Highly Enantioselective α-Chlorination of Cyclic β-Ketoesters Catalyzed by *N,N'*-Dioxide Using NCS as the Chlorine Source

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Supporting Information

Contents of the supporting information:

1. General remarks

Reactions were carried out using commercial available reagents in over-dried apparatus. Toluene, Et_2O and THF were dried and distilled from sodium benzophenone under nitrogen before use. CH_2Cl_2 was dried over powdered CaH_2 and distilled under nitrogen just before use. m-Xylene and mesitylene was directly distilled before use. Enantiomeric excesses (*ee*) were determined by HPLC analysis using the corresponding commercial chiral column as stated in the experimental procedures at 23 ^{o}C with UV detector at 254 nm or GC. Optical rotations were reported as follows: $[\alpha]_D^{25}$ (c g/100 mL, in solvent). ^{1}H NMR spectra were recorded on commercial instruments (400 MHz). Chemical shifts were reported in ppm from tetramethylsilane with the solvent resonance as the internal standard ($CDCl_3$, δ = 7.26). Spectra were reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants (Hz), integration and assignment. ^{13}C NMR spectra were collected on commercial instruments (100 MHz) with complete proton decoupling. Chemical shifts are reported in ppm from the tetramethylsilane with the solvent resonance as internal standard ($CDCl_3$, δ = 77.0). HRMS was recorded on a commercial apparatus (ESI Source).

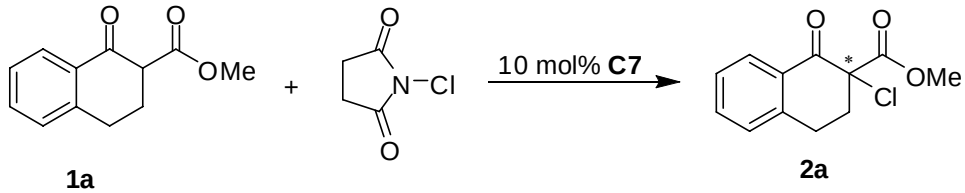
Commercial available NCS (AR) was used without further purification. β-ketoesters **1a-1b,**[1] **1c-1f,**[2] *N,N'*-dioxide catalysts **C1-C11**[3a,b] and racemic samples of **2a-2o**[4] were prepared according to the reported procedure. β-ketoesters **1g-1k** were prepared by analogy to the preparation of **1a-1b**. β-ketoesters **1l-1o** were prepared by analogy to the preparation of **1c-1f**.

2. Typical procedure for the enantioselective α-chlorination of NCS to β-ketoester

N,N'-dioxide **C7** (3.3 mg, 0.005 mmol), β-ketoester **1a** (20.4 mg, 0.10 mmol) were stirred in a reaction tube in toluene (2 mL) at -20 °C for 5 min, then every 30 minutes NCS (14.0 mg, 0.105 mmol) was added separately in four equal lots. The process was monitored by TLC. After β-ketoester **1a** disappeared, the reaction mixture was purified by flash chromatography (petroleum ether : Et_2O = 10 : 1) on silica gel to afford the desired product.

3. Extra Optimizations and ^{1}H NMR for comparision of cyclic and acyclic β-ketoesters

Table: survey of solvent effects

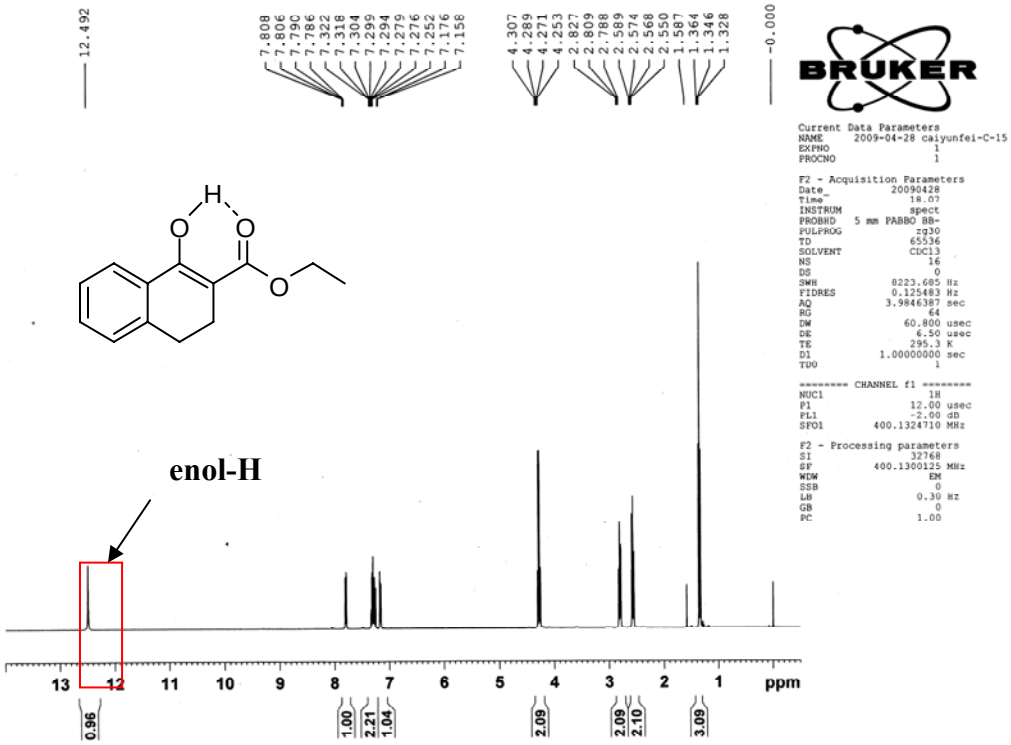


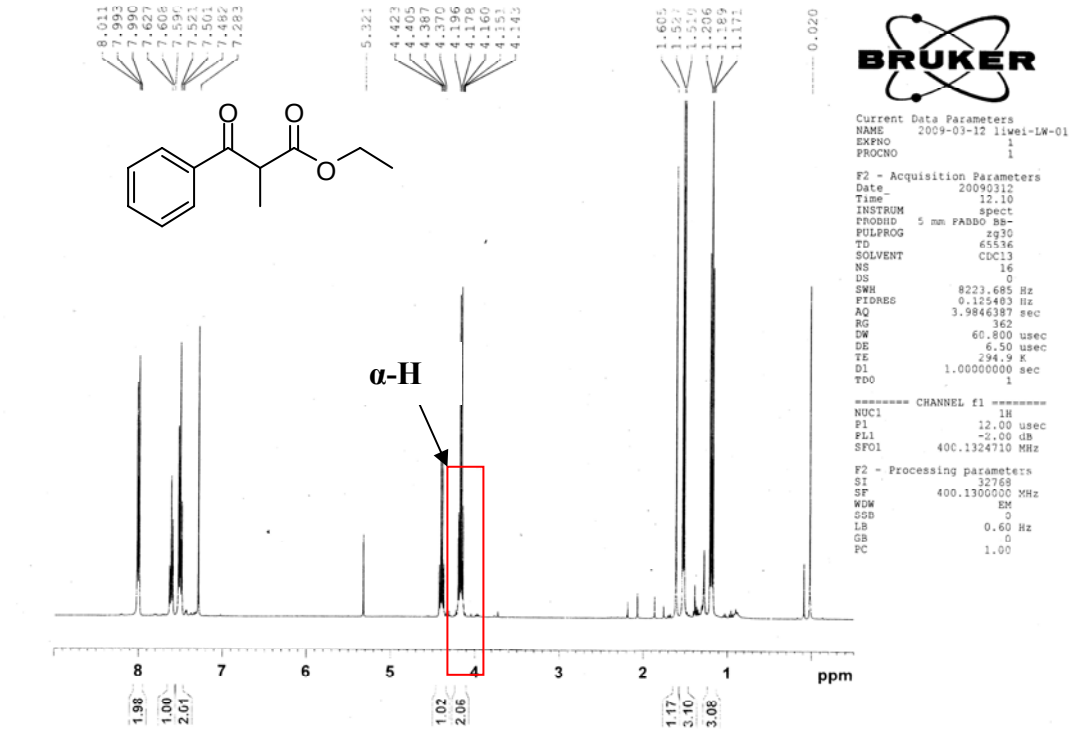
1a **2a**

Entry	Solvent	Yield [%][b]	*ee* [%][c]
1	MeOH	>99	0
2	CH_2Cl_2	>99	48
3	THF	>99	14
4	Et_2O	>99	52
5	m-xylene	>99	79
6	mesitylene	>99	81
7	toluene	>99	85

[a] the reactions were performed with **1a** (0.1 mmol), **C7** (10 mol%), NCS (0.12 mmol) in 0.5 mL solvent at -20 °C for 4 h. [b] Isolated yield. [c] Determined by HPLC analysis (Chiralcel OD-H).

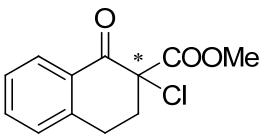
^{1}H NMR for comparision of cyclic and acyclic β-ketoesters





4. Characterization of the products of the α-chlorination of NCS to β-ketoesteres

Methyl 2-chloro-1-oxo-1,2,3,4-tetrahydronaphthalene-2-carboxylate (2a):



2a

Prepared according to the corresponding precedure at -20 ℃ for 4 hours using 5 mol% of *N,N′*-dioxide **C7**.

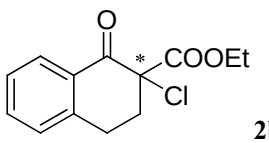
The tittle compound **2a** was purified by silica gel chromatography (petroleum ether : Et_2O = 10 : 1) to afford a colorless oil over 99% yield.

HPLC (Chiralcel OD-H, hexane/ *i*-PrOH = 90/10, flow rate 1.0 mL/min, λ = 254 nm) t_r (major) = 8.37 min, t_r (minor) = 9.85 min, ee = 90%).

^{1}H NMR (400 MHz, $CDCl_3$): δ= 8.08-8.11 (dd, 1H, *J*= 7.9 Hz, 1.3 Hz), 7.52-7.57 (td, 1H, *J*= 7.5 Hz, 1.4 Hz), 7.26-7.39 (m, 2H), 3.86 (s, 3H), 3.25-3.33 (m, 1H), 2.96-3.05 (m, 2H), 2.51-2.57 (m, 1H).

$[\alpha]_D^{25}$ -34.9 (*c* 0.63 in $CHCl_3$).

Ethyl 2-chloro-1-oxo-1,2,3,4-tetrahydronaphthalene-2-carboxylate (2b):



2b

Prepared according to the general precedure at -20 ℃ for 4 hours using 5 mol% of *N,N′*-dioxide **C7**. The tittle

compound **2b** was purified by silica gel chromatography (petroleum ether : Et_2O = 10 : 1) to afford a colorless oil over 99% yield.

HPLC (Chiralcel OD-H, hexane/ *i*-PrOH = 90/10, flow rate 1.0 mL/min, λ = 254 nm) t_r (major) = 7.29 min, t_r (minor) = 8.02 min, ee = 93%).

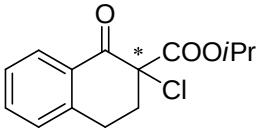
^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.06-8.11 (dd, 1H, *J* = 8.0 Hz, 1.0 Hz), 7.52-7.57 (td, 1H, *J* = 7.2 Hz, 1.2 Hz), 7.26-7.39 (m, 2H), 4.28-4.34 (q, 2H), 3.25-3.33 (m, 1H), 2.96-3.04 (m, 2H), 2.50-2.57 (m, 1H), 1.27-1.31 (t,3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 187.6, 167.4, 142.5, 134.4, 129.7, 128.9, 128.8, 127.3, 70.1, 63.1, 35.0, 25.6, 14.0.

HRMS (ESI-TOF) calcd for $C_{13}H_{13}ClO_3$ ([M]+Na^+) = 275.0445, Found 275.0450.

$[\alpha]_D^{25}$ -27.7 (*c* 0.33 in $CHCl_3$).

Isopropyl 2-chloro-1-oxo-1,2,3,4-tetrahydronaphthalene-2-carboxylate (2c):



2c

Prepared according to the corresponding precedure at -20 ℃ for 4 hours using 5 mol% of *N,N′*-dioxide **C7**.

The tittle compound **2c** was purified by silica gel chromatography (petroleum ether : Et_2O = 10 : 1) to afford a colorless oil in 98% yield.

HPLC (Chiralcel AS-H, hexane/ *i*-PrOH = 98/2, flow rate 1.0 mL/min, λ = 254 nm) t_r (minor) = 7.51 min, t_r (major) = 8.15 min, ee = 93%).

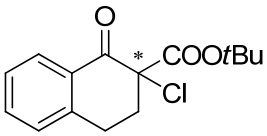
^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.08-8.12 (dd, 1H, *J* = 7.9 Hz, 1.3 Hz), 7.53-7.58 (td, 1H, *J* = 7.5 Hz, 1.4 Hz), 7.36-7.40 (m, 1H), 7.28-7.30 (m, 1H), 5.11-5.21 (m, 1H), 3.26-3.34 (m, 1H), 2.97-3.07 (m, 2H), 2.51-2.57 (m, 1H), 1.30-1.32 (d, 3H, *J* = 6.3 Hz), 1.25-1.27 (d, 3H, *J* = 6.3 Hz).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 187.7, 166.9, 142.5, 134.3, 129.8, 128.9, 128.7, 127.3, 71.10, 35.0, 25.7, 21.5, 21.4.

HRMS (ESI-TOF) calcd for $C_{14}H_{15}ClO_3$ ([M]+Na^+) = 289.0602, Found 289.0601.

$[\alpha]_D^{25}$ -23.9 (*c* 0.27 in $CHCl_3$).

Tert-butyl 2-chloro-1-oxo-1,2,3,4-tetrahydronaphthalene-2-carboxylate (2d):



2d

Prepared according to the corresponding precedure at -20 ℃ for 5 hours using 5 mol% of *N,N′*-dioxide **C7**.

The tittle compound **2d** was purified by silica gel chromatography (petroleum ether : Et_2O = 15 : 1) to afford a colorless oil in 98% yield.

HPLC (Chiralcel OJ-H, hexane/ *i*-PrOH = 98/2, flow rate 1.0 mL/min, λ = 254 nm) t_r (major) = 8.66 min, t_r (minor) = 9.58 min, ee = 95%).

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.09-8.12 (dd, 1H, *J* = 8.0 Hz, 1.3 Hz), 7.53-7.57 (td, 1H, *J* = 7.5 Hz, 1.4 Hz), 7.36-7.40 (m, 1H), 7.27-7.30 (m, 1H), 3.24-3.27 (m, 1H), 2.93-3.07 (m, 2H), 2.50-2.56 (m,1H), 1.48 (s, 9H).

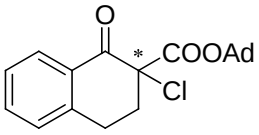
^{13}C NMR (100 MHz, $CDCl_3$): δ = 188.0, 166.2, 142.4, 134.3, 130.1, 128.8, 128.7, 127.2, 84.1, 71.6, 35.3, 27.7,

25.9.

HRMS (ESI-TOF) calcd for $C_{15}H_{17}ClO_3$ ([M]+Na^+) = 303.0758, Found 303.0759.

$[\alpha]_D^{25}$ -13.2 (*c* 0.26 in $CHCl_3$).

1-Adamantyl 2-Chloro-1-oxo-1,2,3,4-tetrahydronaphtalen-2-carboxylate (2e):



2e

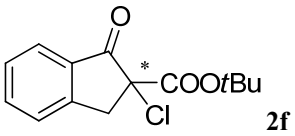
Prepared according to the general precedure at -20 ℃ for 6 hours using 5 mol% of *N,N′*-dioxide **C7**. The tittle compound **2e** was purified by silica gel chromatography (petroleum ether : Et_2O = 15 : 1) to afford a white solid in 98% yield.

HPLC (Chiralcel OJ-H, hexane/ *i*-PrOH = 98/2, flow rate 1.0 mL/min, λ = 254 nm) t_r (major) = 12.92 min, t_r (minor) = 14.37 min, ee = 97%).

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.07-8.10 (dd, 1H, *J* = 8.0 Hz, 1.2 Hz), 7.51-7.55 (td, 1H, *J* = 7.6 Hz, 1.2 Hz), 7.34-7.38 (m, 1H), 7.25-7.27 (m, 1H), 3.20-3.28 (m, 1H), 2.91-3.06 (m, 2H), 2.48-2.54 (m, 1H), 2.16 (s, 3H), 2.10 (s, 6H), 1.64 (s, 6H).

$[\alpha]_D^{25}$ -7.0 (*c* 0.43 in $CHCl_3$).

Tert-butyl 2-chloro-1-oxo-2,3-dihydro-1H-indene-2-carboxylate (2f):



2f

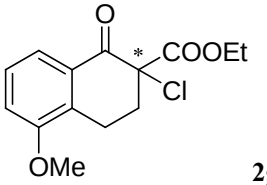
Prepared according to the corresponding precedure at -78 ℃ for 48 hours using 5 mol% of *N,N′*-dioxide **C7**. The tittle compound **2f** was purified by silica gel chromatography (petroleum ether : Et_2O = 10 : 1) to afford a white solid in 60% yield.

HPLC (Chiralcel OJ-H, hexane/ *i*-PrOH = 90/10, flow rate 1.0 mL/min, λ = 254 nm) t_r (major) = 8.86 min, t_r (minor) = 11.76 min, ee = 98%).

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.85-7.87 (d, 1H, *J* = 7.6 Hz), 7.68-7.71 (m, 1H), 7.44-7.49 (m, 2H), 4.00-4.04 (d, 1H, *J* = 17.7), 3.51-3.56 (d, 1H, *J* = 17.7), 1.43 (s, 9H).

$[\alpha]_D^{25}$ -15.0 (*c* 0.04 in $CHCl_3$).

Ethyl 2-chloro-5-methoxy-1-oxo-1,2,3,4-tetrahydronaphthalene-2-carboxylate (2g):



2g

Prepared according to the corresponding precedure at -20 ℃ for 4 hours using 5 mol% of *N,N′*-dioxide **C7**. The tittle compound **2g** was purified by silica gel chromatography (petroleum ether : Et_2O = 10 : 1) to afford a white solid in 99% yield.

HPLC (Chiralcel OJ-H, hexane/ *i*-PrOH = 70/30, flow rate 1.0 mL/min, λ = 254 nm) t_r (major) = 10.70 min, t_r

(minor) = 8.28 min, ee = 91%).

