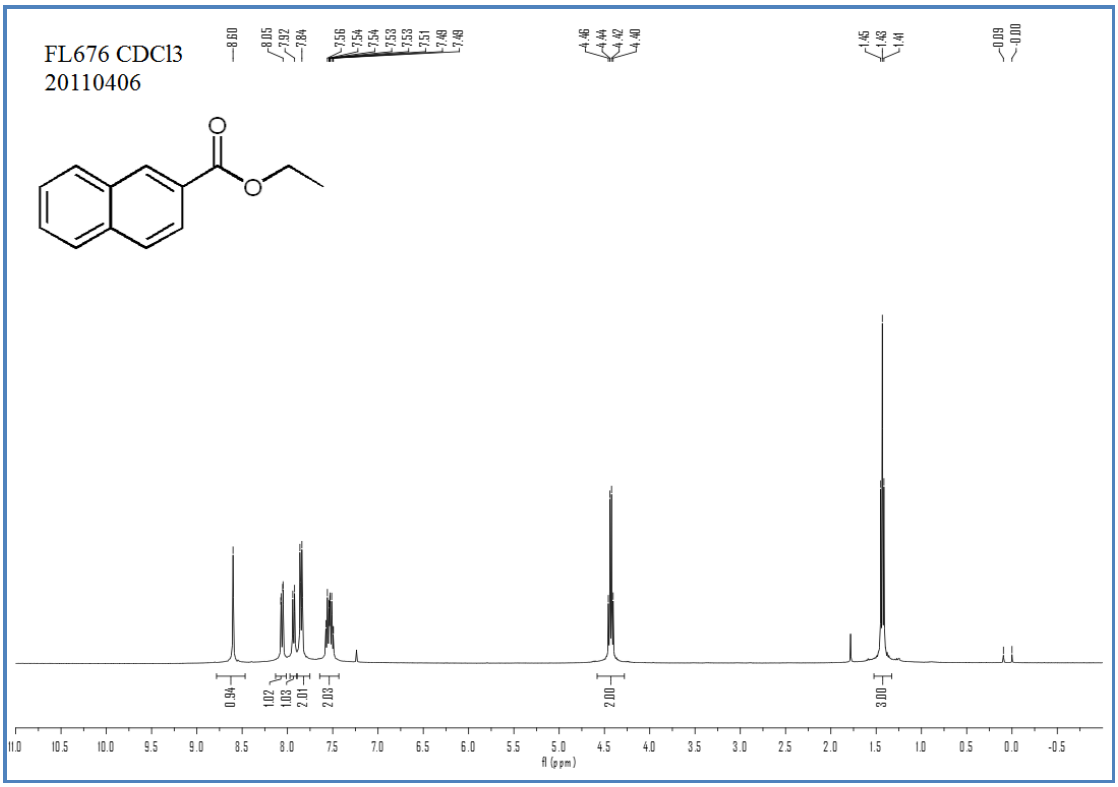
f1 (ppm)



