Supporting Information

Metal-free *n*-Et_4NBr-catalyzed radical cyclization of disulfides and alkynes leading to benzothiophenes under mild conditions

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General experimental procedures

All reagents and solvents were obtained from commercial suppliers and used without further purification. Mass analyses and HRMS were obtained on a Finnigan-LCQDECA mass spectrometer. Flash chromatography was performed on silica gel (200 ~ 300 mesh). ^{1}H and ^{13}C NMR data were recorded at 400 and 100 MHz on a BRUKER 400 spectrometer. Chemical shifts (δ) are expressed in parts per million (ppm) coupling constants (J) are in Hz. Proton and carbon magnetic resonance spectra (^{1}H NMR and ^{13}C NMR) were recorded using tetramethylsilane (TMS) in the solvent of $CDCl_3$ as the internal standard (^{1}H NMR: TMS at 0.00 ppm, $CDCl_3$ at 7.28 ppm; ^{13}C NMR: $CDCl_3$ at 77.0 ppm).

Kinetic Isotopic Effect (KIE) Studies:

1a-*d5* were synthesized deuterium substrates according the literature procedure.

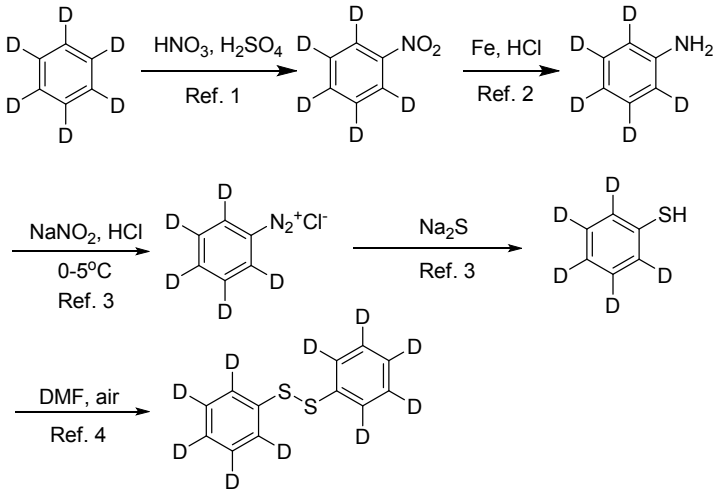
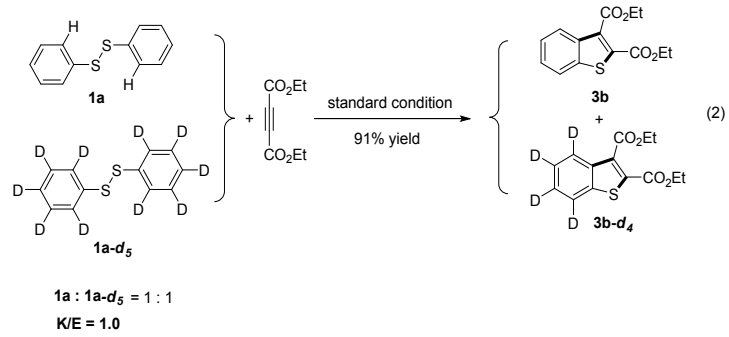


Figure S1 General synthesis route for preparation of **1a**-d_5

A 25 mL Schlenk tube equipped with a magnetic stirring bar was charged with TEAB (10.5 mg), potassium persulfate(1.1 mmol), 1,2-diphenyldisulfane disulfides (**1a**) (0.25 mmol), **1a**-d_5(0.25 mmol) and diethyl but-2-ynedioate (**2b**) (1.7 mmol). The tube was evacuated twice and backfilled with nitrogen, and DCE (1.5 mL) was added to the tube under nitrogen atmosphere. The tube was sealed with a balloon and then the mixture was allowed to stir under nitrogen atmosphere at 90 ^{o}C for 24 h. After completion of the reaction, the resulting solution was cooled to room temperature, and the solvent was removed with the aid of a rotary evaporator. The residue was purified

by column chromatography on silica gel using petroleum ether/ethyl acetate as eluent to provide the desired product , the product was analyzed by ^{1}H-NMR (400 MHz) (Figure S2). The result was summarized in equation S1:



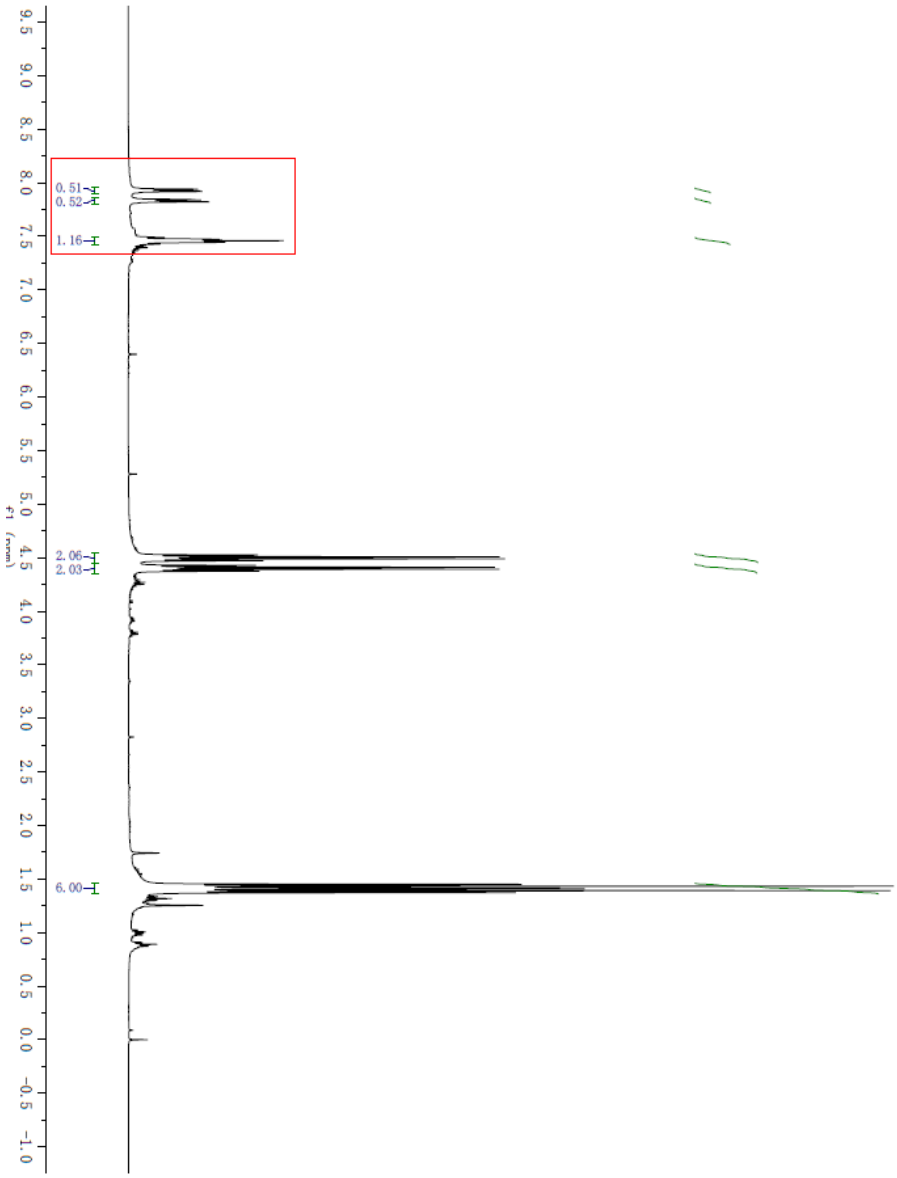


Figure S2 The 1H-NMR spectrum of 3b and 3b-d_4

General procedure for synthesis of substituted benzothiophenes:

A 25 mL Schlenk tube equipped with a magnetic stirring bar was charged with TEAB (10.5 mg), potassium persulfate(1.1 mmol), substituted various disulfides (**1**) (0.5 mmol) and alkynes (**2**) (1.7 mmol). The tube was evacuated twice and backfilled with nitrogen, and DCE (1.5 mL) was added to the tube under nitrogen atmosphere. The tube was sealed with a balloon and then the mixture was allowed to stir under nitrogen atmosphere at 90 ^{o}C for 24 h. After completion of the reaction, the resulting solution was cooled to room temperature, and the solvent was removed with the aid of a rotary evaporator. The residue was purified by column chromatography on silica gel using petroleum ether/ethyl acetate as eluent to provide the desired product (**3**).

References

[1] M. P. Mehn, S. D. Brown, D. M. Jenkins, J. C. Peters, L. Que, *Inorg. Chem.,* 2006, 7417-7427

[2] R.S. Downing, P.J. Kunkeler, H.V. Bekkum, *Catalysis Today.,* 1997, **37***,* 121-136.

[3] A. Khazaei, M. Kazem-Rostami, A. R. Moosavi-Zare, M. Bayat, S. Saednia. *Synlett.*, 2012, **23**, 1893-1896.

[4] J. L. G. Ruano, A. Parra, J. Alemán, *Green Che*m., 2008, **10**, 706-711.

Crystal preparation and X-ray diffraction analysis of compound 3b

Crystal preparation of compound 3b.

Compound **3b** (20 mg) was dissolved in 5 mL of diethyl ether, and it was crystallized to give crystal as colorless prisms after the solvent was slowly volatilized in 2 days at room temperature (~ 25 °C).

X-Ray diffraction analysis of compound 3b.

Table 1. Crystal data and structure refinement for a.

Identification code	a
Empirical formula	C_{14} H_{14} O_4 S
Formula weight	278.31
Temperature	295(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, P21/n
Unit cell dimensions	a = 10.7820(18) A alpha = 90 deg.
	b = 10.5128(17) A beta = 106.290(2) deg.
	c = 12.334(2) A gamma = 90 deg.
Volume	1341.9(4) A^3
Z, Calculated density	4, 1.378 Mg/m^3
Absorption coefficient	0.248 mm^-1
F(000)	584
Crystal size	0.36 x 0.28 x 0.20 mm
Theta range for data collection	2.22 to 25.99 deg.
Limiting indices	-13<=h<=13, -12<=k<=12, -15<=l<=14
Reflections collected / unique	10113 / 2639 [R(int) = 0.0256]
Completeness to theta = 25.99	100.0 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9521 and 0.9160
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2639 / 0 / 173

Goodness-of-fit on F^2	1.048
Final R indices [I>2sigma(I)]	R1 = 0.0442, wR2 = 0.1241
R indices (all data)	R1 = 0.0504, wR2 = 0.1302
Extinction coefficient	0.015(2)
Largest diff. peak and hole	0.542 and -0.243 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for a. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

x	y	z	U(eq)	
C(1)	1194(2)	10568(2)	1340(2)	42(1)
C(2)	1380(2)	11881(2)	1256(2)	51(1)
C(3)	645(2)	12691(2)	1684(2)	56(1)
C(4)	-273(2)	12236(2)	2194(2)	55(1)
C(5)	-460(2)	10955(2)	2281(2)	48(1)
C(6)	277(2)	10099(2)	1851(1)	39(1)
C(7)	248(2)	8735(2)	1841(1)	40(1)
C(8)	-661(2)	7995(2)	2319(2)	46(1)
C(9)	-825(3)	6765(2)	3887(2)	67(1)
C(10)	-823(3)	5428(2)	3511(2)	77(1)
C(11)	1098(2)	8221(2)	1338(2)	40(1)
C(12)	1229(2)	6844(2)	1161(2)	43(1)
C(13)	2247(2)	5235(2)	410(2)	54(1)
C(14)	3190(3)	5118(2)	-245(2)	75(1)
O(1)	-1781(2)	7870(2)	1850(2)	72(1)
O(2)	-68(2)	7567(1)	3343(1)	55(1)
O(3)	576(1)	6059(1)	1448(1)	58(1)
O(4)	2117(1)	6587(1)	639(1)	51(1)