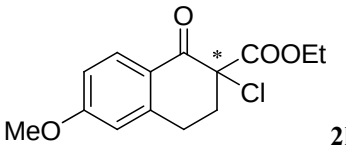
^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.68-7.70 (dd, 1H, *J* = 8.0 Hz, 1.0 Hz), 7.33 (t, 1H, *J* = 8.0 Hz), 7.05-7.08 (dd, 1H, *J* = 8.1 Hz, 0.9 Hz), 4.27-4.33 (q, 2H), 3.88 (s, 3H), 3.02-3.06 (m, 1H), 2.94-2.99 (m, 2H), 2.51-2.55 (m, 1H), 1.26-1.30 (t, 3H, *J* = 7.1 Hz).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 187.9, 167.4, 156.5, 131.6, 130.7, 127.5, 120.3, 115.0, 70.7, 63.0, 55.7, 34.3, 19.7, 13.9.

HRMS (ESI-TOF) calcd for $C_{14}H_{15}ClO_4$ ([M]+Na^+) = 305.0551, Found 305.0565.

$[\alpha]_D^{25}$ -16.4 (*c* 0.33 in $CHCl_3$).

Ethyl 2-chloro-6-methoxy-1-oxo-1,2,3,4-tetrahydronaphthalene-2-carboxylate (2h):



2h

Prepared according to the corresponding precedure at -20 ^{0}C for 8 hours using 5 mol% of *N,N'*-dioxide **C7**. The tittle compound **2h** was purified by silica gel chromatography (petroleum ether : Et_2O = 5 : 1) to afford a white solid in 95% yield.

HPLC (Chiralcel OD-H, hexane/ *i*-PrOH = 90/10, flow rate 1.0 mL/min, λ = 254 nm) t_r (major) = 10.59 min, t_r (minor) = 12.13 min, ee = 95%).

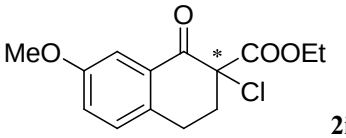
^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.07 (d, 1H, *J* = 8.8 Hz), 6.86-6.90 (dd, 1H, *J* = 8.8 Hz, 2.5 Hz), 6.71 (d, 1H, *J* = 2.5 Hz), 4.28-4.34 (q, 2H), 3.87 (s, 3H), 3.22-3.29 (m, 1H), 2.93-3.01 (m, 2H), 2.46-2.53 (m, 1H), 1.28-1.32 (t, 3H, *J* = 7.1 Hz).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 186.4, 167.7, 164.4, 145.2, 131.6, 123.0, 114.1, 112.5, 70.8, 63.0, 55.6, 35.2, 25.9, 14.0.

HRMS (ESI-TOF) calcd for $C_{14}H_{15}ClO_4$ ([M]+Na^+) = 305.0551, Found 305.0552.

$[\alpha]_D^{25}$ -33.6 (*c* 0.30 in $CHCl_3$).

Ethyl 2-chloro-7-methoxy-1-oxo-1,2,3,4-tetrahydronaphthalene-2-carboxylate (2i):



2i

Prepared according to the general precedure at -20 ^{0}C for 4 hours using 5 mol% of *N,N'*-dioxide **C7**. The tittle compound **2i** was purified by silica gel chromatography (petroleum ether : Et_2O = 10 : 1) to afford a colorless oil in 99% yield.

HPLC (Chiralcel OD-H, hexane/ *i*-PrOH = 90/10, flow rate 1.0 mL/min, λ = 254 nm) t_r (major) = 7.34 min, t_r (minor) = 7.94 min, ee = 91%).

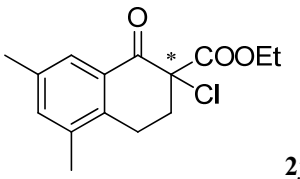
^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.55 (d, 1H, *J* = 2.76 Hz), 7.10-7.20 (m, 2H), 4.28-4.34 (q, 2H), 3.84 (s, 3H), 3.17-3.25 (m, 1H), 2.91-3.01 (m, 2H), 2.48-2.55 (m, 1H), 1.28-1.32 (t, 3H, *J* = 7.1 Hz).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 187.7, 167.5, 158.7, 135.2, 130.4, 130.0, 123.1, 110.5, 70.8, 63.1, 55.5, 35.3, 24.9, 14.0.

HRMS (ESI-TOF) calcd for $C_{14}H_{15}ClO_4$ ([M]+Na^+) = 305.0551, Found 305.0546.

$[\alpha]_D^{25}$ -29.9 (*c* 0.27 in $CHCl_3$).

Ethyl 2-chloro-5,7-dimethyl-1-oxo-1,2,3,4-tetrahydronaphthalene-2-carboxylate (2j):



2j

Prepared according to the general precedure at -20 ^{0}C for 4 hours using 5 mol% of *N,N'*-dioxide **C7**. The tittle compound **2j** was purified by silica gel chromatography (petroleum ether : Et_2O = 10 : 1) to afford a colorless over 99% yield.

HPLC (Chiralcel OJ-H, hexane/ *i*-PrOH = 95/5, flow rate 1.0 mL/min, λ = 254 nm) t_r (major) = 9.99 min, t_r (minor) = 10.97 min, ee = 91%).

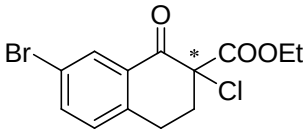
^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.77 (s, 1H), 7.24 (s, 1H), 4.27-4.33 (m, 2H), 2.89-3.04 (m, 3H), 2.51-2.58 (m, 1H), 2.34 (s, 3H), 2.28 (s, 3H), 1.27-1.30 (t, 3H, *J* = 7.2 Hz).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 188.3, 167.5, 137.9, 136.9, 136.5, 136.3, 129.7, 126.8, 70.6, 63.0, 34.3, 22.8, 20.8, 19.2, 13.9.

HRMS (ESI-TOF) calcd for $C_{15}H_{17}ClO_3$ ([M]+Na^+) = 303.0758, Found 303.0756.

$[\alpha]_D^{25}$ -12.3 (*c* 0.24 in $CHCl_3$).

Ethyl 7-bromo-2-chloro-1-oxo-1,2,3,4-tetrahydronaphthalene-2-carboxylate (2k):



2k

Prepared according to the general precedure at -78 ^{0}C for 10 hours using 5 mol% of *N,N'*-dioxide **C7**. The tittle compound **2k** was purified by silica gel chromatography (petroleum ether : Et_2O = 10 : 1) to afford a white solid in 98% yield.

HPLC (Chiralcel OJ-H, hexane/ *i*-PrOH = 90/10, flow rate 1.0 mL/min, λ = 254 nm) t_r (major) = 14.92 min, t_r (minor) = 16.60 min, ee = 93%).

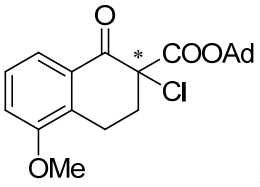
^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.20 (d, 1H, *J* = 2.1Hz), 7.63-7.66 (dd, 1H, *J* = 5.2Hz, 2.3Hz), 7.18 (d, 1H, 8.2 Hz), 4.30-4.34 (m, 2H), 3.17-3.26 (m, 1H), 2.94-3.01 (m, 2H), 2.50-2.56 (m, 1H), 1.28-1.32 (t, 3H, *J* = 7.2 Hz).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 186.4, 167.1, 141.2, 137.2, 131.6, 131.2, 130.6, 121.2, 70.6, 63.3, 34.8, 25.2, 14.0.

HRMS (ESI-TOF) calcd for $C_{13}H_{12}BrClO_3$ ([M]+H^+) = 330.9731, Found 330.9735.

$[\alpha]_D^{25}$ -28.3 (*c* = 0.85 in $CHCl_3$).

1-Adamantyl 2-chloro-5-methoxy-1-oxo-1,2,3,4-tetrahydronaphthalene-2-carboxylate (2l):



2l

Prepared according to the general precedure at -20 ℃ for 6 hours using 5 mol% of *N,N'*-dioxide **C7**. The tittle compound **2l** was purified by silica gel chromatography (petroleum ether : Et_2O = 15 : 1) to afford a white solid in 96% yield.

HPLC (Chiralcel OJ-H, hexane/ *i*-PrOH = 90/10, flow rate 1.0 mL/min, λ = 254 nm) t_r (major) = 11.89 min, t_r

(minor) = 10.62 min, ee = 94%).

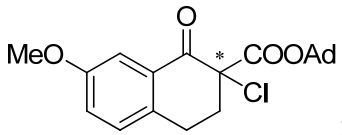
^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.67-7.69 (d, 1H, *J* = 7.6 Hz), 7.30-7.34 (t, 1H, *J* = 8.0 Hz), 7.05-7.07 (d, 1H, *J* = 8.0 Hz), 3.88 (s, 3H), 3.00-3.04 (m, 2H), 2.88-2.94 (m, 1H), 2.46-2.52 (m, 1H), 2.09-2.16 (m, 9H), 1.62-1.64 (m, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 188.3, 165.8, 156.5, 131.5, 131.2, 127.5, 120.1, 114.7, 84.1, 71.6, 55.7, 40.9, 36.0, 34.7, 30.9, 20.6.

HRMS (ESI-TOF) calcd for $C_{22}H_{25}ClO_4$ ([M]+Na^+) = 411.1334, Found 411.1234.

$[\alpha]_D^{25}$ -4.8 (*c* 0.45 in $CHCl_3$).

1-Adamantyl 2-chloro-7-methoxy-1-oxo-1,2,3,4-tetrahydronaphthalene-2-carboxylate (2m):



2m

Prepared according to the general precedure at -20 ℃ for 6 hours using 5 mol% of *N,N′*-dioxide **C7**. The tittle compound **2m** was purified by silica gel chromatography (petroleum ether : Et_2O = 15 : 1) to afford a white solid in 98% yield.

HPLC (Chiralcel OD-H, hexane/ *i*-PrOH = 90/10, flow rate 1.0 mL/min, λ = 254 nm) t_r (major) = 5.76 min, t_r (minor) = 6.66 min, ee = 94%).

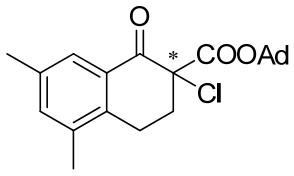
^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.54-7.55 (d, 1H, *J* = 2.8 Hz), 7.09-7.18 (m, 2H), 3.84 (s, 1H), 3.12-36.20 (m, 1H), 2.88-2.99 (m, 2H), 2.46-2.53 (m, 1H), 2.10-2.17 (m, 9H), 1.63-1.64 (m, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 188.1, 165.9, 158.7, 135.1, 130.9, 129.9, 122.9, 110.3, 84.2, 71.7, 55.6, 41.0, 36.0, 35.6, 30.9, 25.2.

HRMS (ESI-TOF) calcd for $C_{22}H_{25}ClO_4$ ([M]+Na^+) = 411.1334, Found 411.1337.

$[\alpha]_D^{25}$ 1.7 (*c* 0.47 in $CHCl_3$).

1-Adamantyl 2-chloro-5,7-dimethyl-1-oxo-1,2,3,4-tetrahydronaphthalene-2-carboxylate (2n):



2n

Prepared according to the general precedure at -20 ℃ for 6 hours using 5 mol% of *N,N′*-dioxide **C7**. The tittle compound **2e** was purified by silica gel chromatography (petroleum ether : Et_2O = 15 : 1) to afford a white solid in 99% yield.

HPLC (Chiralcel OD-H, hexane/ *i*-PrOH = 90/10, flow rate 1.0 mL/min, λ = 254 nm) t_r (major) = 5.76 min, t_r (minor) = 6.66 min, ee = 94%).

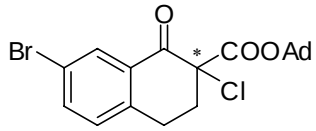
^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.77 (s, 1H), 7.23 (s, 1H), 2.86-3.02 (m, 3H), 2.47-2.53 (m, 1H), 2.34 (s, 3H), 2.17 (s, 3H), 2.12-2.16 (m, 9H), 1.63-1.64 (m, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 188.6, 165.9, 137.9, 136.7, 136.4, 136.3, 130.1, 126.6, 84.0, 71.5, 40.9, 36.0, 34.6, 30.9, 23.1, 20.8, 19.2.

HRMS (ESI-TOF) calcd for $C_{24}H_{27}ClO_3$ ([M]+Na^+) = 409.1541, Found 409.1532.

$[\alpha]_D^{25}$ 1.2 (*c* 0.49 in $CHCl_3$).

1-Adamantyl 7-bromo-2-chloro-1-oxo-1,2,3,4-tetrahydronaphthalene-2-carboxylate (2o):



2o

Prepared according to the general precedure at -78 ℃ for 10 hours using 5 mol% of *N,N'*-dioxide **C7**. The tittle compound **2o** was purified by silica gel chromatography (petroleum ether : Et_2O = 15 : 1) to afford a white solid up to 99% yield.

HPLC (Chiralcel OD-H, hexane/ *i*-PrOH = 90/10, flow rate 1.0 mL/min, λ = 254 nm) t_r (major) = 5.63 min, t_r (minor) = 6.09 min, ee = 86%).

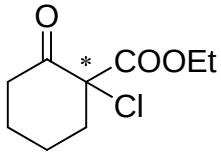
^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.20 (d, 1H, *J* = 2.2 Hz), 7.62-7.64 (dd, 1H, *J* = 8.2Hz, 2.2Hz), 7.15-7.17 (d, 1H, *J* = 8.2 Hz), 3.13-3.21 (m, 1H), 2.88-3.01 (m, 2H), 2.47-2.54 (m, 1H), 2.08-2.17 (m, 9H), 1.63-1.65 (m, 6H)

^{13}C NMR (100 MHz, $CDCl_3$): δ = 186.8, 165.4, 141.1, 136.9, 131.7, 131.4, 130.5, 121.2, 84.5, 71.3, 40.9, 35.9, 35.2, 30.9, 25.6.

HRMS (ESI-TOF) calcd for $C_{21}H_{22}BrClO_3$ ([M]+H^+) = 437.0514, Found 437.0662.

$[\alpha]_D^{25}$ 5.6 (*c* 0.92 in $CHCl_3$).

Ethyl 1-chloro-2-oxocyclohexanecarboxylate



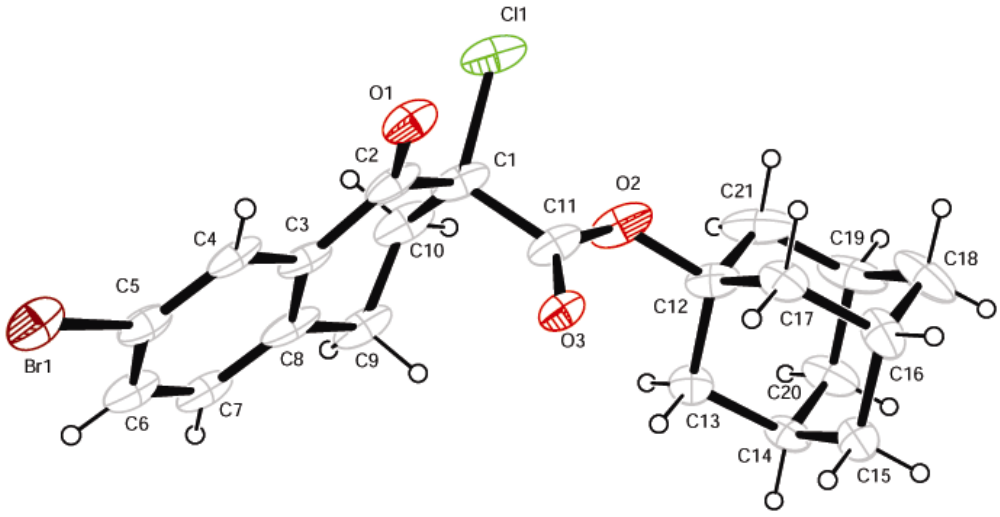
Prepared according to the general precedure at -20 ℃ for 10 hours using 5 mol% of *N,N'*-dioxide **C7**. The tittle compound **2o** was purified by silica gel chromatography (petroleum ether : Et_2O = 15 : 1) to afford a colorless oil in 76% yield.

The ee was determined by GC analysis using a BetaDEX-CB chiral column (T = 115 °C). t_r (major) = 43.41 min, t_r (minor) = 44.69 min, ee = 40%).

^{1}H NMR (400 MHz, $CDCl_3$): δ = 4.28-4.33 (q, 2H), 2.77-2.90 (m, 2H), 2.40-2.48 (m, 1H), 2.11-2.18 (m, 1H), 1.84-2.00 (m, 3H), 1.71-1.80 (m, 1H), 1.30-1.34 (t, 3H, *J* = 7.2 Hz).

$[\alpha]_D^{25}$ -10.5 (*c* 0.36 in $CHCl_3$).

5. The X-ray crystal structure of product 2o



Single crystals of $C_{21}H_{22}BrClO_3$ **2o** were recryatallised from mixed solvents of dichloromethane and petroleum ether. The absolute configuration of C1 is *R*. The thermal ellipsoids' level is 30 % for the above crystal structure.

Crystal data. $C_{21}H_{22}BrClO_3$, M = 437.05, orthorhombic, a = 6.9369(14), b = 7.2436(14), c = 36.835(7) Å, U = 1850.9(6) Å^3, T = 113 K, space group $P2_12_12_1$ (no. 19), Z = 4, 12953 reflections measured, 4334 unique (R_{int} = 0.0468) which used in all calculations. The final $wR(F_2)$ was 0.1510 (all data).

6. References

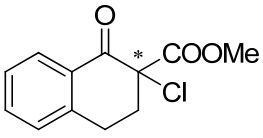
[1] D. S. Brown, B. A. Marples, P. Smith, L. Walton, *Tetrahedron*. 1995, **51**, 3587.

[2] M. Nakajima, S. Yamamoto, Y. Yamaguchi, S. Nakamura, S. Hashimoto, *Tetrahedron.* 2003, **59**, 7307.

[3] a) Y. H. Wen, X. Huang, J. L. Huang, Y. Xiong, B. Qin, X. M. Feng, *Synlett.* 2005, 2445; b) Z. P. Yu, X. H. Liu, Z. H. Dong, M. S. Xie, X. M. Feng, *Angew. Chem. Int. Ed.* 2008, **47**, 1308.