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7.54
7.53
7.53
7.51
7.49
7.49
4.46
4.44
4.42
4.40
1.45
1.43
1.41
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0.00
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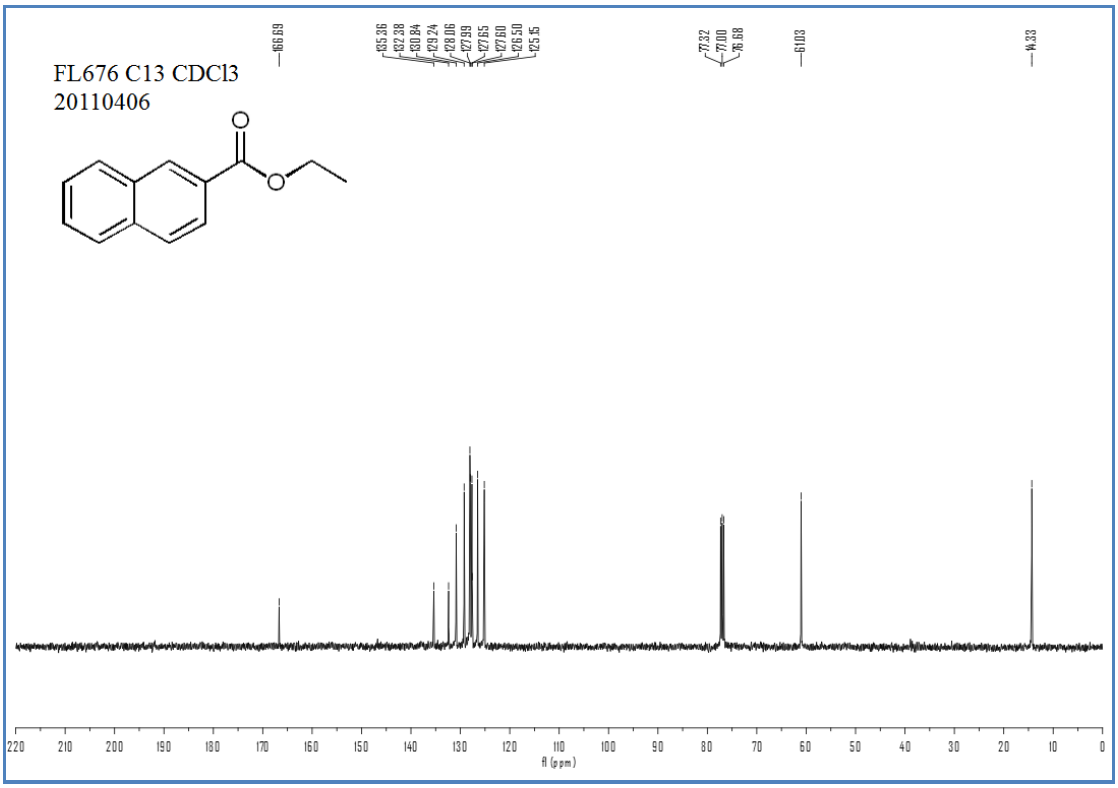
1.02
1.03
2.01
2.03
2.00
3.00
f1 (ppm)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135.36
132.38
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129.24