S(1)	1983(1)	9355(1)	858(1)	47(1)

Table 3. Bond lengths [A] and angles [deg] for a.

C(1)-C(2)	1.403(3)
C(1)-C(6)	1.403(2)
C(1)-S(1)	1.7284(18)
C(2)-C(3)	1.367(3)
C(2)-H(2)	0.9300
C(3)-C(4)	1.398(3)
C(3)-H(3)	0.9300
C(4)-C(5)	1.370(3)
C(4)-H(4)	0.9300
C(5)-C(6)	1.399(3)
C(5)-H(5)	0.9300
C(6)-C(7)	1.434(3)
C(7)-C(11)	1.356(2)
C(7)-C(8)	1.495(2)
C(8)-O(1)	1.191(2)
C(8)-O(2)	1.324(2)
C(9)-O(2)	1.461(2)
C(9)-C(10)	1.480(3)
C(9)-H(9A)	0.9700
C(9)-H(9B)	0.9700
C(10)-H(10A)	0.9600
C(10)-H(10B)	0.9600
C(10)-H(10C)	0.9600
C(11)-C(12)	1.476(3)
C(11)-S(1)	1.7316(18)
C(12)-O(3)	1.200(2)

C(12)-O(4)	1.323(2)
C(13)-O(4)	1.463(2)
C(13)-C(14)	1.470(3)
C(13)-H(13A)	0.9700
C(13)-H(13B)	0.9700
C(14)-H(14A)	0.9600
C(14)-H(14B)	0.9600
C(14)-H(14C)	0.9600
C(2)-C(1)-C(6)	120.82(18)
C(2)-C(1)-S(1)	127.30(15)
C(6)-C(1)-S(1)	111.87(13)
C(3)-C(2)-C(1)	118.34(19)
C(3)-C(2)-H(2)	120.8
C(1)-C(2)-H(2)	120.8
C(2)-C(3)-C(4)	121.38(19)
C(2)-C(3)-H(3)	119.3
C(4)-C(3)-H(3)	119.3
C(5)-C(4)-C(3)	120.6(2)
C(5)-C(4)-H(4)	119.7
C(3)-C(4)-H(4)	119.7
C(4)-C(5)-C(6)	119.47(19)
C(4)-C(5)-H(5)	120.3
C(6)-C(5)-H(5)	120.3
C(5)-C(6)-C(1)	119.36(17)
C(5)-C(6)-C(7)	129.31(16)
C(1)-C(6)-C(7)	111.33(15)
C(11)-C(7)-C(6)	112.74(15)
C(11)-C(7)-C(8)	125.11(16)
C(6)-C(7)-C(8)	122.13(15)
O(1)-C(8)-O(2)	125.35(18)