[4] Y. Mei, P. A. Bentley, J. Du, *Tetrahedron lett.* 2008, **49**, 3802.

7. Copy of HPLC Charts, ^{1}H NMR and ^{13}C NMR spectra for α-chlorination products



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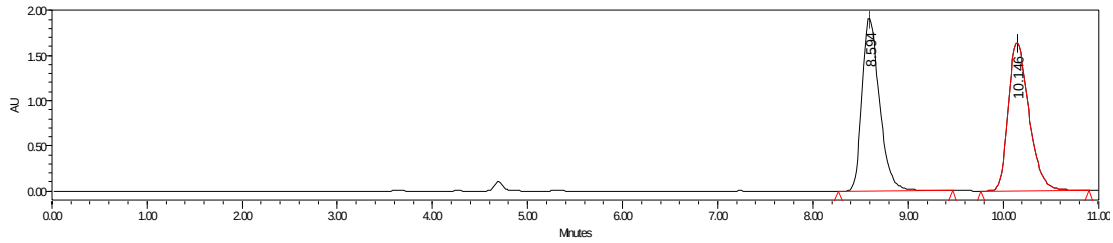

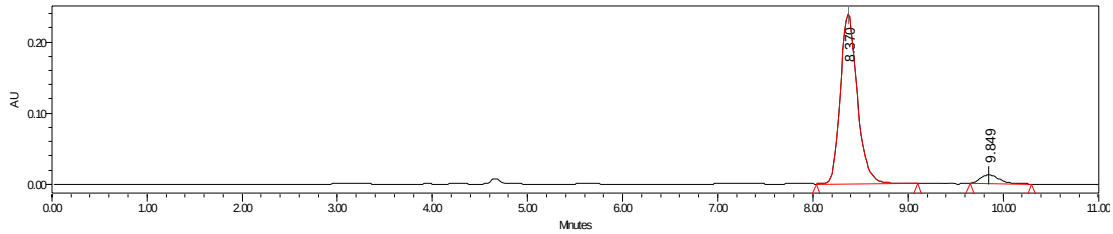

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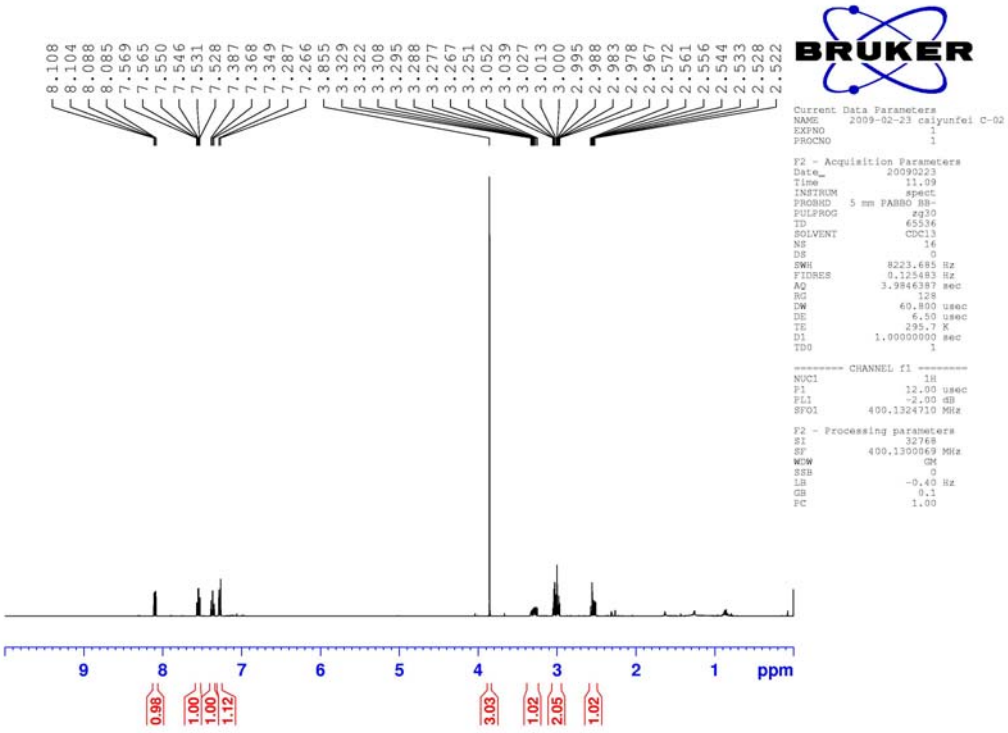

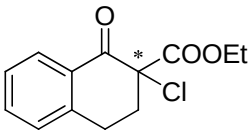

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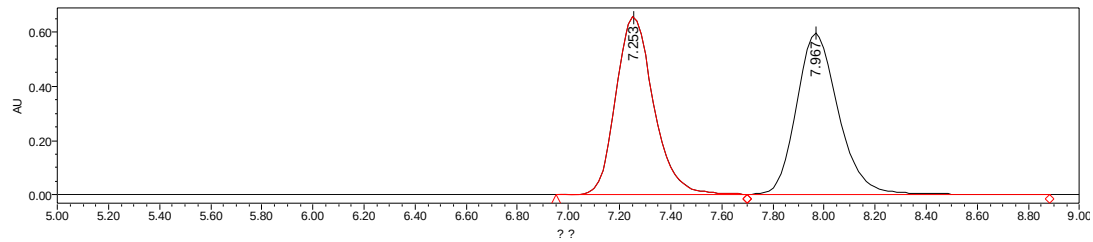

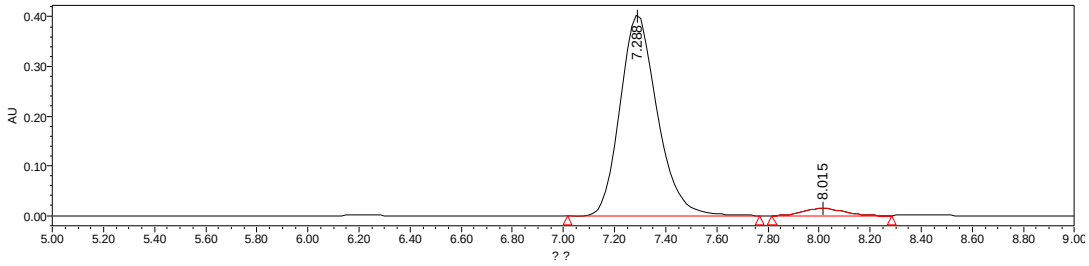

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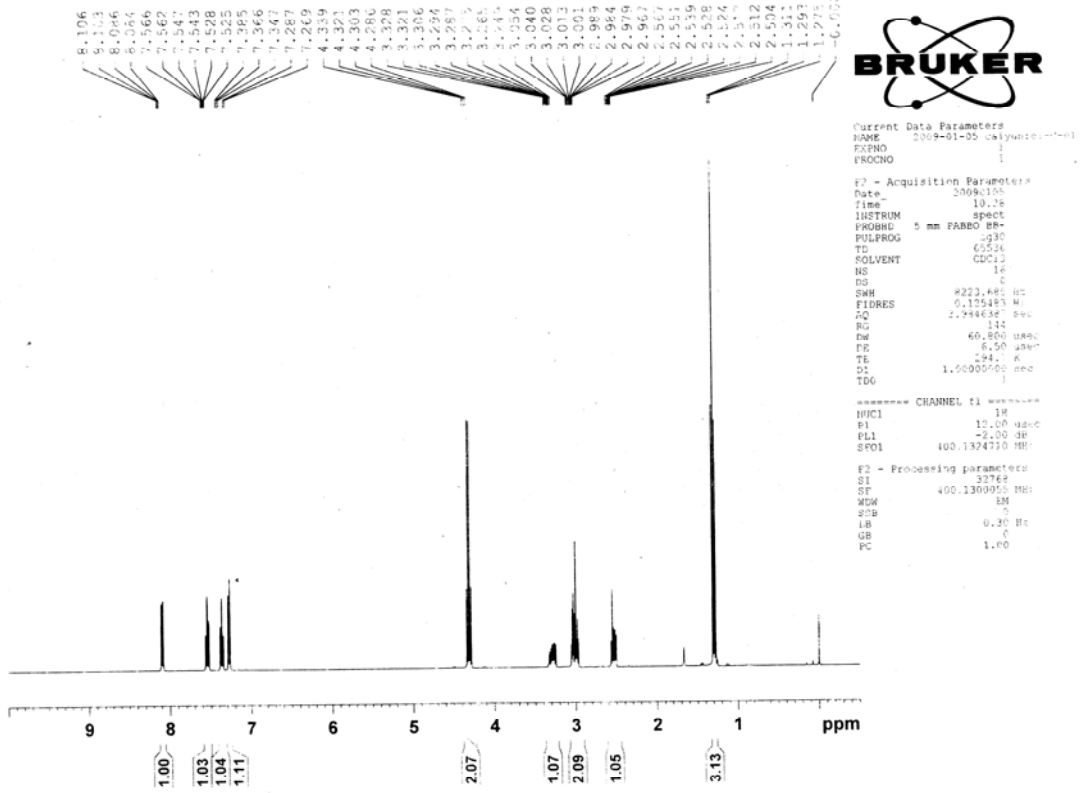

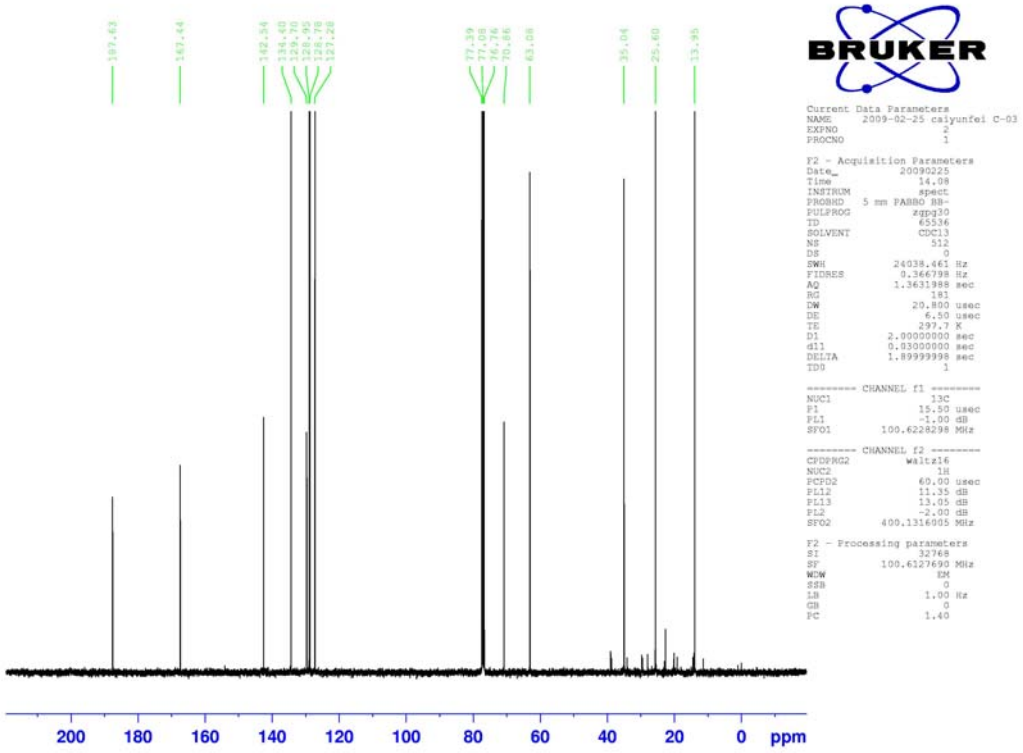

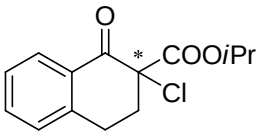

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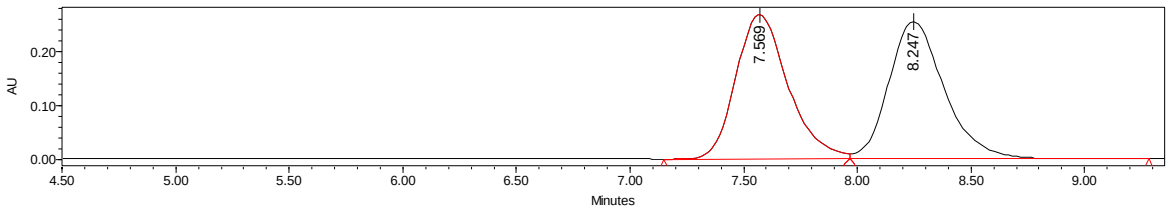

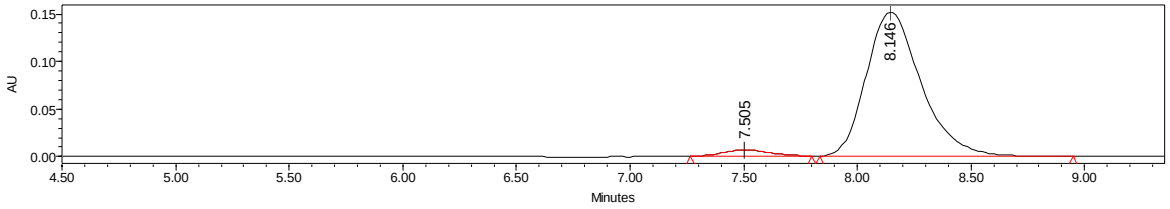

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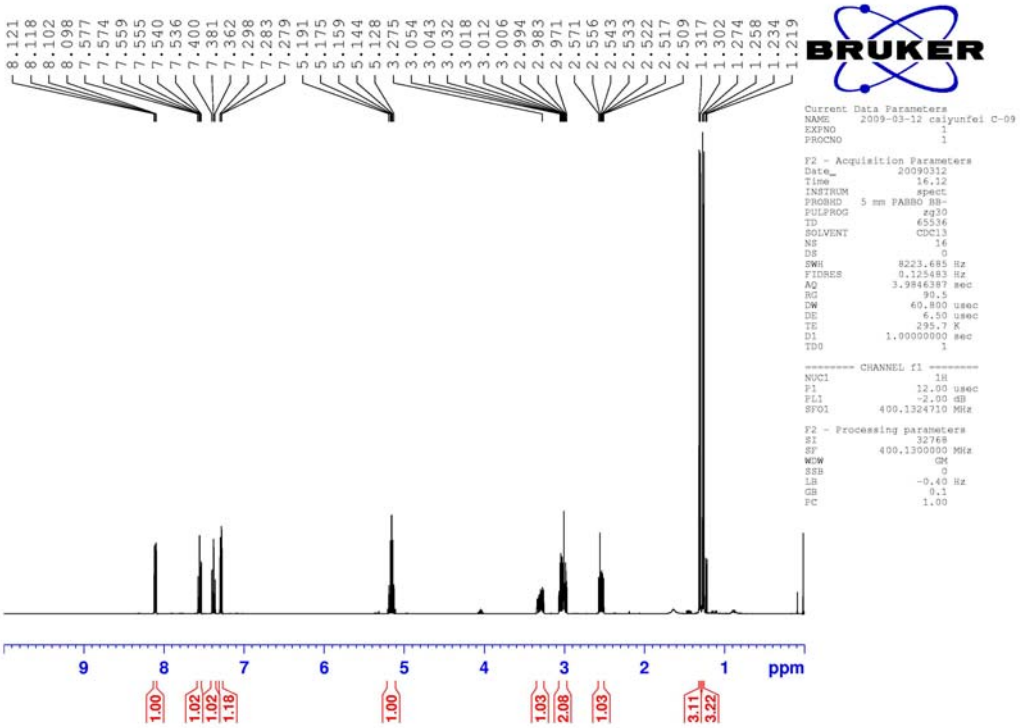
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DE 6.50 usec
TE 295.7 K
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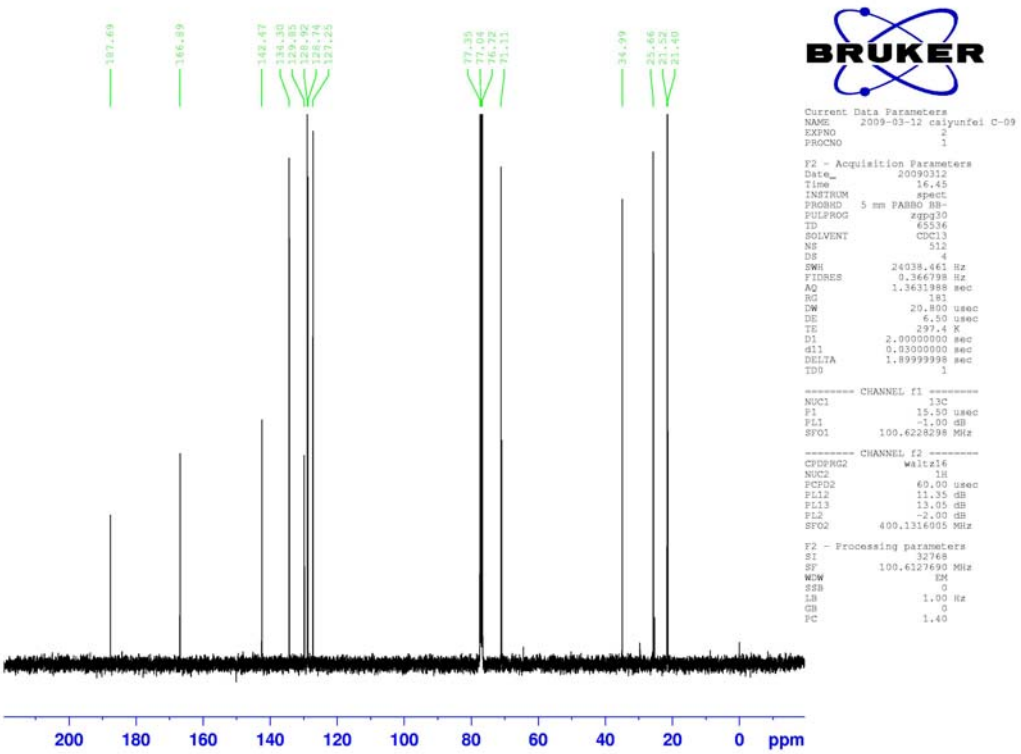
3.22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75.11
36.99
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21.52
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BRUKER
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DE 6.50 usec
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d11 0.03000000 sec
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SFO1 100.6228298 MHz
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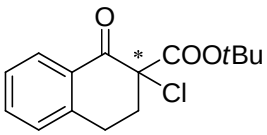