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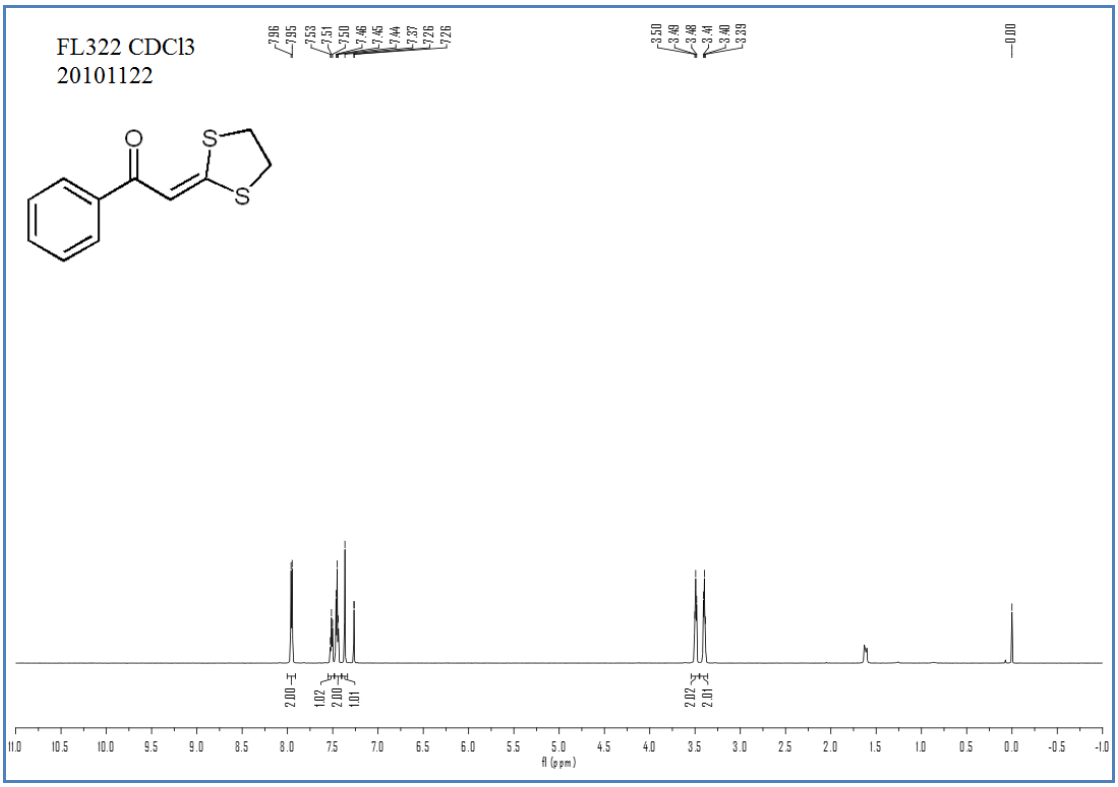
77.00
76.68
61.03
14.33
f1 (ppm)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7.44
7.37
7.26
7.26
3.50
3.49
3.48
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2.00
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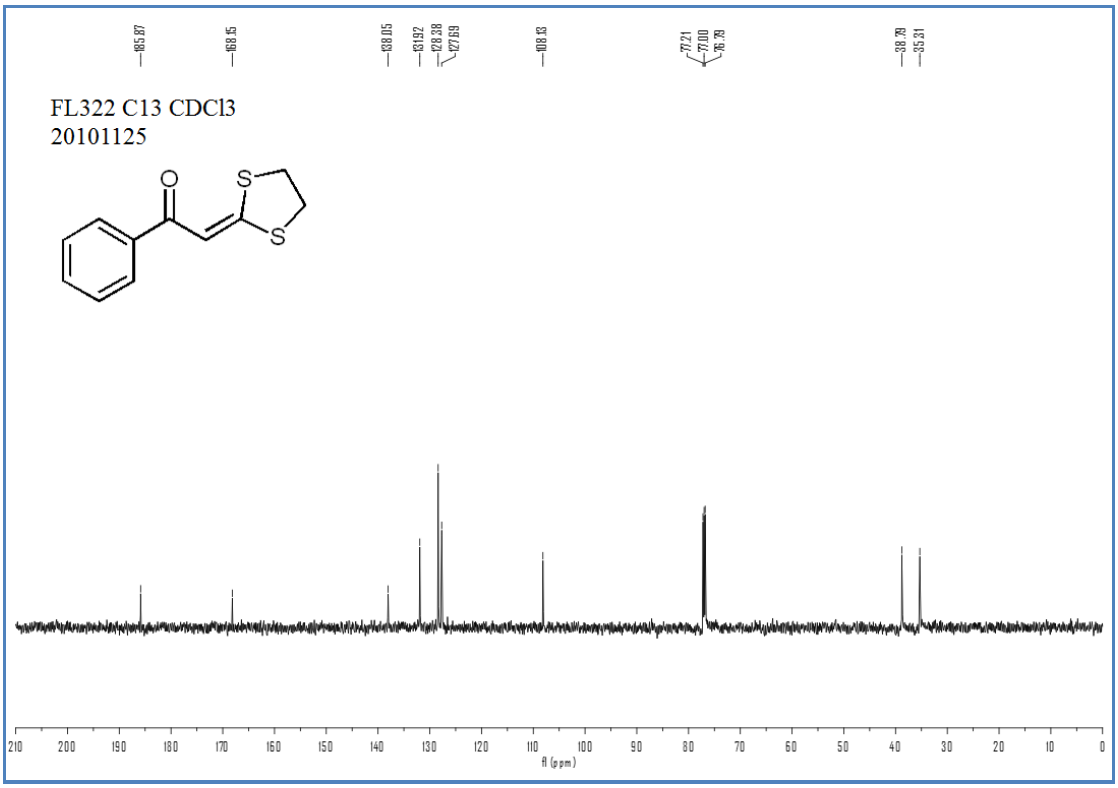
2.02
2.01