O(1)-C(8)-C(7)	123.60(18)
O(2)-C(8)-C(7)	110.96(16)
O(2)-C(9)-C(10)	110.7(2)
O(2)-C(9)-H(9A)	109.5
C(10)-C(9)-H(9A)	109.5
O(2)-C(9)-H(9B)	109.5
C(10)-C(9)-H(9B)	109.5
H(9A)-C(9)-H(9B)	108.1
C(9)-C(10)-H(10A)	109.5
C(9)-C(10)-H(10B)	109.5
H(10A)-C(10)-H(10B)	109.5
C(9)-C(10)-H(10C)	109.5
H(10A)-C(10)-H(10C)	109.5
H(10B)-C(10)-H(10C)	109.5
C(7)-C(11)-C(12)	124.26(16)
C(7)-C(11)-S(1)	112.99(13)
C(12)-C(11)-S(1)	122.69(13)
O(3)-C(12)-O(4)	124.52(18)
O(3)-C(12)-C(11)	122.88(17)
O(4)-C(12)-C(11)	112.59(16)
O(4)-C(13)-C(14)	107.83(18)
O(4)-C(13)-H(13A)	110.1
C(14)-C(13)-H(13A)	110.1
O(4)-C(13)-H(13B)	110.1
C(14)-C(13)-H(13B)	110.1
H(13A)-C(13)-H(13B)	108.5
C(13)-C(14)-H(14A)	109.5
C(13)-C(14)-H(14B)	109.5
H(14A)-C(14)-H(14B)	109.5
C(13)-C(14)-H(14C)	109.5

H(14A)-C(14)-H(14C)	109.5
H(14B)-C(14)-H(14C)	109.5
C(8)-O(2)-C(9)	116.80(17)
C(12)-O(4)-C(13)	114.48(15)
C(1)-S(1)-C(11)	91.06(9)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for a. The anisotropic displacement factor exponent takes the form:

-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
C(1)	44(1)	38(1)	42(1)	2(1)	10(1)	-1(1)
C(2)	57(1)	39(1)	57(1)	6(1)	16(1)	-9(1)
C(3)	68(1)	34(1)	64(1)	3(1)	13(1)	-5(1)
C(4)	65(1)	38(1)	62(1)	-3(1)	19(1)	5(1)
C(5)	54(1)	42(1)	52(1)	1(1)	21(1)	1(1)
C(6)	41(1)	36(1)	39(1)	2(1)	10(1)	-1(1)
C(7)	41(1)	36(1)	43(1)	1(1)	12(1)	-1(1)
C(8)	49(1)	32(1)	64(1)	0(1)	24(1)	1(1)
C(9)	93(2)	49(1)	74(1)	12(1)	46(1)	-6(1)
C(10)	87(2)	55(1)	96(2)	11(1)	41(2)	-3(1)
C(11)	40(1)	35(1)	45(1)	1(1)	12(1)	-2(1)
C(12)	43(1)	40(1)	47(1)	-3(1)	13(1)	1(1)
C(13)	59(1)	40(1)	67(1)	-6(1)	25(1)	1(1)
C(14)	88(2)	55(1)	98(2)	-10(1)	53(2)	5(1)
O(1)	45(1)	73(1)	99(1)	21(1)	20(1)	-6(1)
O(2)	67(1)	48(1)	54(1)	7(1)	24(1)	-9(1)
O(3)	66(1)	40(1)	80(1)	-6(1)	37(1)	-7(1)

O(4)	53(1)	40(1)	66(1)	-6(1)	26(1)	1(1)
S(1)	47(1)	43(1)	55(1)	2(1)	23(1)	-2(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for a.

_	x	y	z	U(eq)
H(2)	1989	12191	918	61
H(3)	758	13564	1635	68
H(4)	-762	12808	2478	66
H(5)	-1071	10657	2622	58
H(9A)	-1707	7076	3701	81
H(9B)	-465	6808	4700	81
H(10A)	-1326	4917	3875	115
H(10B)	49	5116	3706	115
H(10C)	-1189	5383	2707	115
H(13A)	1419	4890	-15	65
H(13B)	2543	4768	1114	65
H(14A)	3291	4238	-408	113
H(14B)	4006	5457	186	113
H(14C)	2887	5583	-939	113