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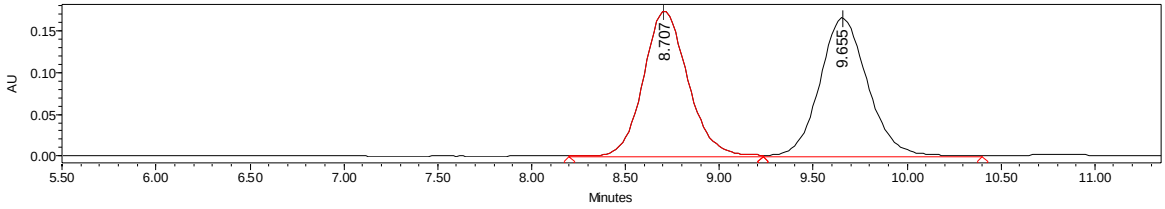

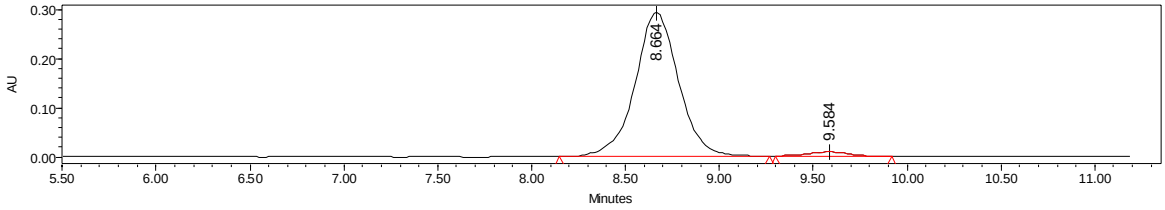

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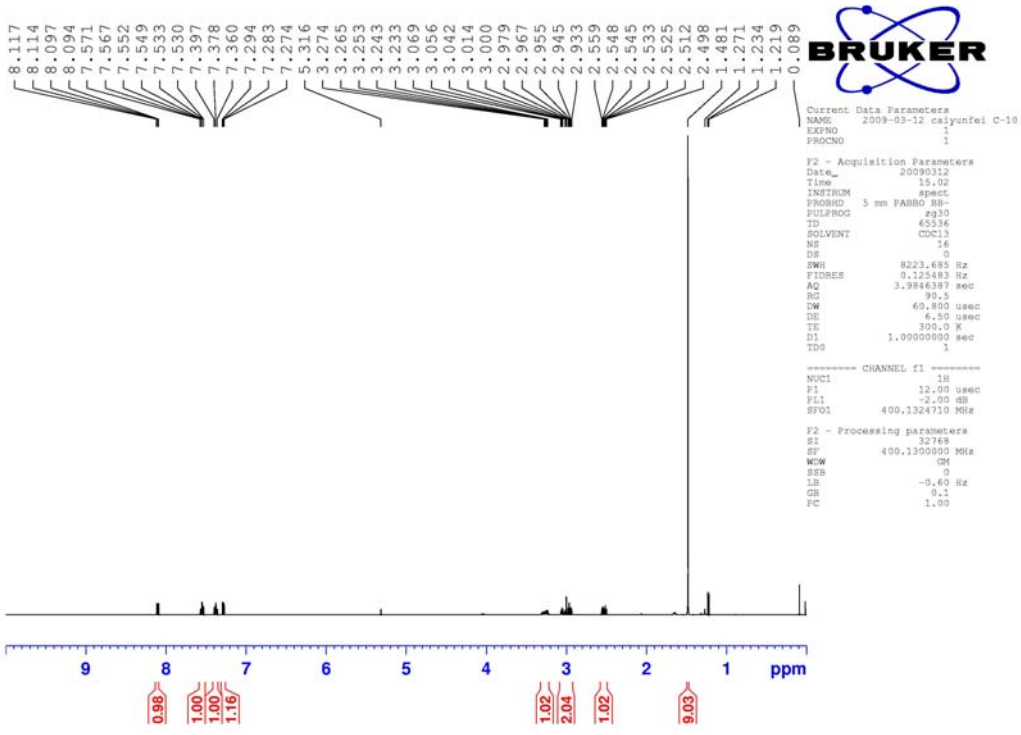
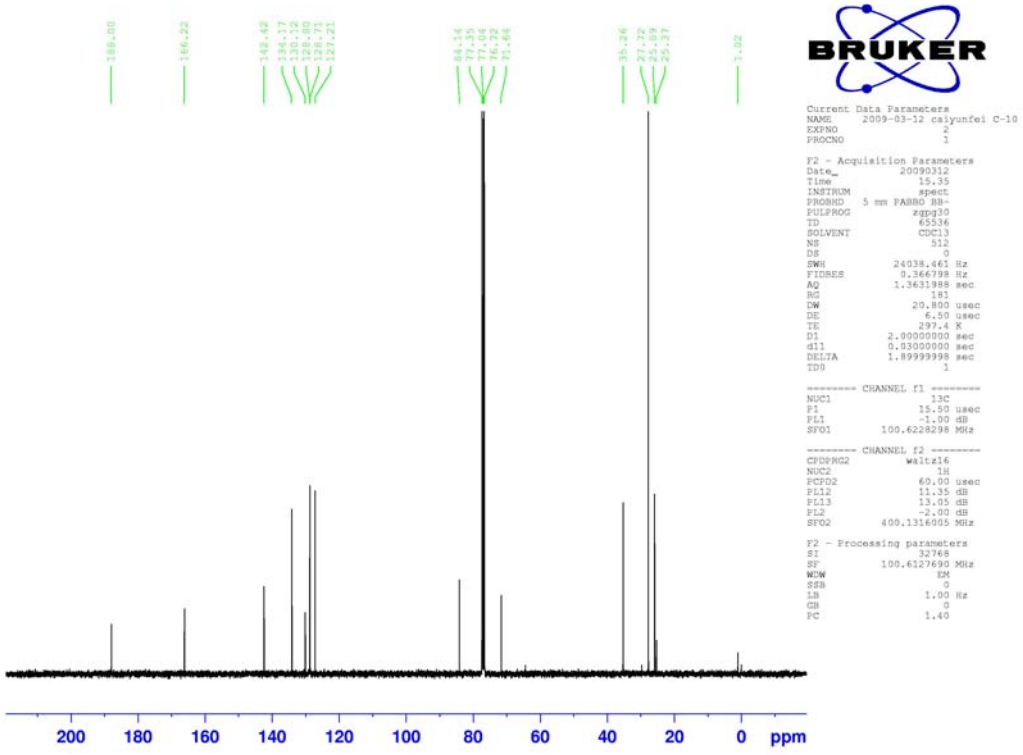
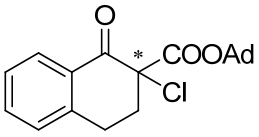
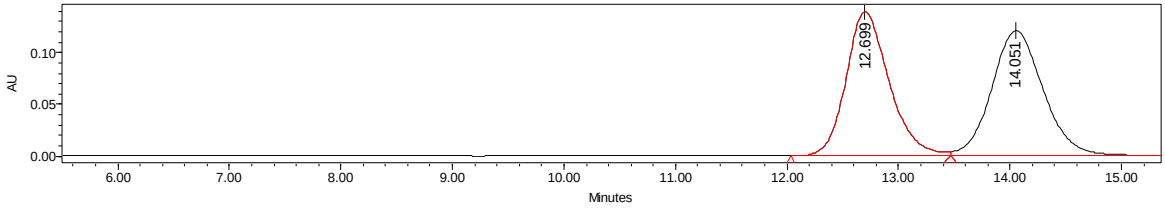
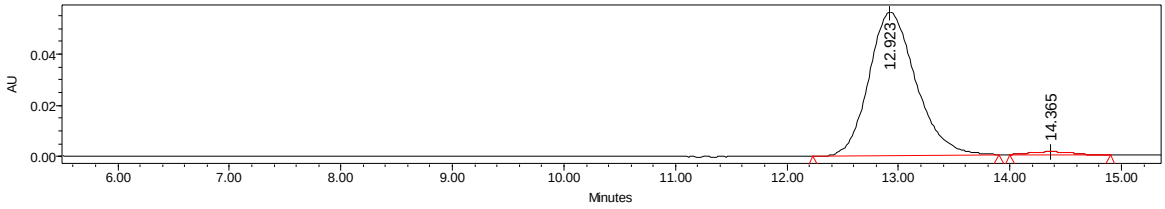






2e





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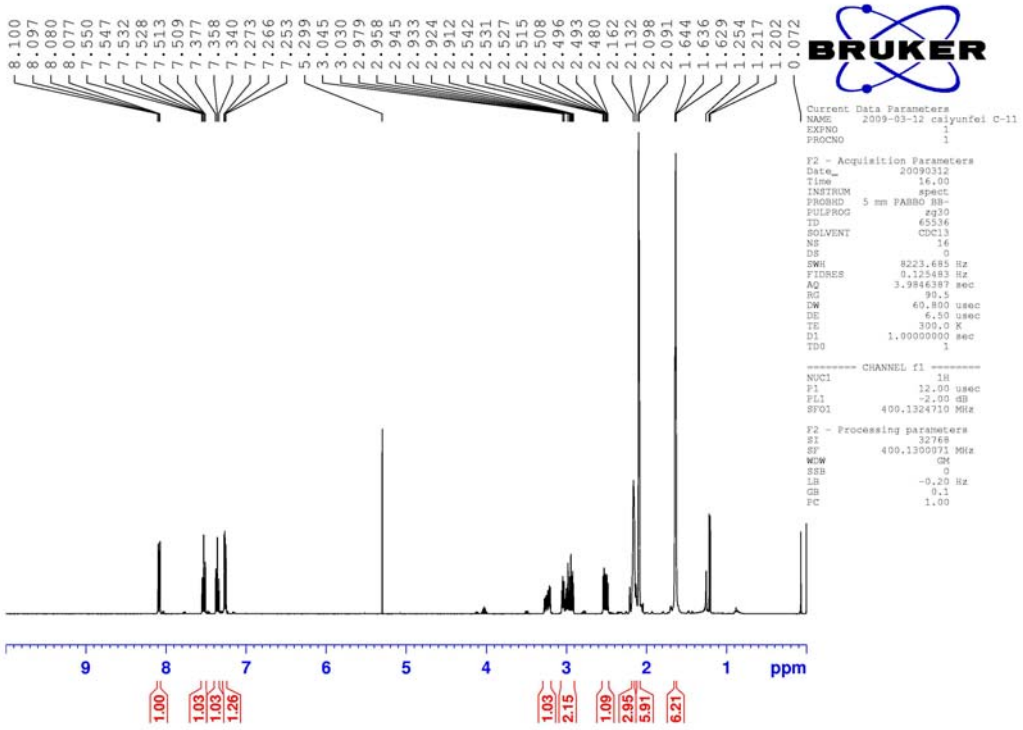

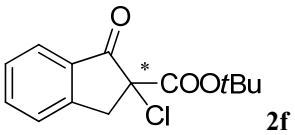

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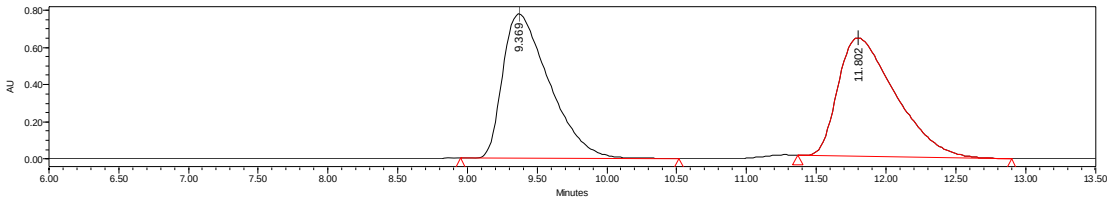

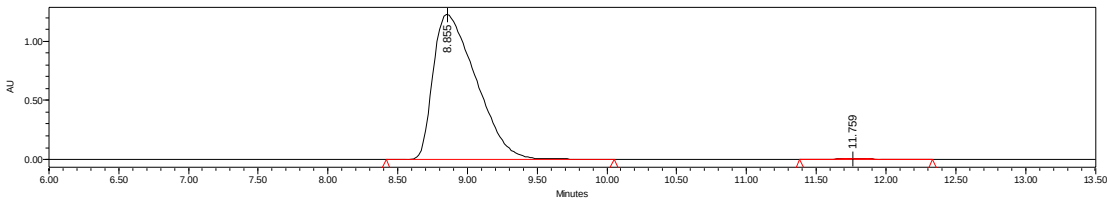

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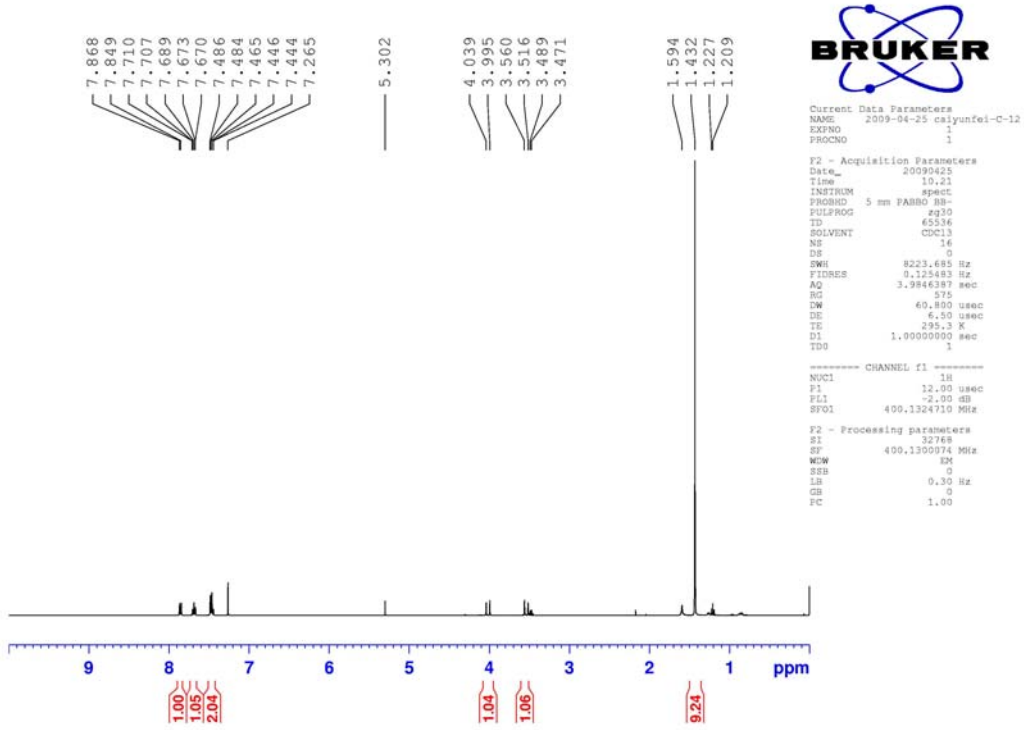
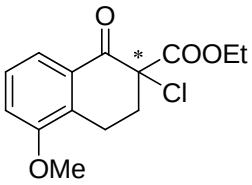
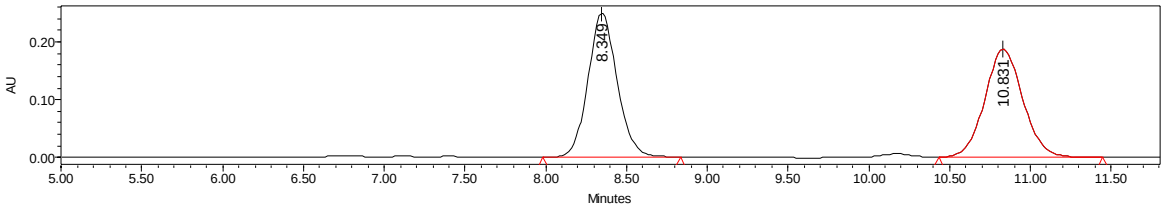
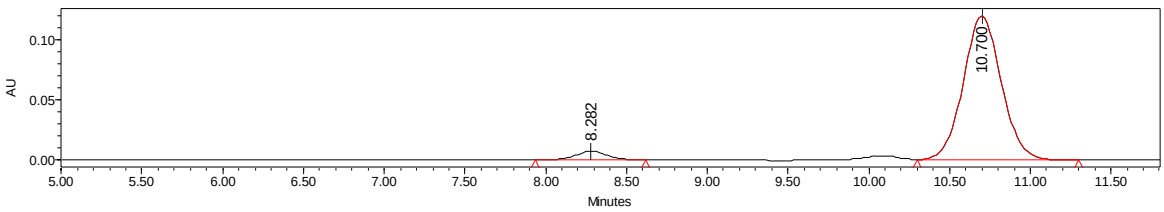
2		11.759	186062	0.71