f1 (ppm)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127.69
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76.79
38.78
35.31
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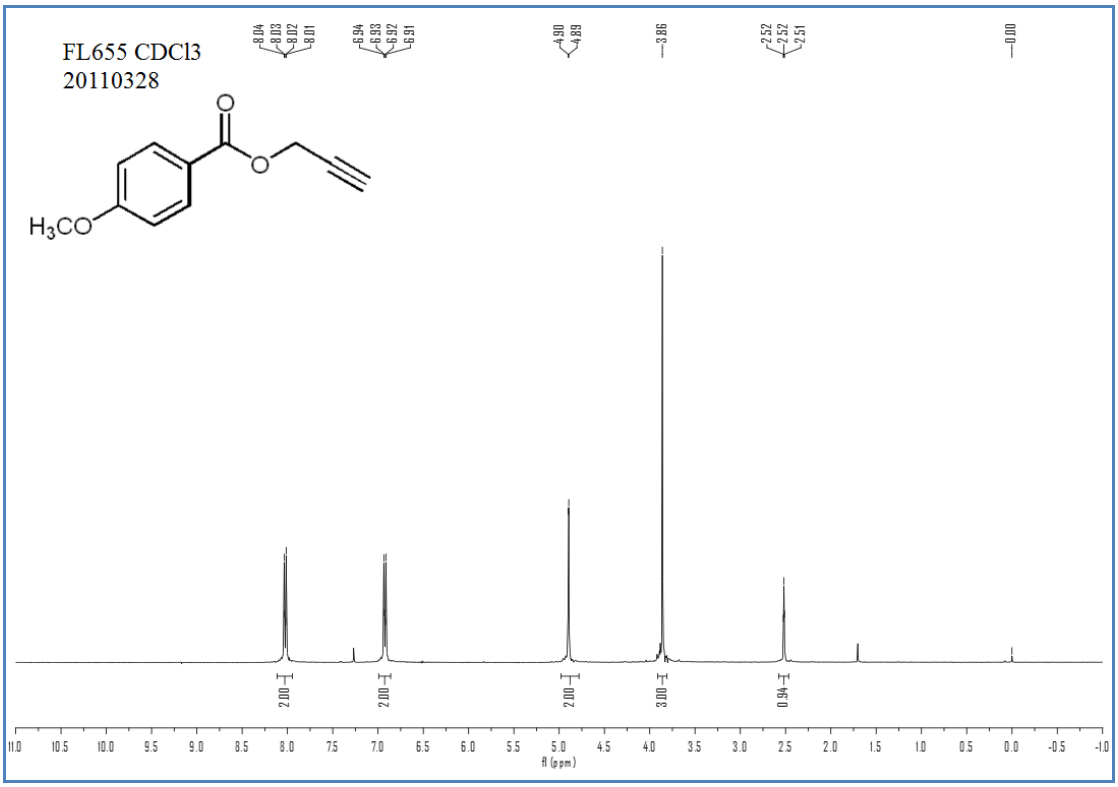
20101125
f1 (ppm)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8.02
8.01
6.94
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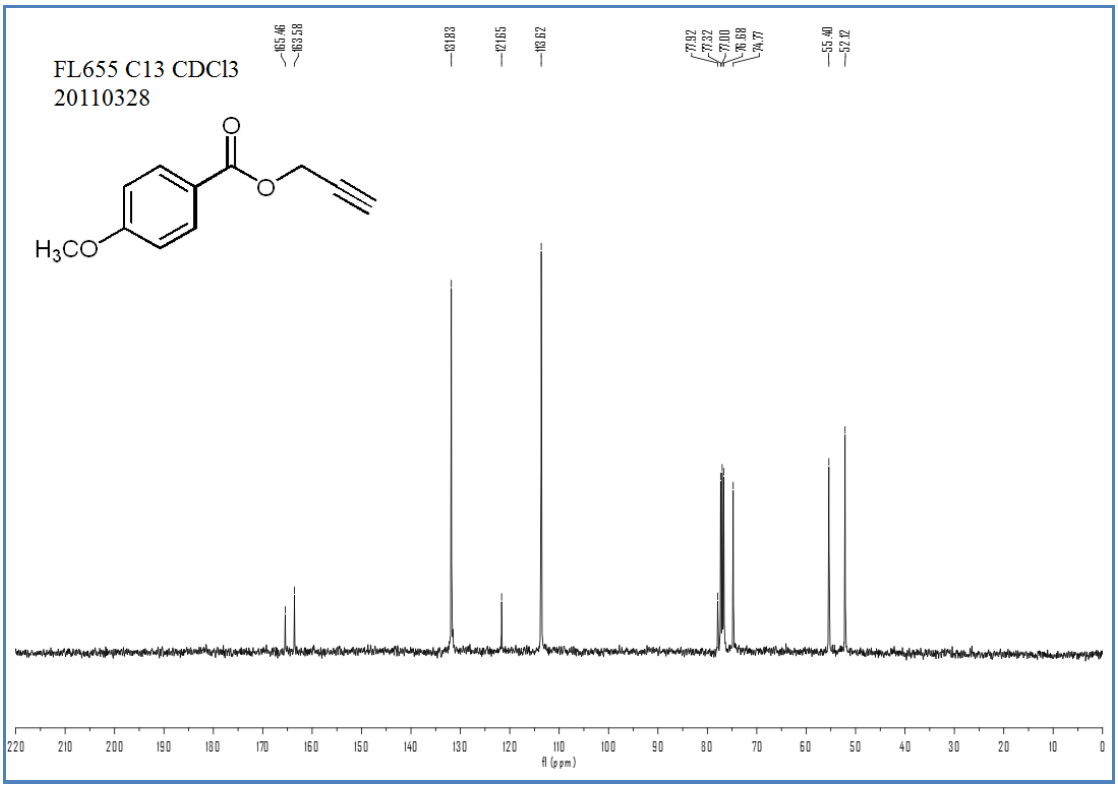
f1 (ppm)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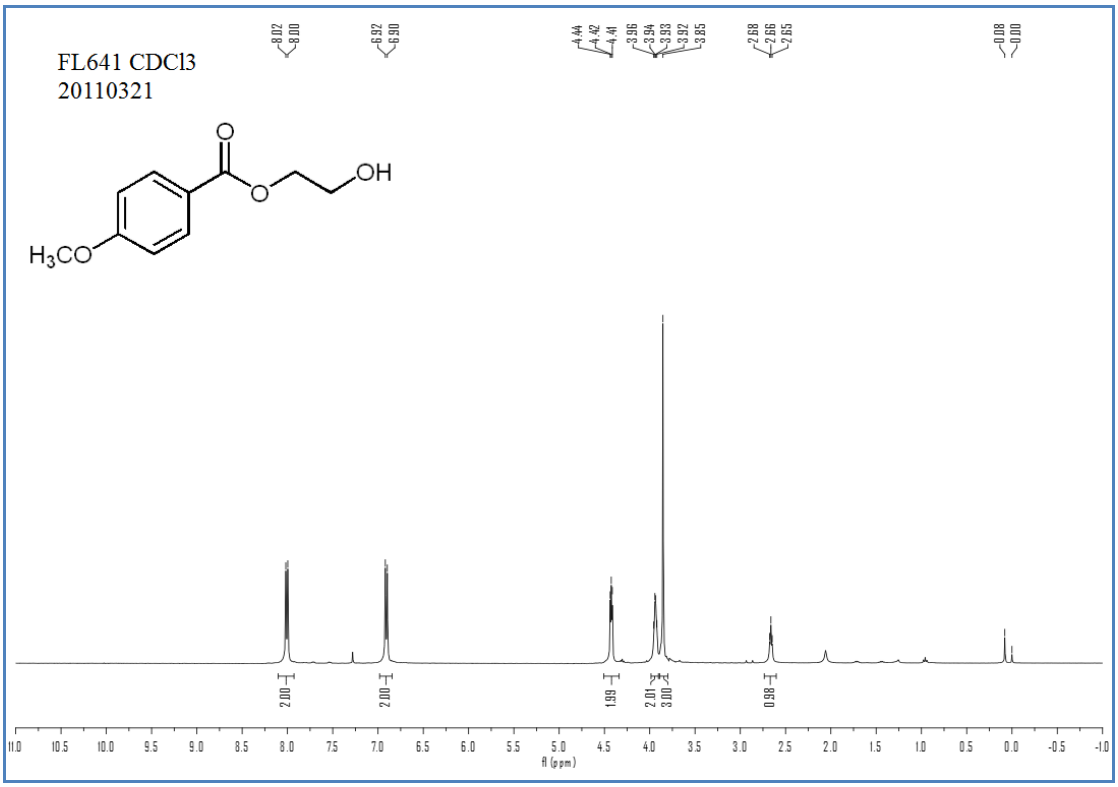
f1 (ppm)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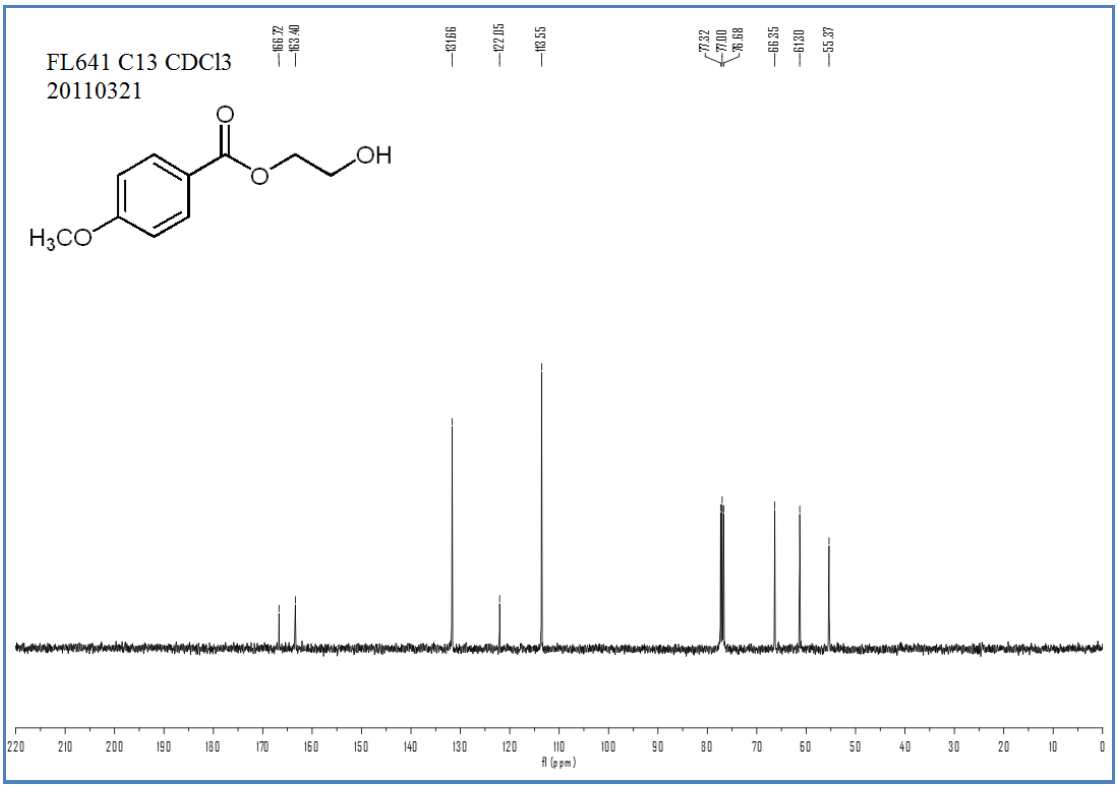
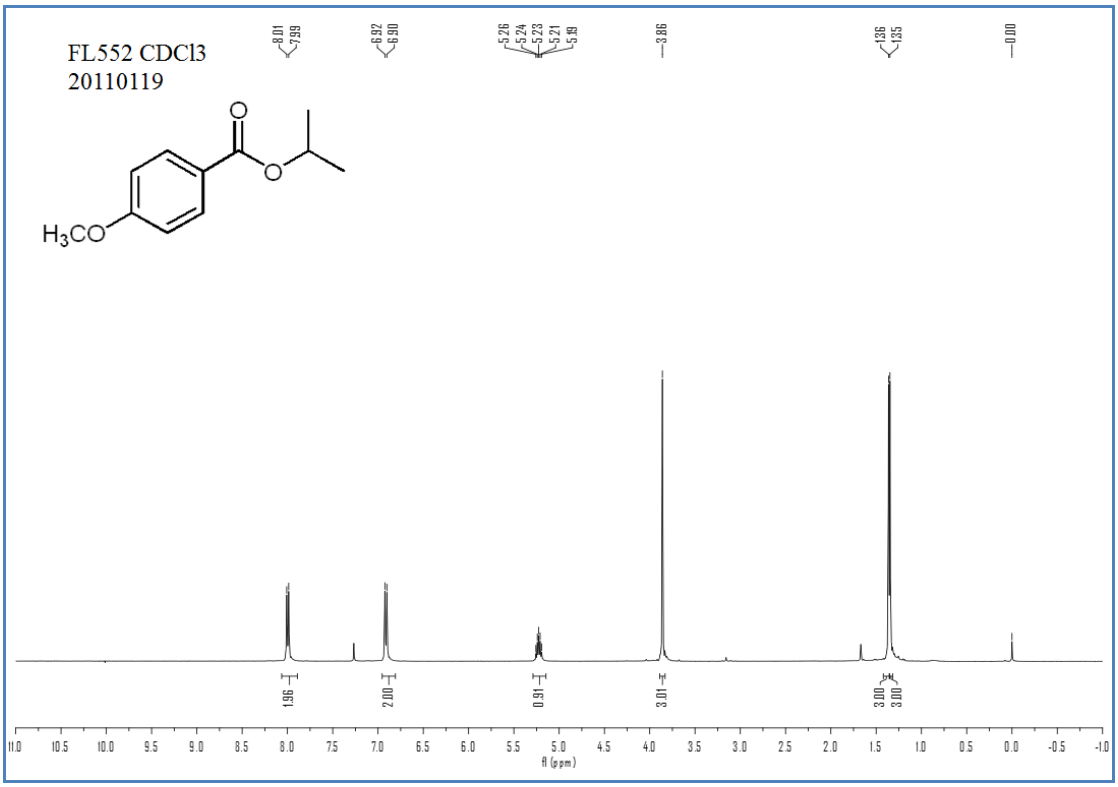
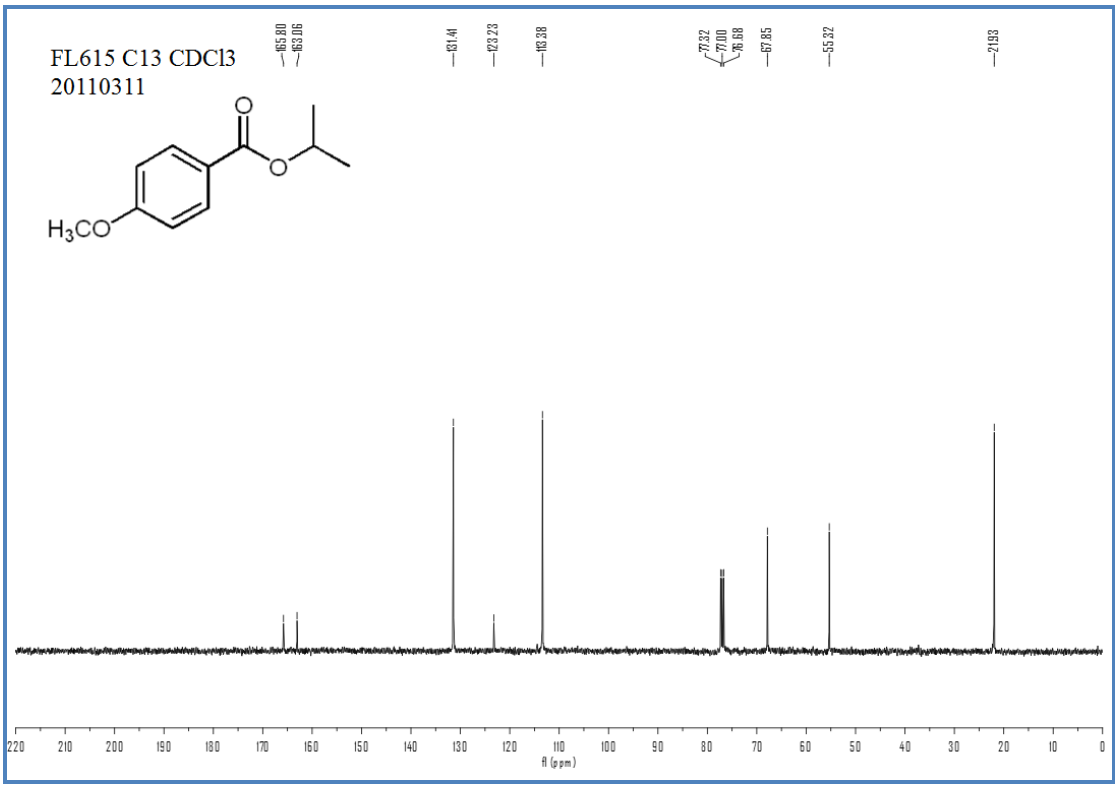
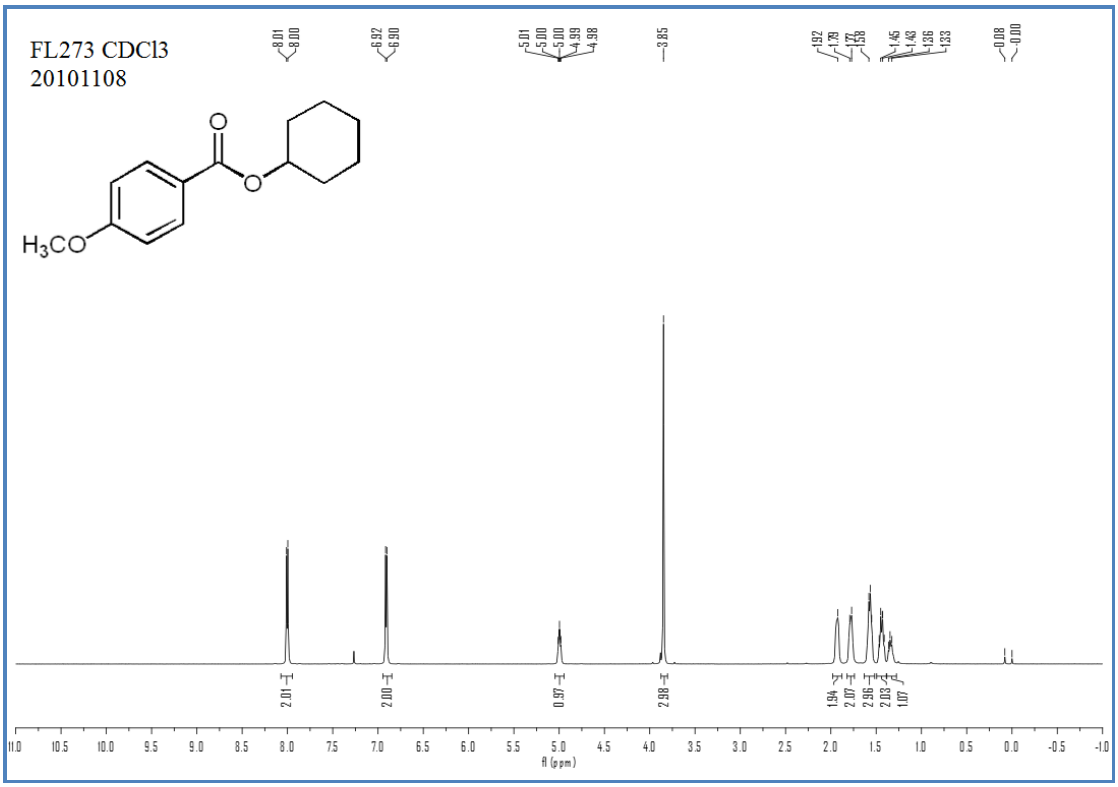
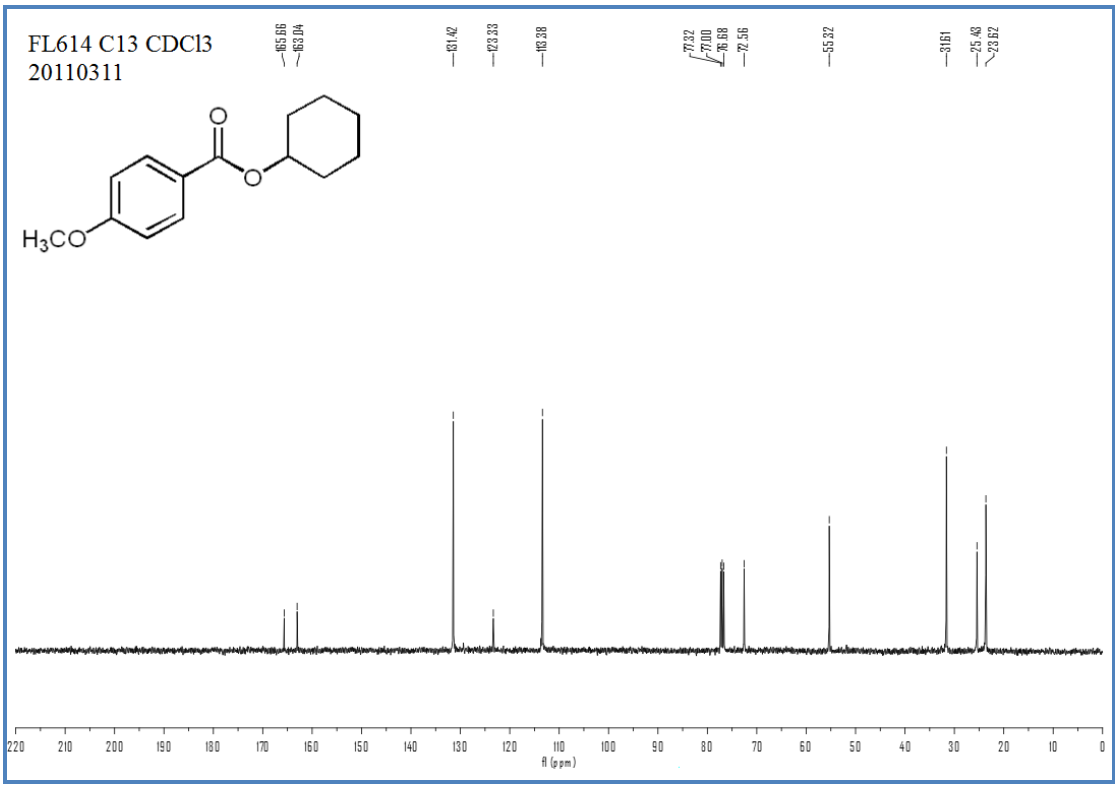
4.41
3.96
3.94
3.93
3.92
3.85
2.68
2.66
2.65
0.08
0.00
2.00
2.00
1.99
2.01
3.00
0.98
f1 (ppm)