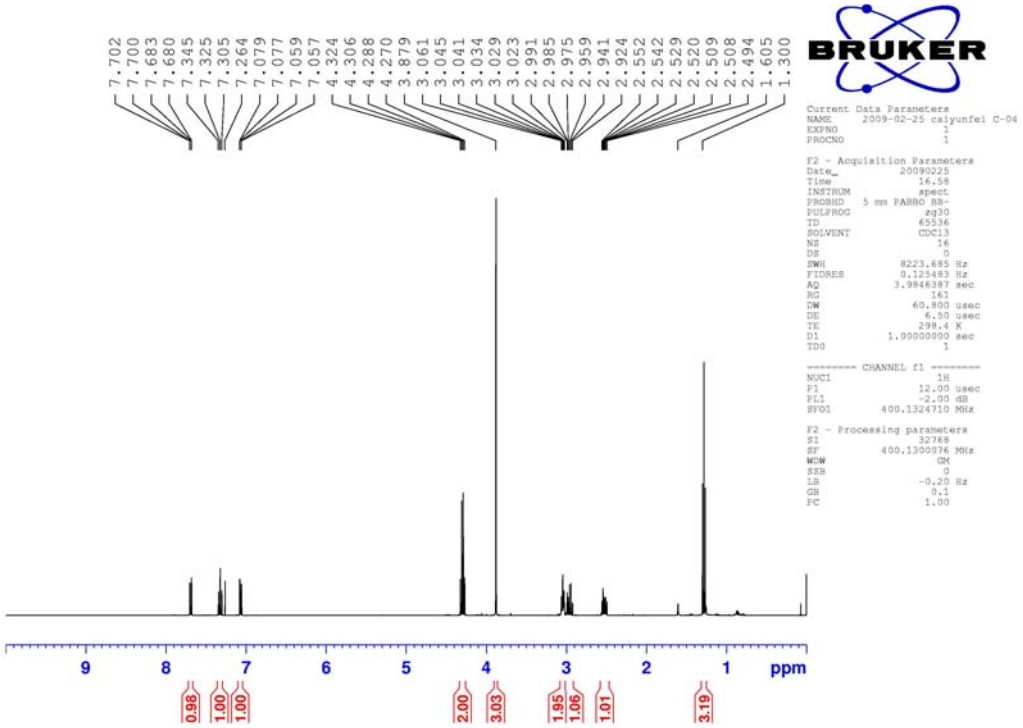


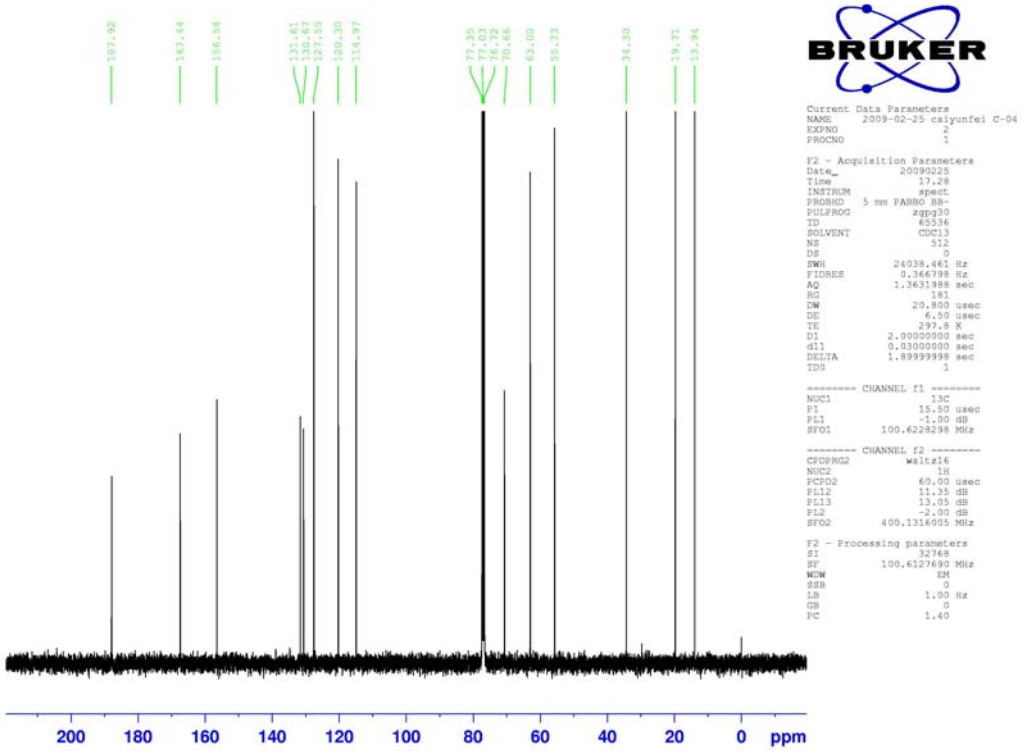
2g

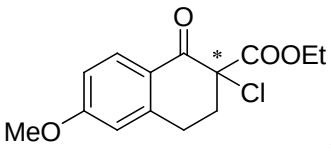




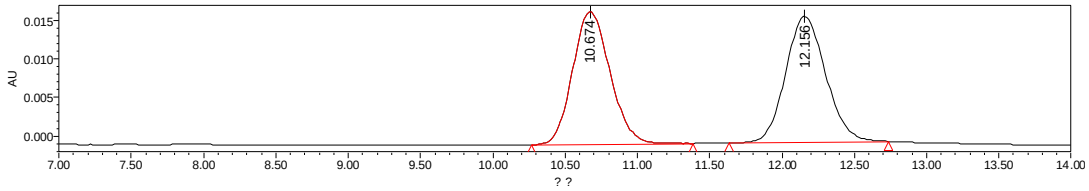
	Name	Retention Time	Area	% Area
1		8.282	96965	4.74
2		10.700	1947686	95.26

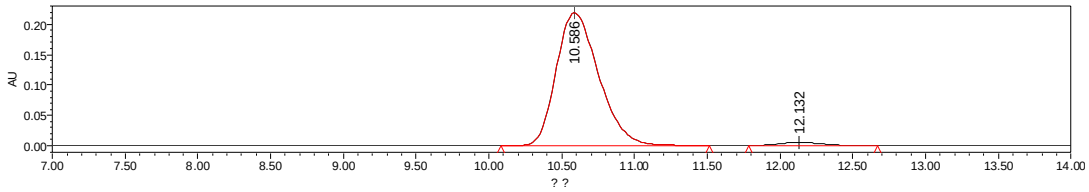

7.702
7.700
7.683
7.680
7.345
7.325
7.305
7.264
7.079
7.077
7.059
7.057
4.324
4.306
4.288
4.270
3.879
3.061
3.045
3.041
3.034
3.029
3.023
2.991
2.985
2.975
2.959
2.941
2.924
2.552
2.542
2.529
2.520
2.509
2.508
2.494
1.605
1.300
BRUKER
Current Data Parameters
NAME 2009-02-25 caiyunfei C-04
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090225
Time 16.58
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 161
DW 60.800 usec
DE 6.50 usec
TE 298.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300076 MHz
WDW EM
SSB 0
LB -0.20 Hz
GB 0.1
PC 1.00
9
8
7
6
5
4
3
2
1
ppm
0.98
1.00
1.00
2.00
3.03
1.95
1.06
1.01
3.19


167.92
167.44
156.54
131.61
130.67
127.55
120.30
114.97
77.35
77.03
76.72
70.66
63.00
55.73
34.30
19.71
13.94
BRUKER
Current Data Parameters
NAME 2009-02-25 caiyunfei C-04
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090225
Time 17.28
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 181
DW 20.800 usec
DE 6.50 usec
TE 297.8 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL12 11.35 dB
PL13 13.05 dB
PL2 -2.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

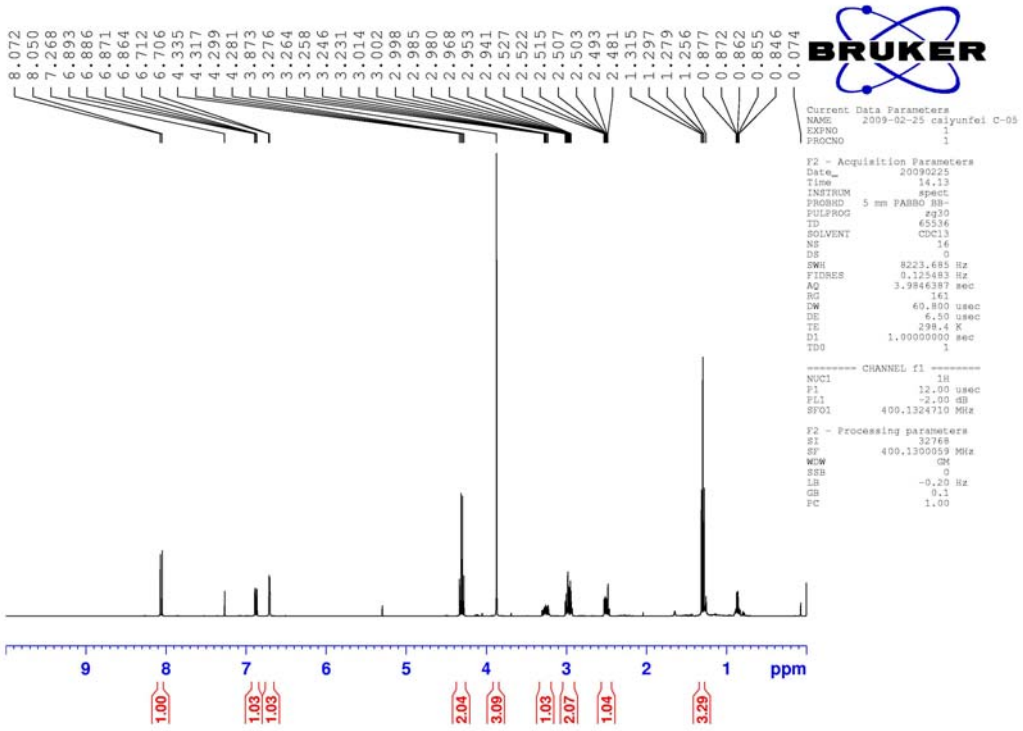


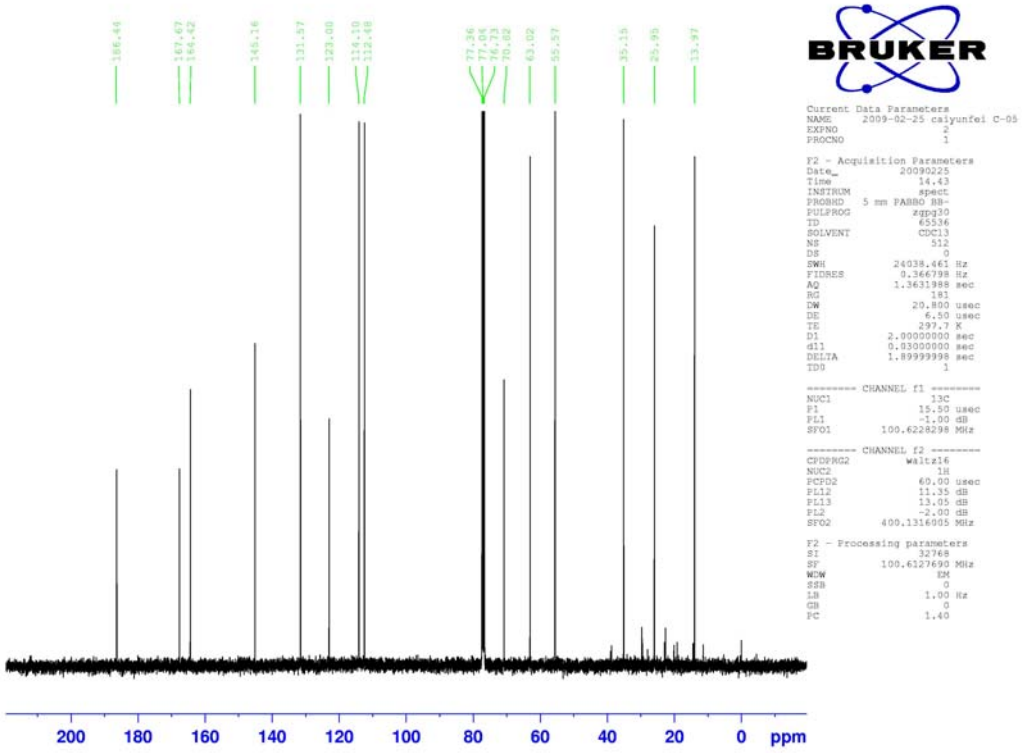
2h

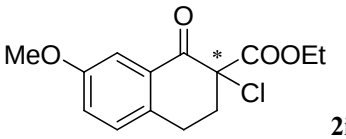




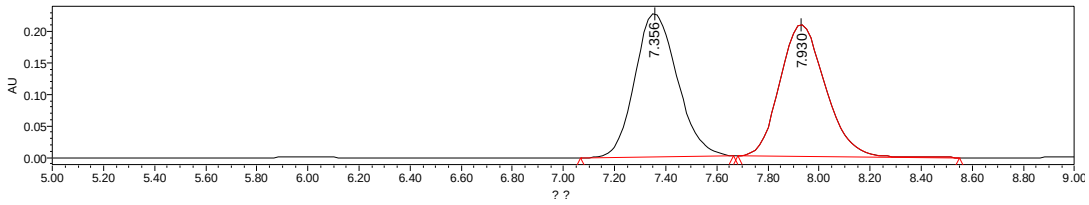
	Name	Retention Time	Area	% Area
1		10.586	4452088	97.52
2		12.132	113089	2.48

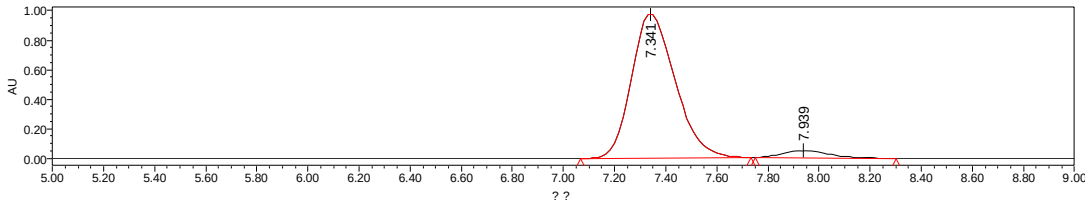




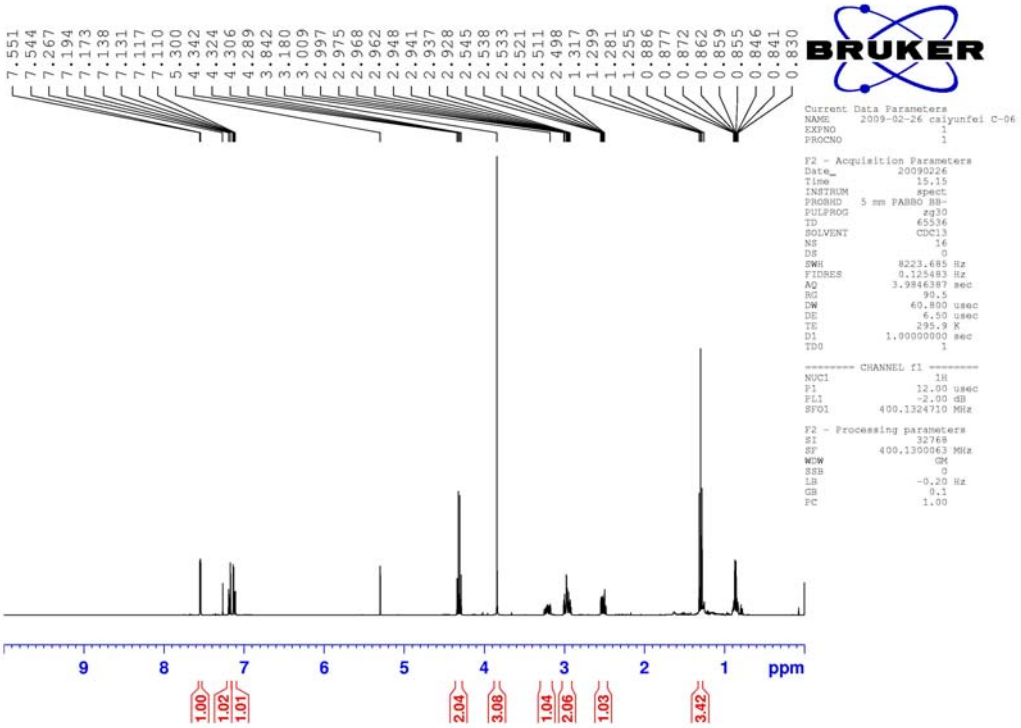


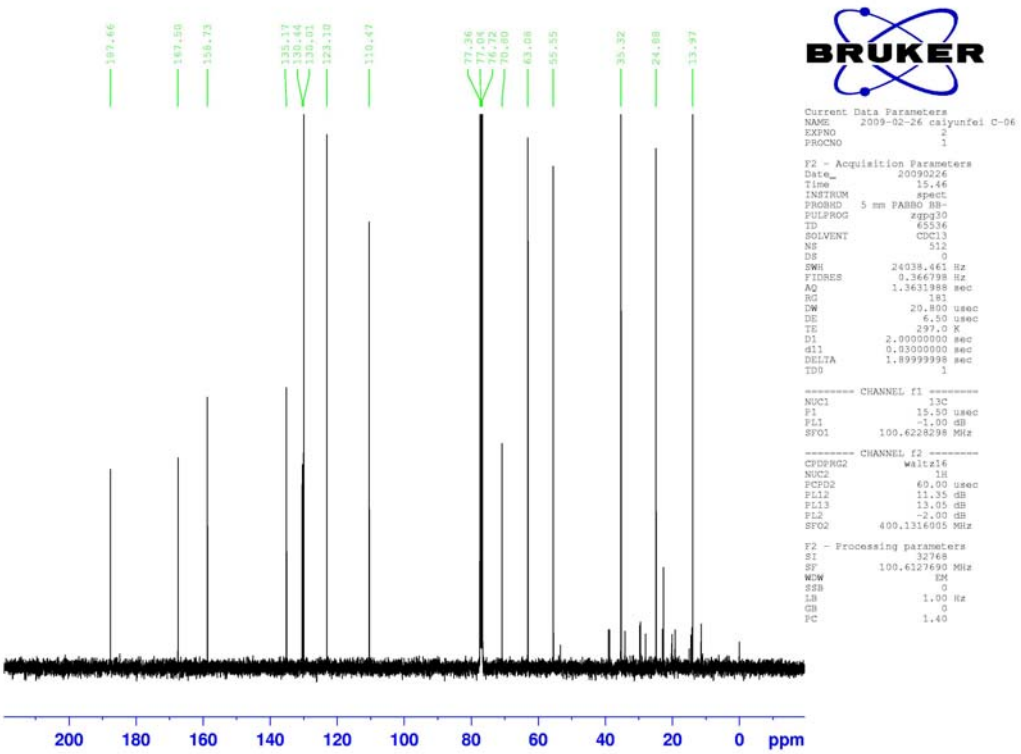
2i

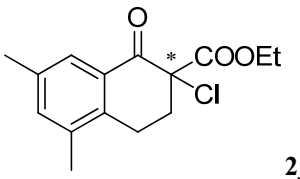




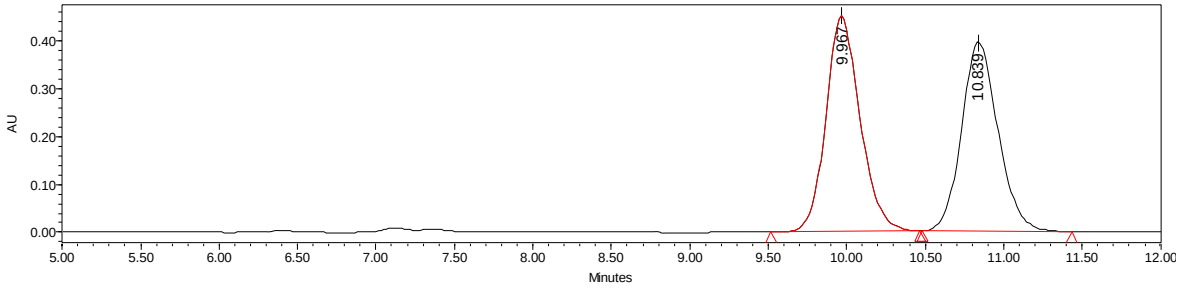
	Name	Retention Time	Area	% Area
1		7.341	11536606	95.29
2		7.939	569848	4.71