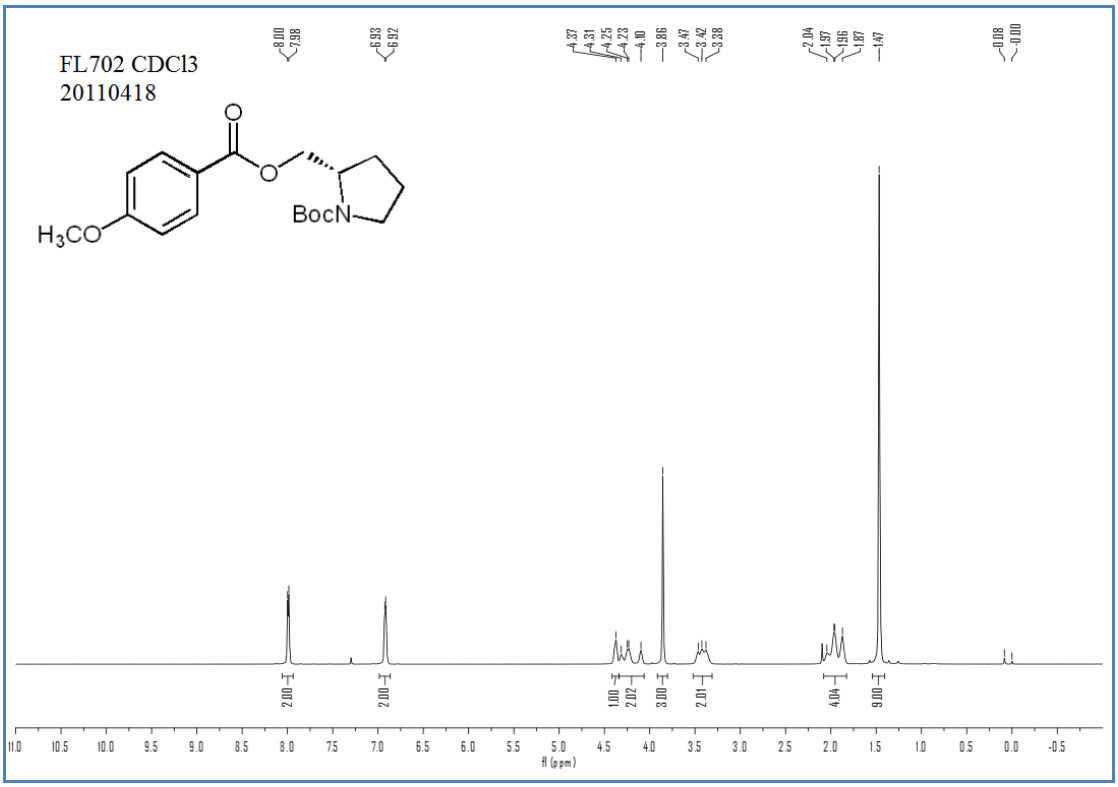

FL641 C13 CDCl3
20110321
OH
H3CO
166.72
163.40
131.66
122.05
113.55
77.32
77.00
76.68
66.35
61.30
55.37
f1 (ppm)

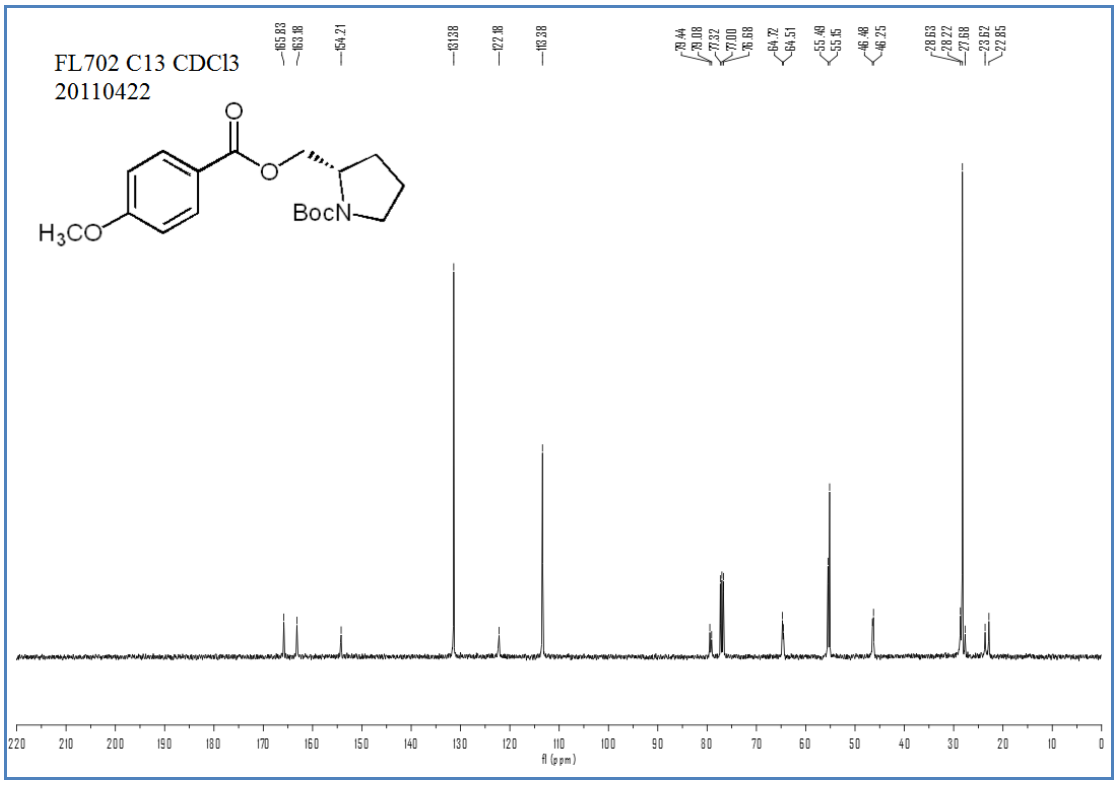


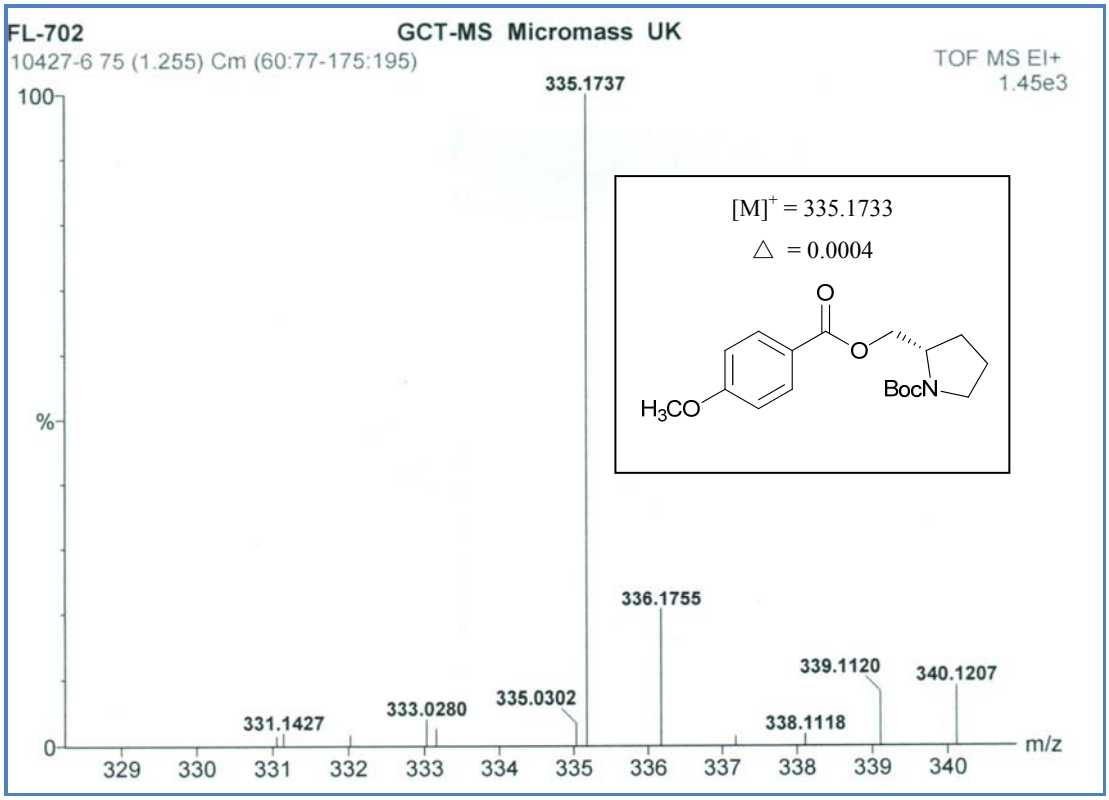


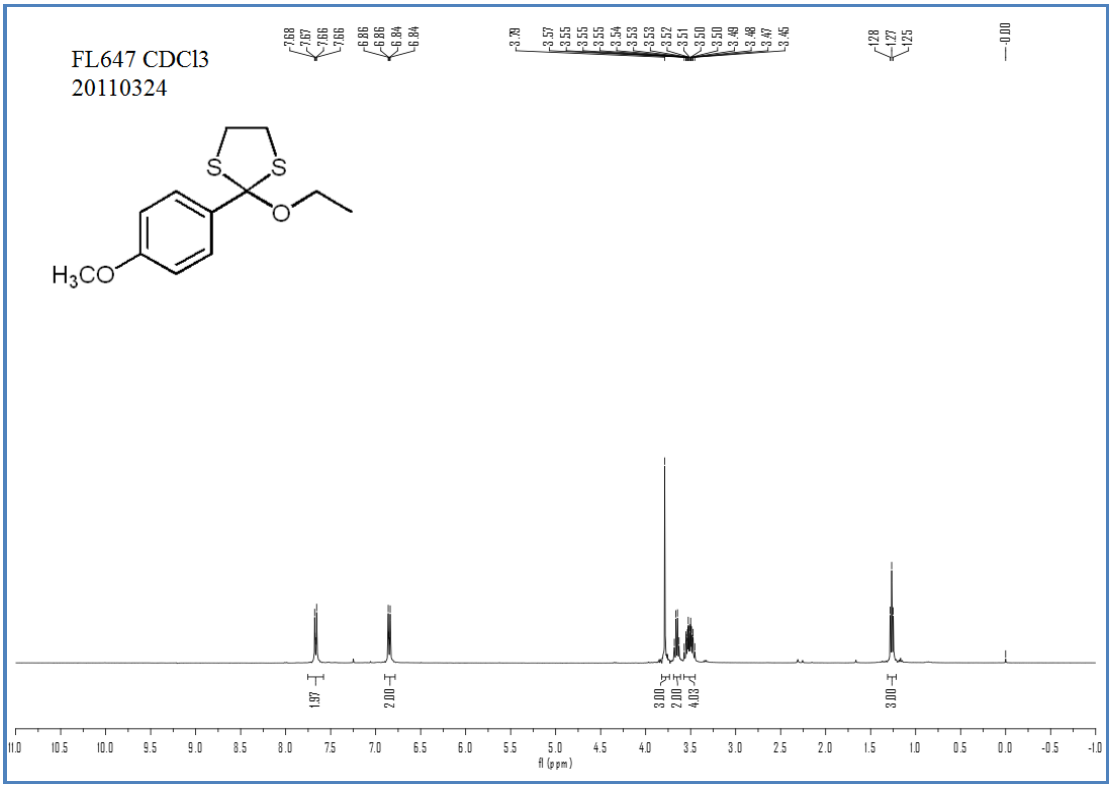

FL273 CDCl3
20101108
H3CO
f1 (ppm)