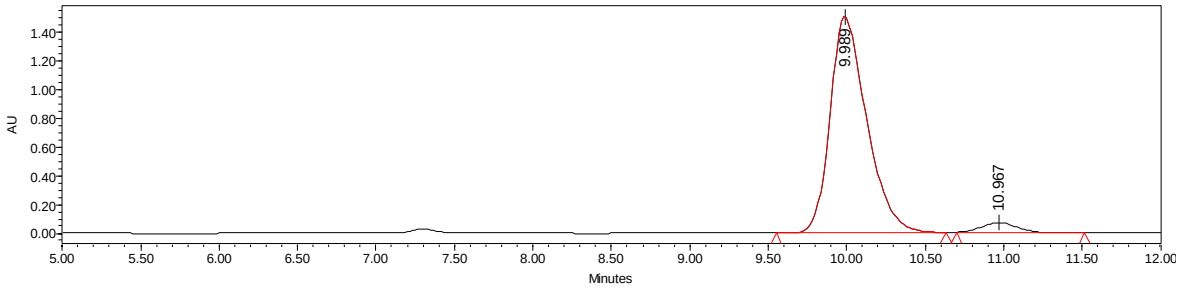

7.551
7.544
7.267
7.194
7.173
7.138
7.131
7.117
7.110
5.300
4.342
4.324
4.306
4.289
3.842
3.180
3.009
2.997
2.975
2.968
2.962
2.948
2.941
2.937
2.928
2.545
2.538
2.533
2.521
2.511
2.498
1.317
1.299
1.281
1.255
0.886
0.877
0.872
0.862
0.859
0.855
0.846
0.841
0.830
BRUKER
Current Data Parameters
NAME 2009-02-26 caiyunfei C-06
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090226
Time 15.15
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 90.5
DW 60.800 usec
DE 6.50 usec
TE 295.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300063 MHz
WDW EM
SSB 0
LB -0.20 Hz
GB 0.1
PC 1.00
9
8
7
6
5
4
3
2
1
ppm
1.00
1.02
1.01
2.04
3.08
1.04
2.06
1.03
3.42


197.46
167.58
158.73
135.17
130.44
130.01
123.19
110.47
77.36
77.04
76.72
70.60
63.08
55.55
35.32
24.88
13.97
BRUKER
Current Data Parameters
NAME 2009-02-26 caiyunfei C-06
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090226
Time 15.46
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 181
DW 20.800 usec
DE 6.50 usec
TE 297.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL12 11.35 dB
PL13 13.05 dB
PL2 -2.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

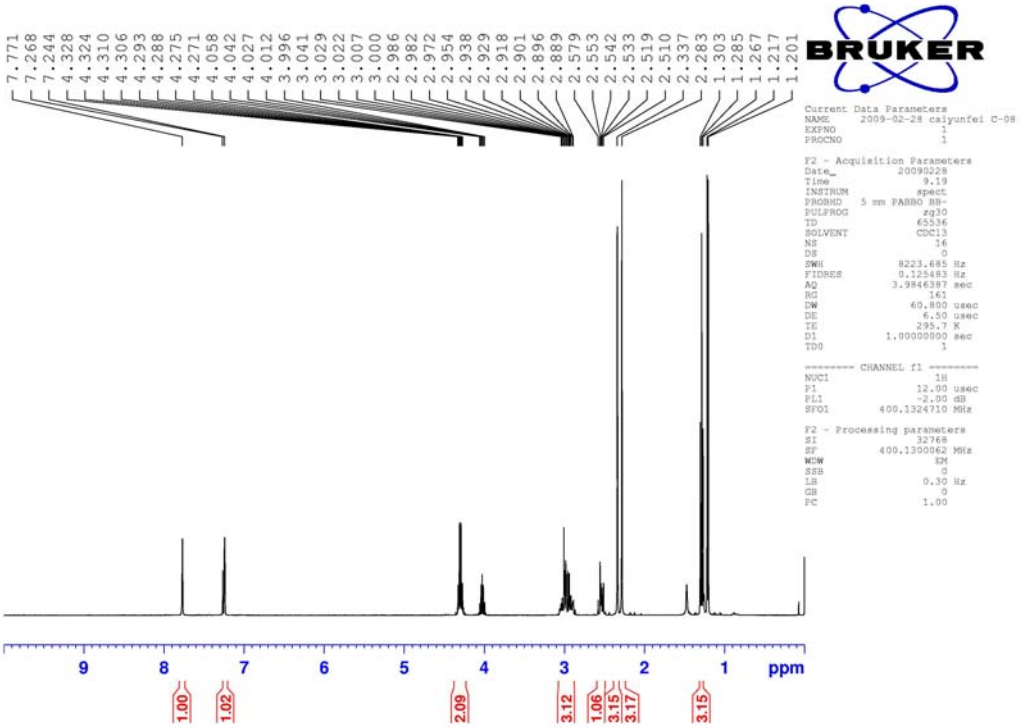


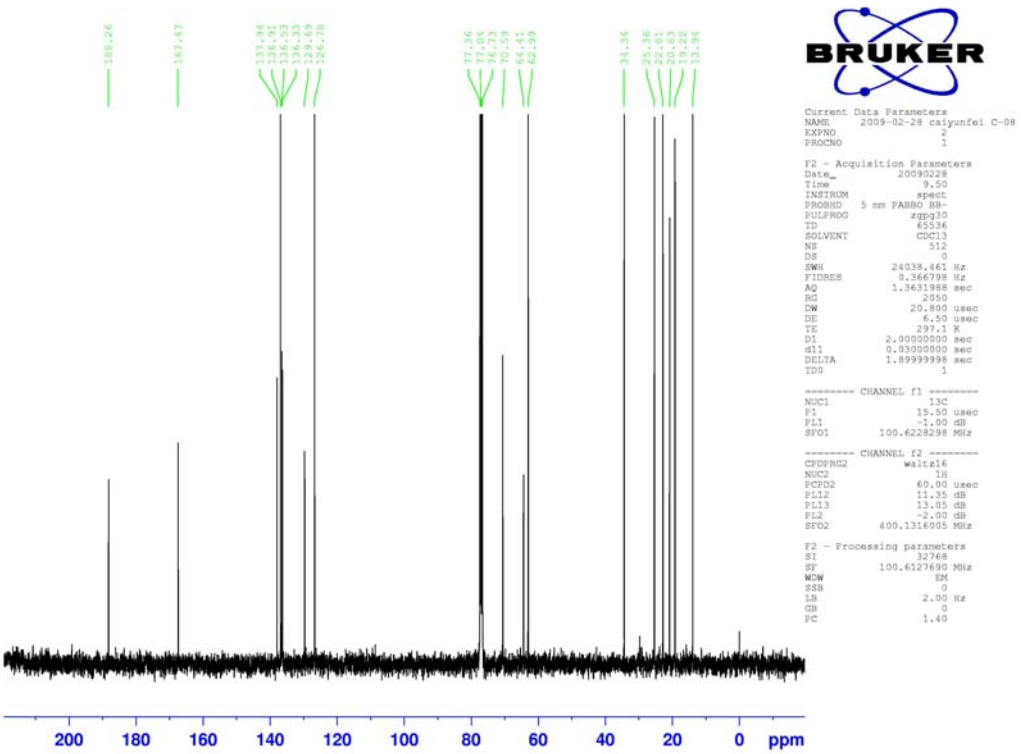
2j

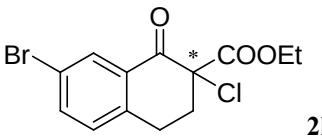




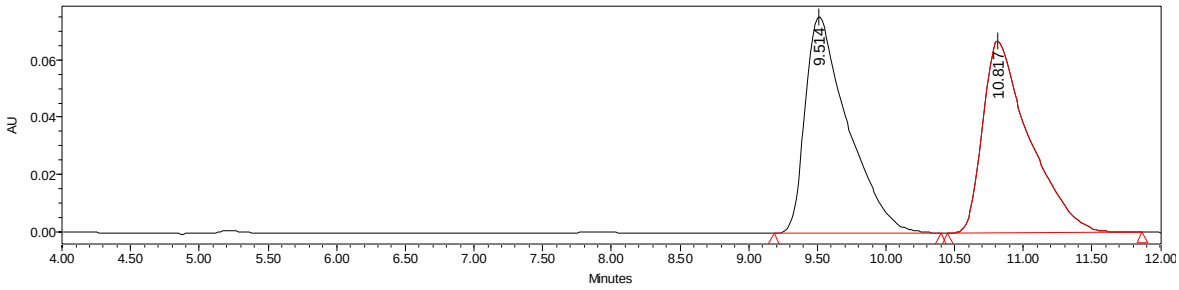
	Name	Retention Time	Area	% Area
1		9.989	24418250	95.81
2		10.967	1066883	4.19

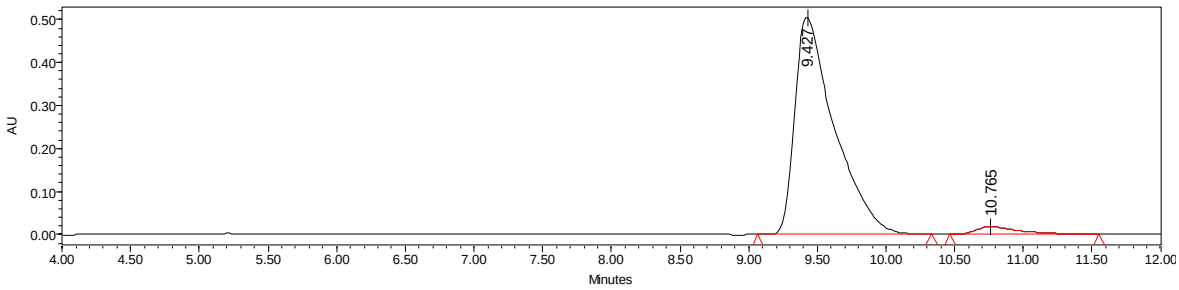

7.771
7.268
7.244
4.328
4.324
4.310
4.306
4.293
4.288
4.275
4.271
4.058
4.042
4.027
4.012
3.996
3.041
3.029
3.022
3.007
3.000
2.986
2.982
2.972
2.954
2.938
2.929
2.918
2.901
2.896
2.889
2.579
2.553
2.542
2.533
2.519
2.510
2.337
2.283
1.303
1.285
1.267
1.217
1.201
BRUKER
Current Data Parameters
NAME 2009-02-28 caiyunfei C-08
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090228
Time 9.19
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 161
DW 60.800 usec
DE 6.50 usec
TE 295.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300062 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
ppm
1.00
1.02
2.09
3.12
1.06
3.15
3.17
3.15


166.26
147.47
137.94
136.91
136.53
136.33
129.69
126.78
77.36
77.04
76.73
70.59
64.41
62.99
34.34
25.36
22.81
20.63
19.22
13.94
BRUKER
Current Data Parameters
NAME 2009-02-28 caiyunfei C-08
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090228
Time 9.50
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 297.1 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL12 11.35 dB
PL13 13.05 dB
PL2 -2.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

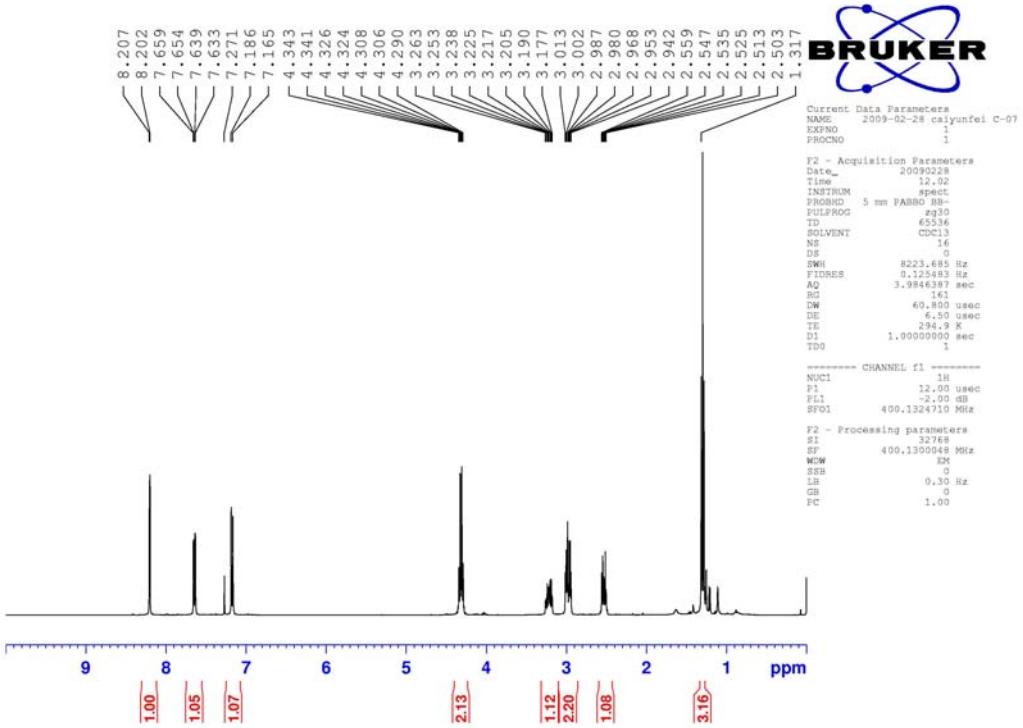


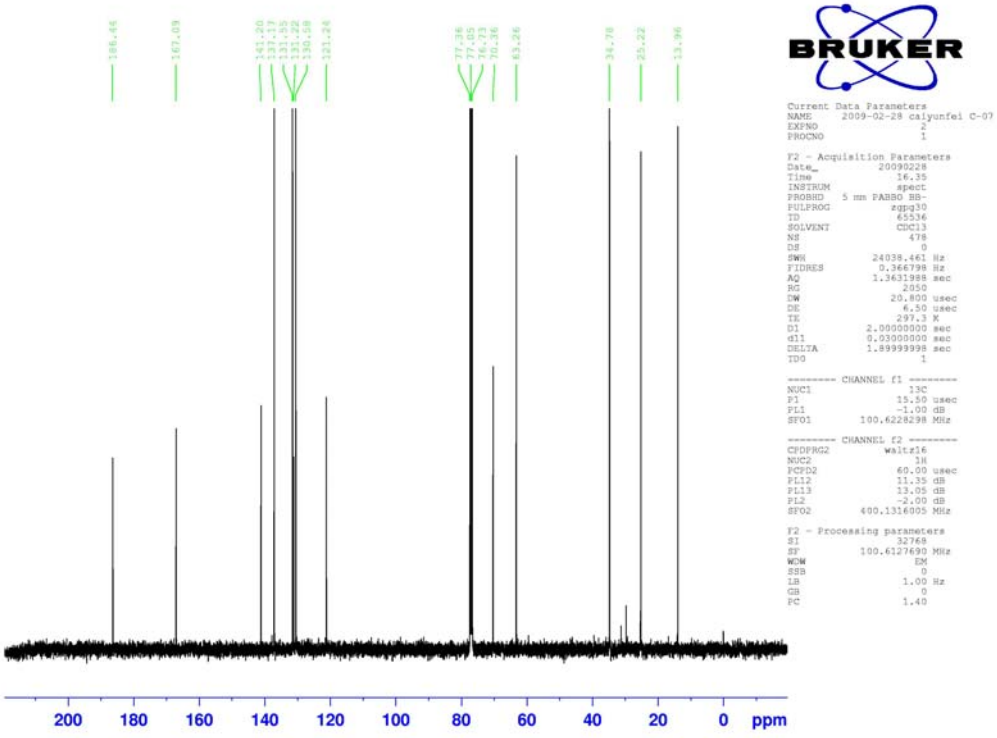
2k

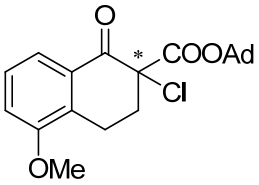


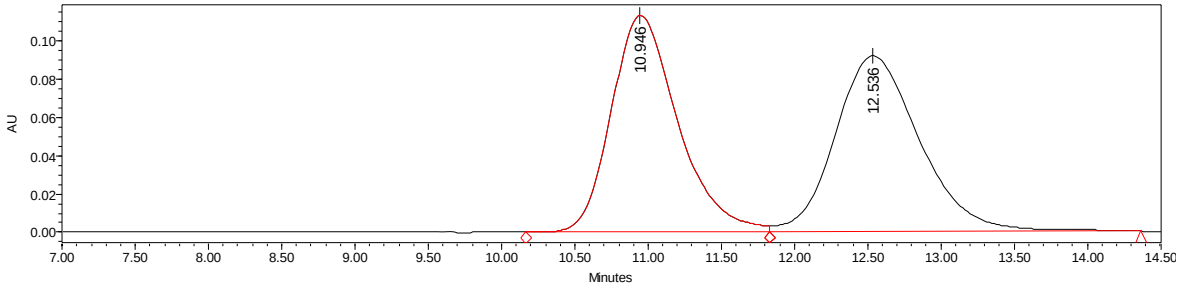


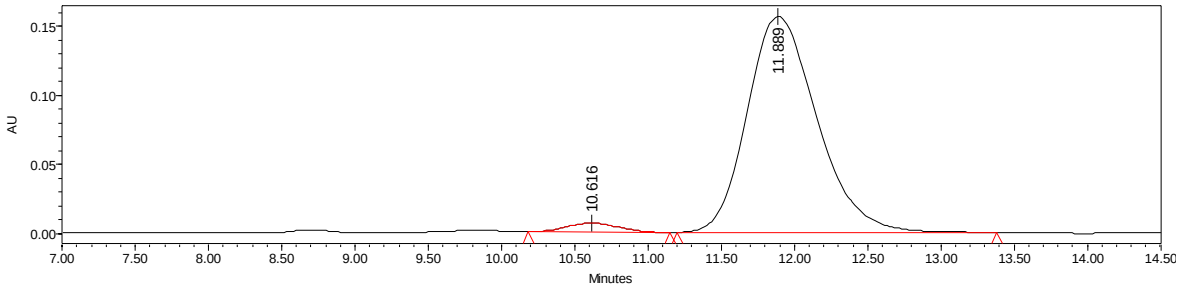
	Name	Retention Time	Area	% Area
1		9.427	10116441	96.30
2		10.765	388423	3.70


8.207
8.202
7.659
7.654
7.639
7.633
7.271
7.186
7.165
4.343
4.341
4.326
4.324
4.308
4.306
4.290
3.263
3.253
3.238
3.225
3.217
3.205
3.190
3.177
3.013
3.002
2.987
2.980
2.968
2.953
2.942
2.559
2.547
2.535
2.525
2.513
2.503
1.317
BRUKER
Current Data Parameters
NAME 2009-02-28 caiyunfei C-07
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090228
Time 12.02
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 161
DW 60.800 usec
DE 6.50 usec
TE 294.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300048 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
ppm
1.00
1.05
1.07
2.13
1.12
2.20
1.08
3.16

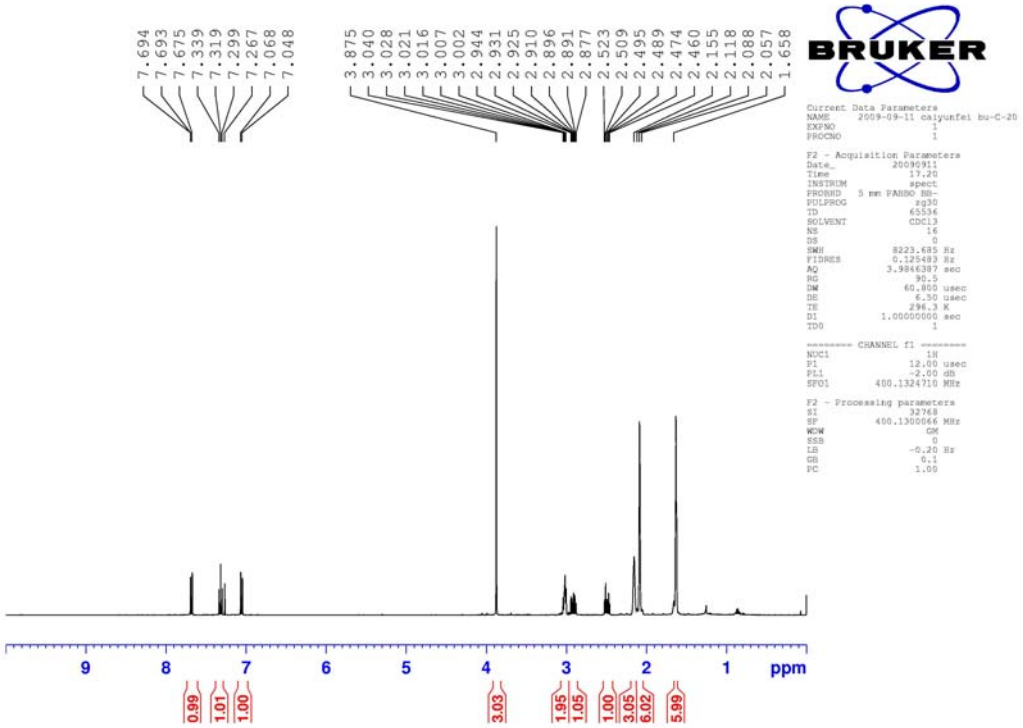

166.44
167.09
141.20
137.17
131.35
131.22
130.58
121.24
77.36
77.05
76.73
70.36
63.26
34.78
25.22
13.96
BRUKER
Current Data Parameters
NAME 2009-02-28 caiyunfei C-07
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090228
Time 16.35
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 478
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 297.3 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL12 11.35 dB
PL13 13.05 dB
PL2 -2.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

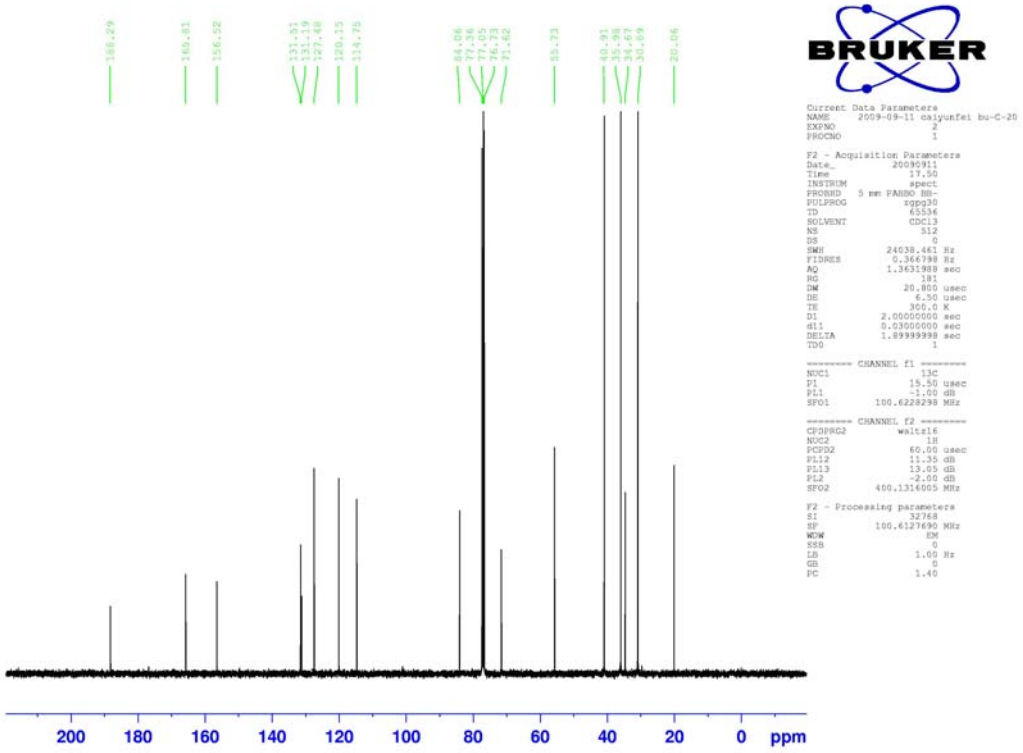


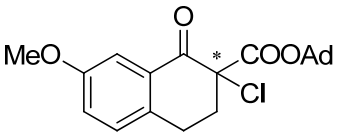


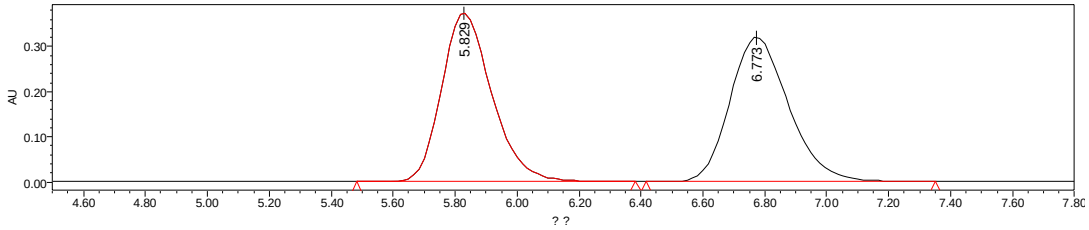


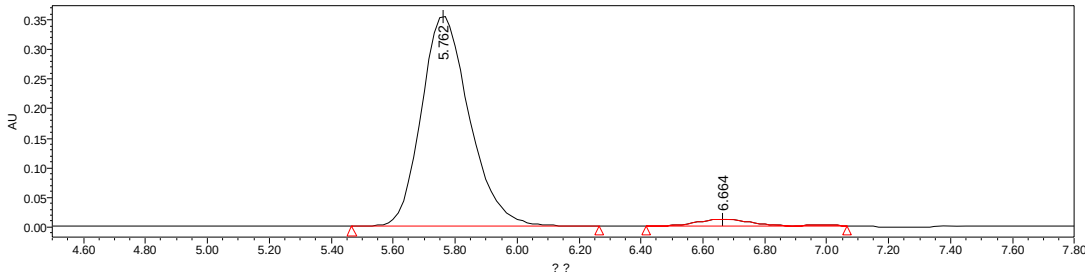
	Name	Retention Time	Area	% Area
1		10.616	161871	3.02
2		11.889	5191451	96.98


7.694
7.693
7.675
7.339
7.319
7.299
7.267
7.068
7.048
3.875
3.040
3.028
3.021
3.016
3.007
3.002
2.944
2.931
2.925
2.910
2.896
2.891
2.877
2.523
2.509
2.495
2.489
2.474
2.460
2.155
2.118
2.088
2.057
1.658
BRUKER
Current Data Parameters
NAME 2009-09-11 caiyunfei bu-C-20
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090911
Time 17.20
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 90.5
DW 60.800 usec
DE 6.50 usec
TE 296.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300666 MHz
WDW GM
SSB 0
LB -0.20 Hz
GB 0.1
PC 1.00
9
8
7
6
5
4
3
2
1
ppm
0.99
1.01
1.00
3.03
1.95
1.05
1.00
3.05
6.02
5.99

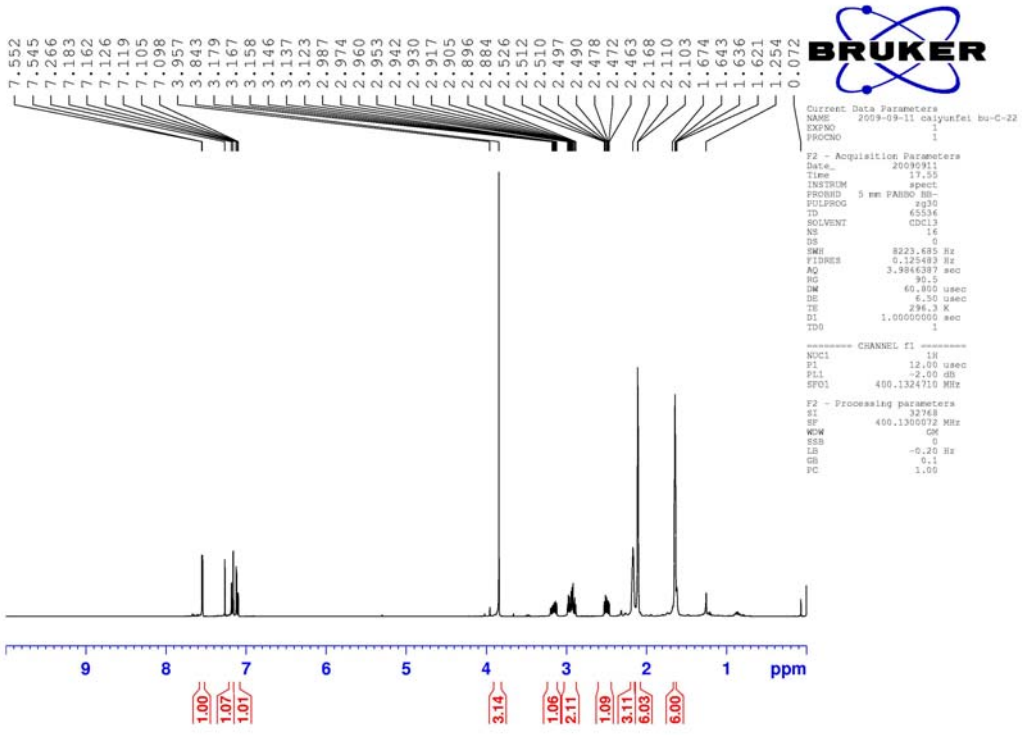

166.29
159.81
156.52
131.51
131.19
127.48
120.15
114.75
84.06
77.36
77.05
76.73
71.62
55.73
40.91
35.98
34.67
30.69
20.06
BRUKER
Current Data Parameters
NAME 2009-09-11 caiyunfei bu-C-20
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090911
Time 17.50
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 181
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL12 11.35 dB
PL13 13.05 dB
PL2 -2.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