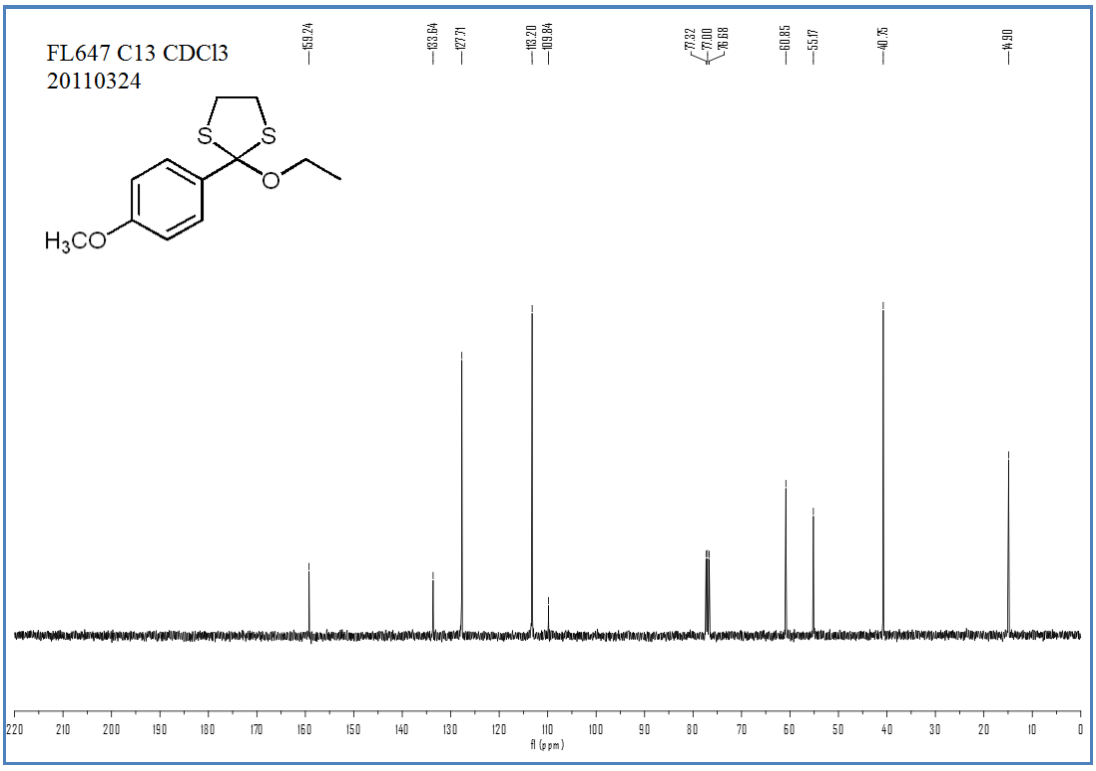

FL614 C13 CDCl3
20110311
H3CO
f1 (ppm)

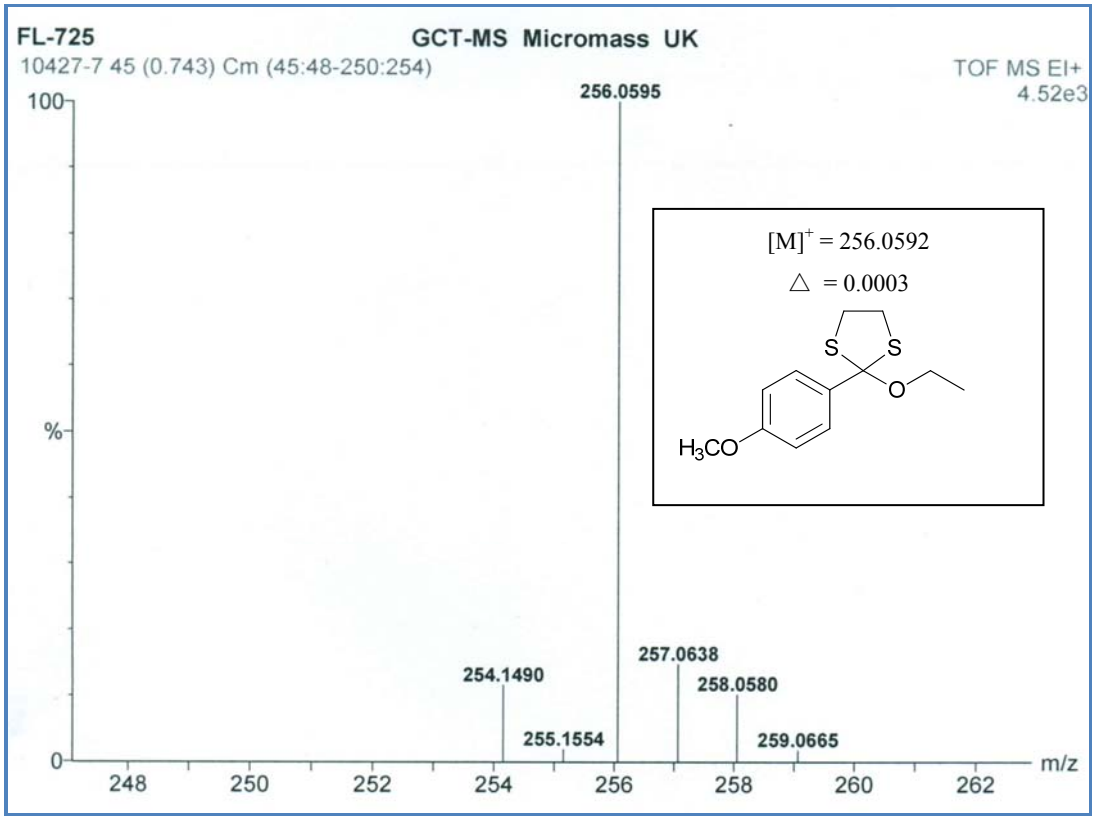

FL702 CDCl3
20110418
H3CO
BocN
8.00
7.98
6.93
6.92
4.37
4.31
4.25
4.23
4.10
3.86
3.47
3.42
3.38
2.04
1.97
1.96
1.87
1.47
0.08
0.00
2.00
2.00
1.00
2.02
3.00
2.01
4.04
9.00
f1 (ppm)


FL702 C13 CDCl3
20110422
H3CO
BocN
165.83
163.38
154.21
131.38
122.18
113.38
79.44
79.08
77.32
77.00
76.68
64.72
64.51
55.48
55.15
46.48
46.25
28.63
28.22
27.68
23.62
22.85
f1 (ppm)


FL-702
GCT-MS Micromass UK
10427-6 75 (1.255) Cm (60:77-175:195)
TOF MS EI+
1.45e3
100
%
0
335.1737
336.1755
339.1120
340.1207
331.1427
333.0280
335.0302
338.1118
329 330 331 332 333 334 335 336 337 338 339 340
m/z
[M]+ = 335.1733
△ = 0.0004
O
O
H3CO
BocN


FL647 CDCl3
20110324
S
S
O
H3CO
1.97
2.00
3.00
2.00
4.03
3.00
f1 (ppm)


FL647 C13 CDCl3
20110324
159.24
133.64
127.71
113.20
109.84
77.32
77.00
76.68
60.85
55.17
40.75
14.90
f1 (ppm)


FL-725
GCT-MS Micromass UK
10427-7 45 (0.743) Cm (45:48-250:254)
TOF MS EI+
4.52e3
256.0595
[M]+ = 256.0592
△ = 0.0003
254.1490
255.1554
257.0638
258.0580
259.0665
m/z