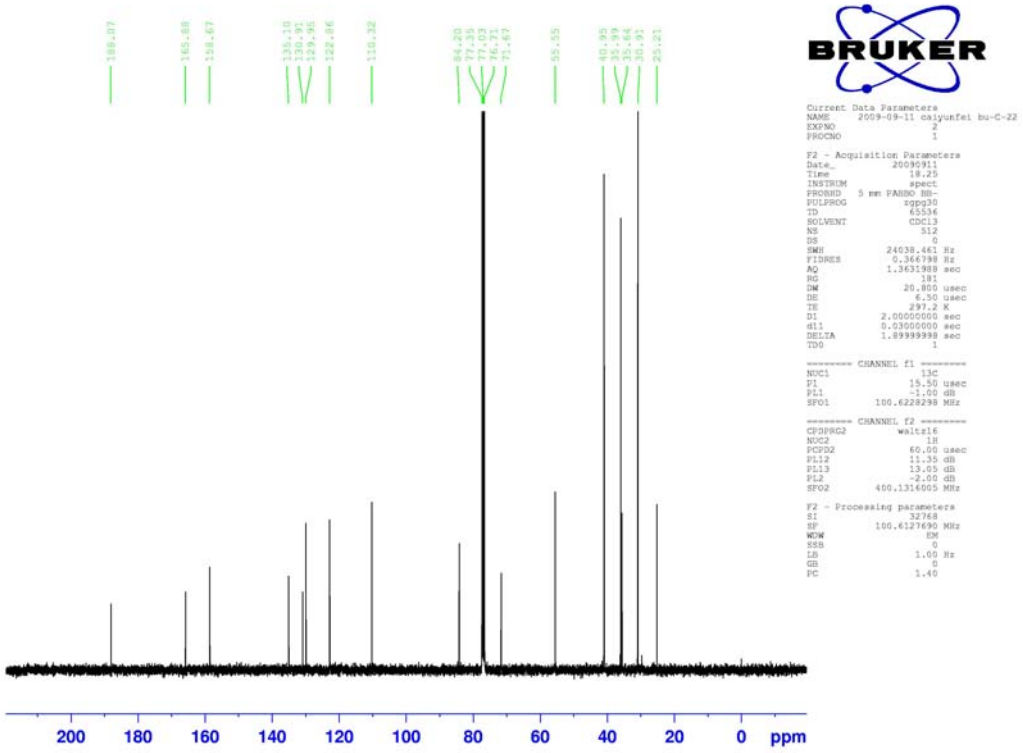


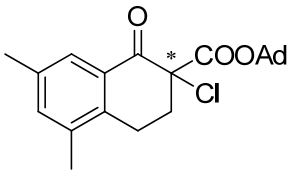


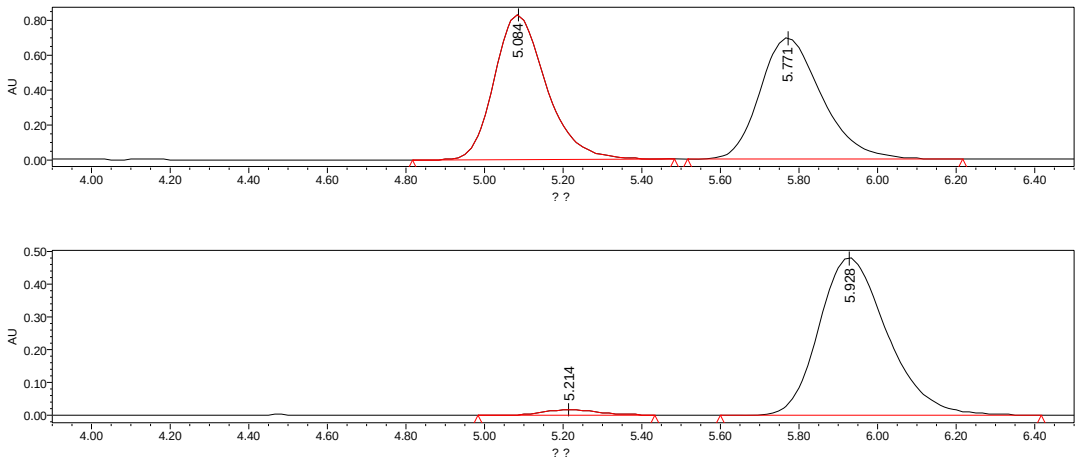


	Name	Retention Time	Area	% Area
1		5.762	3859116	96.27
2		6.664	149484	3.73

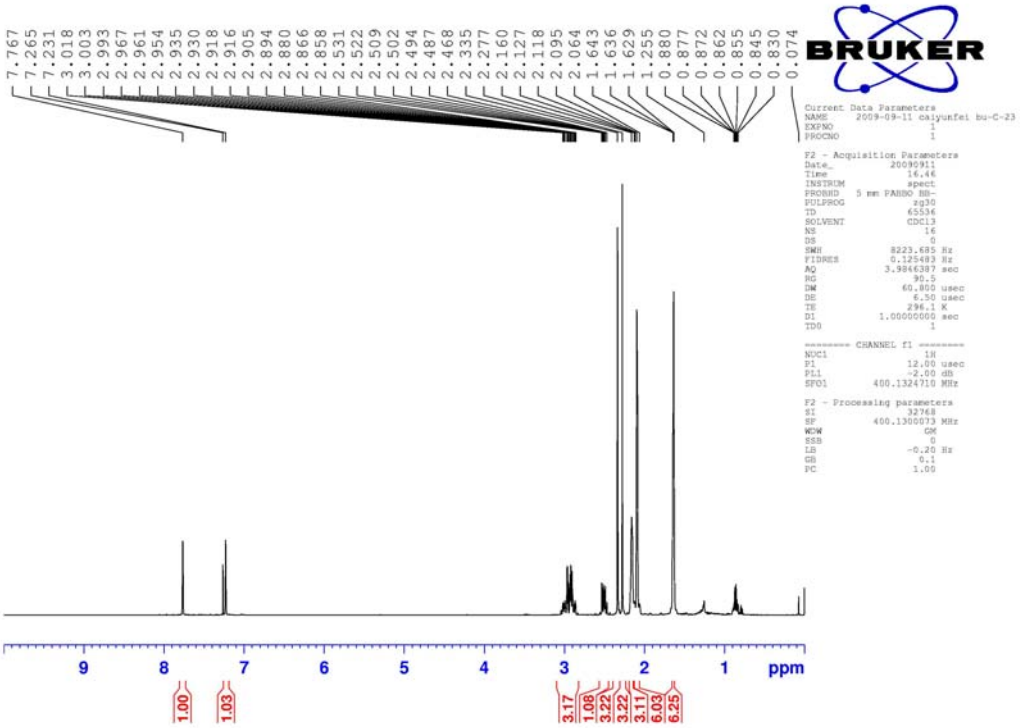


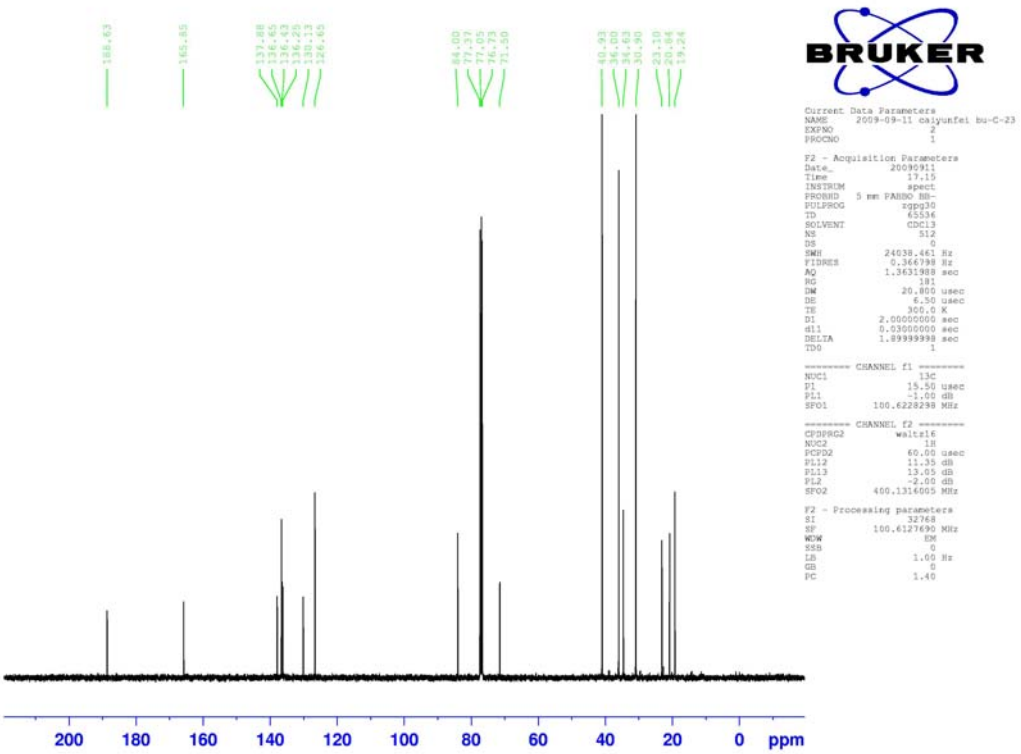


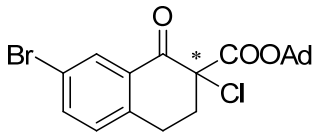


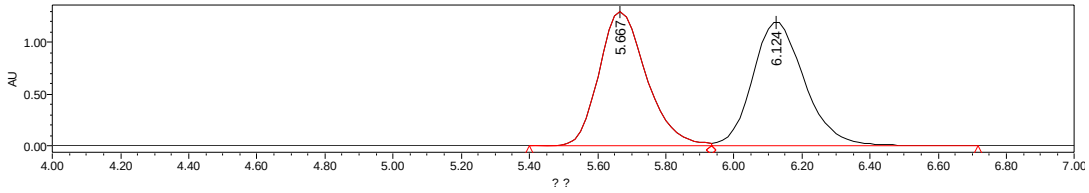


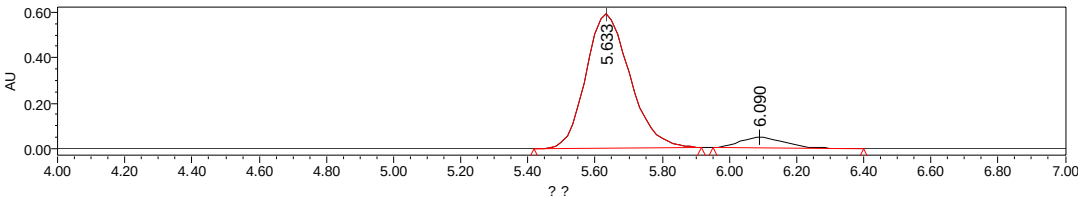
	Name	Retention Time	Area	% Area
1		5.214	159979	2.69
2		5.928	5782700	97.31


7.767
7.265
7.231
3.018
3.003
2.993
2.967
2.961
2.954
2.935
2.930
2.918
2.916
2.905
2.894
2.880
2.866
2.858
2.531
2.522
2.509
2.502
2.494
2.487
2.468
2.335
2.277
2.160
2.127
2.118
2.095
2.064
1.643
1.636
1.629
1.255
0.880
0.877
0.872
0.862
0.855
0.845
0.830
0.074
BRUKER
Current Data Parameters
NAME 2009-09-11 caiyunfei bu-C-23
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090911
Time 16.46
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 90.5
DW 60.800 usec
DE 6.50 usec
TE 296.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300073 MHz
WDW EM
SSB 0
LB -0.20 Hz
GB 0.1
PC 1.00
9
8
7
6
5
4
3
2
1
ppm
1.00
1.03
3.17
1.08
3.22
3.22
3.11
6.03
6.25

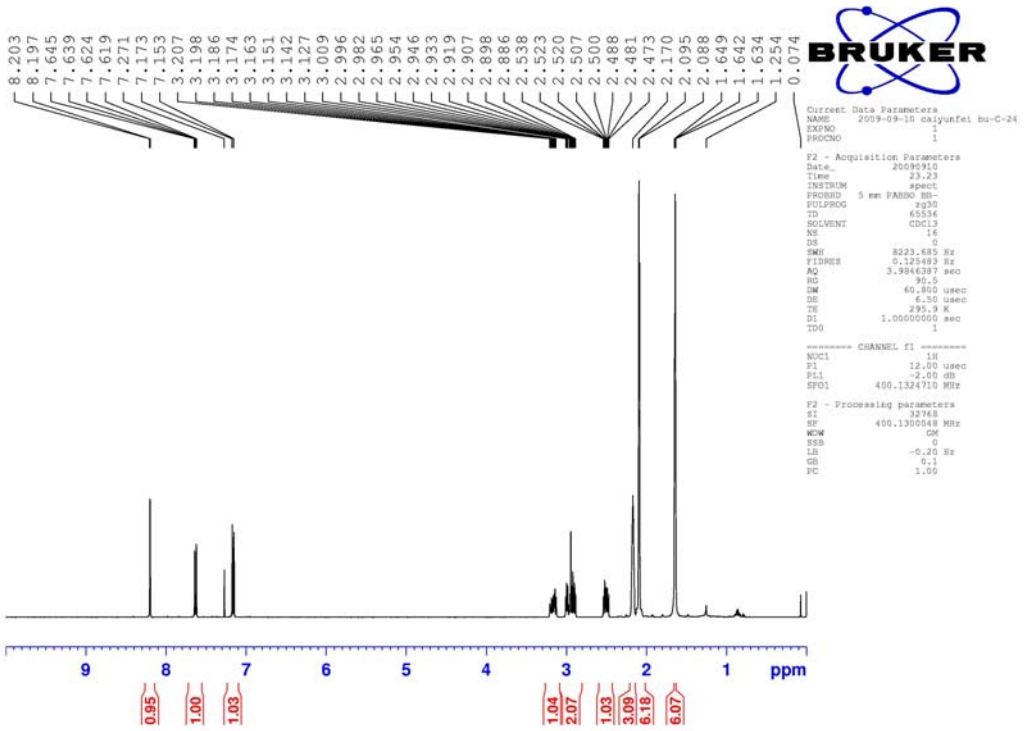

168.63
165.45
137.88
136.45
136.43
136.20
130.13
126.45
84.00
77.37
77.05
76.73
71.50
40.93
36.00
34.63
30.90
23.10
20.84
19.24
BRUKER
Current Data Parameters
NAME 2009-09-11 caiyunfei bu-C-23
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090911
Time 17.15
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 181
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL12 11.35 dB
PL13 13.05 dB
PL2 -2.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

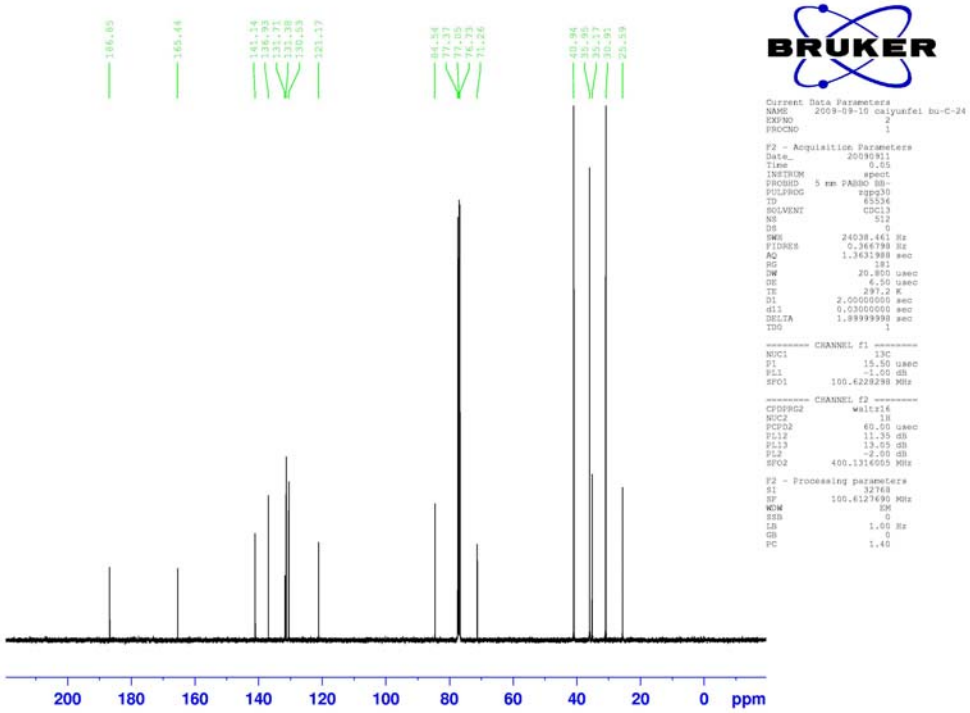


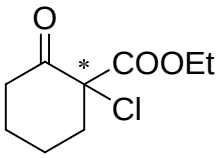


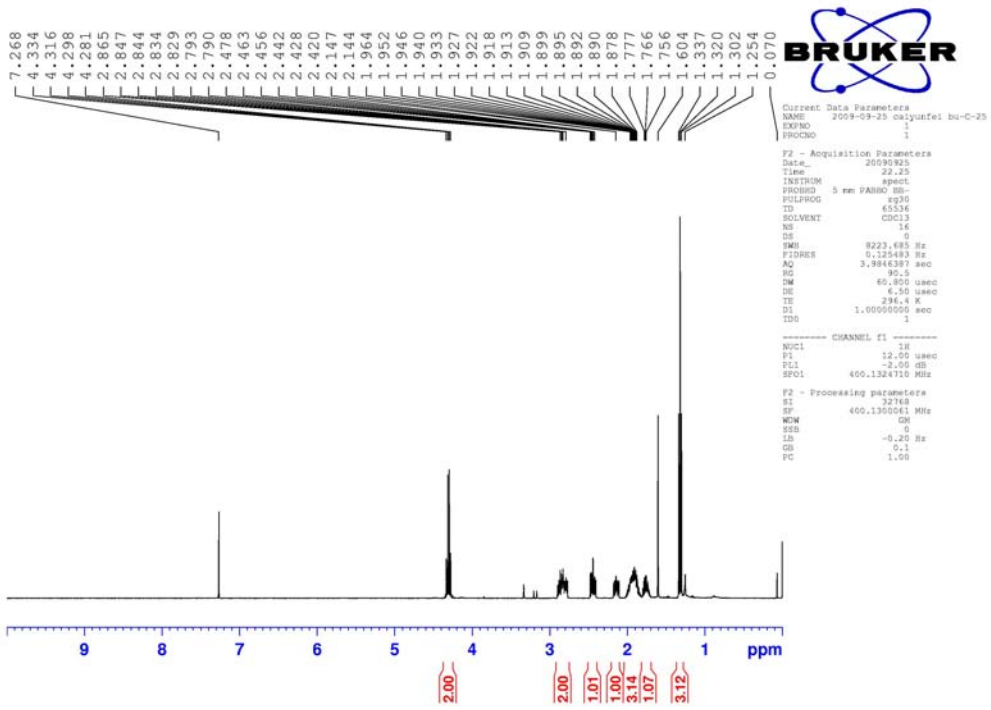


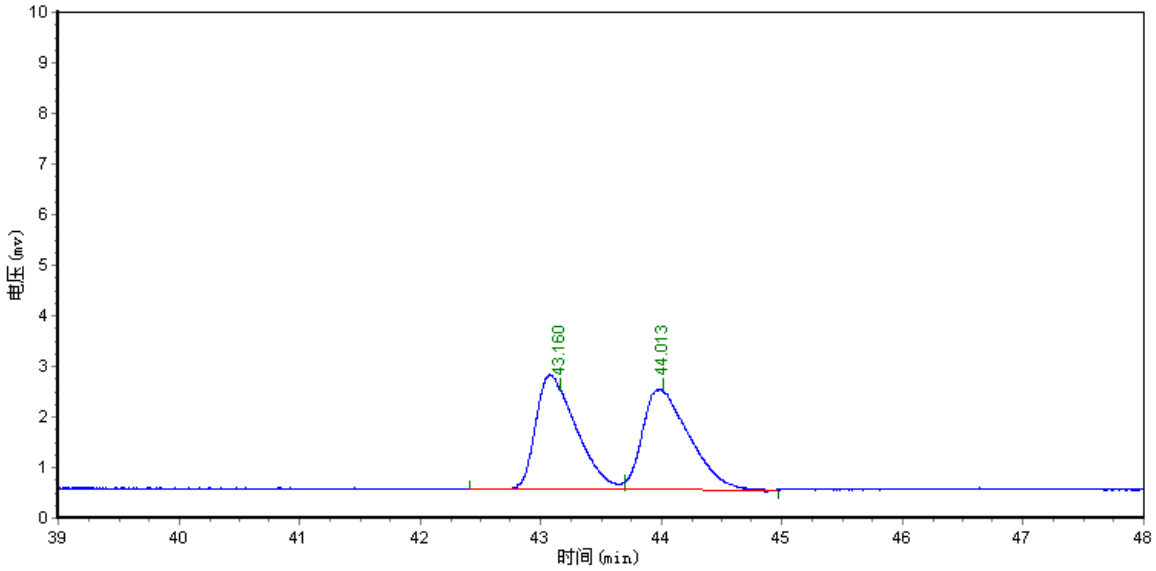
	Name	Retention Time	Area	% Area
1		5.633	5358306	92.89
2		6.090	410197	7.11

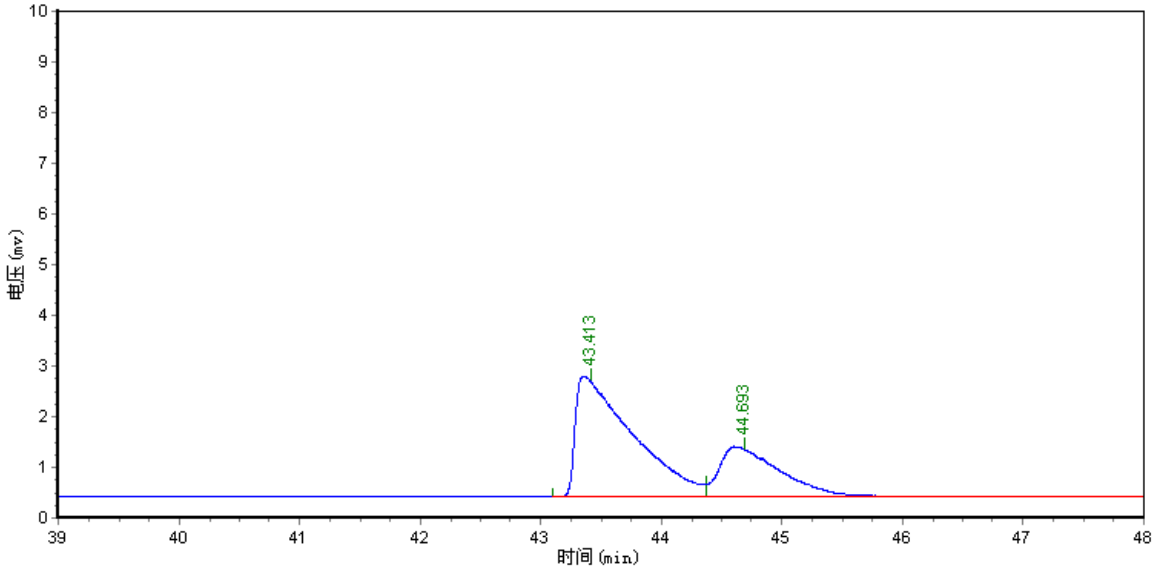



166.69
165.44
141.54
136.93
135.71
134.38
130.39
121.17
84.54
77.37
77.05
76.73
71.26
40.84
32.95
32.17
30.91
25.59
BRUKER
200
180
160
140
120
100
80
60
40
20
0
ppm


O
*
COOEt
Cl


7.268
4.334
4.316
4.298
4.281
2.865
2.847
2.844
2.834
2.829
2.793
2.790
2.478
2.463
2.456
2.442
2.428
2.420
2.147
2.144
1.964
1.952
1.946
1.940
1.933
1.927
1.922
1.918
1.913
1.909
1.899
1.895
1.892
1.890
1.878
1.777
1.766
1.756
1.604
1.337
1.320
1.302
1.254
0.070
BRUKER
9
8
7
6
5
4
3
2
1
ppm
2.00
2.00
1.01
1.00
3.14
1.07
3.12


电压 (mv)
时间 (min)
43.160
44.013


电压 (mv)
时间 (min)
43.413
44.693