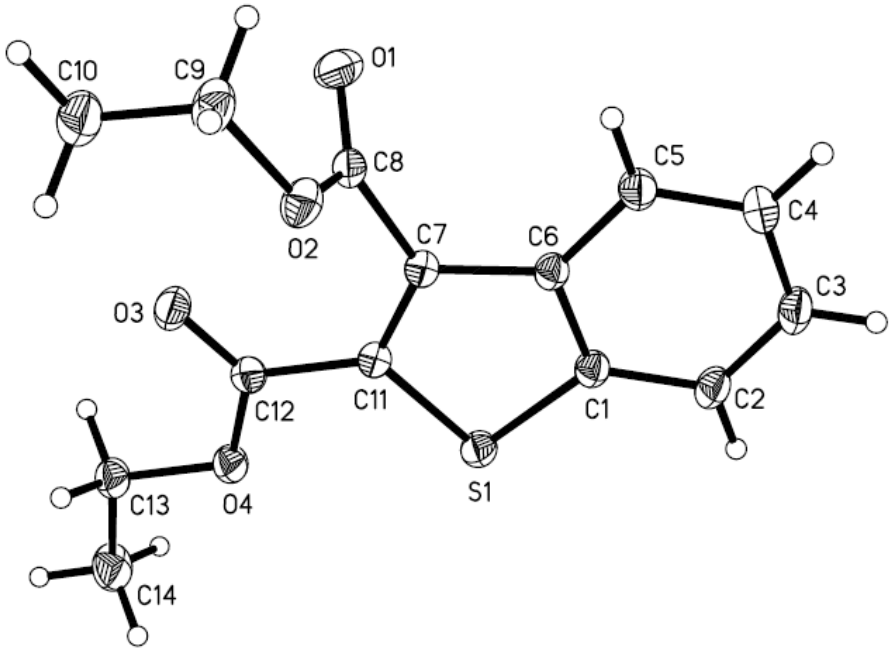


Figure 1. ORTEP drawing of $C_{14}H_{14}O_4S$ with 50% probability ellipsoids, showing the atomic numbering scheme.

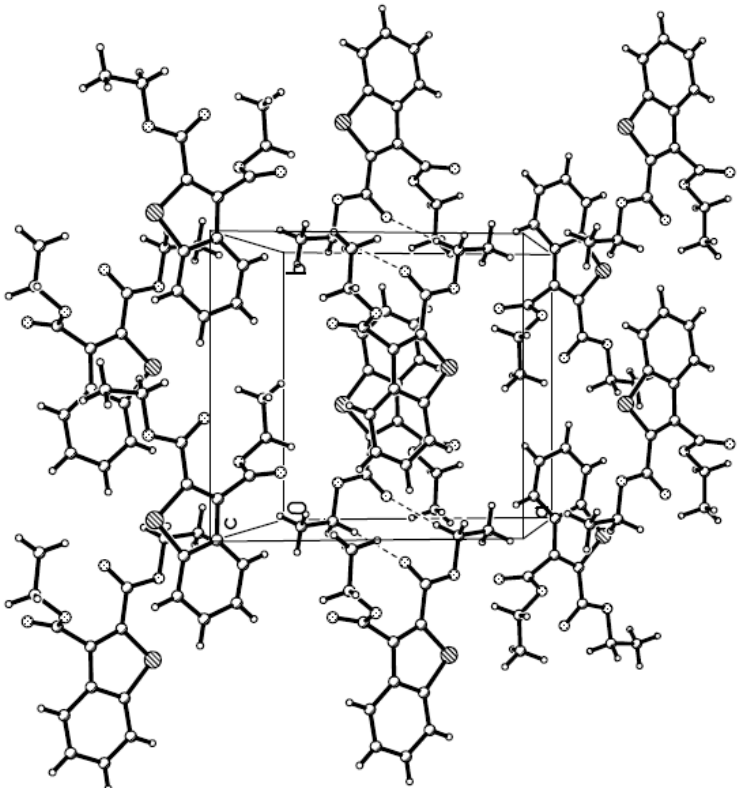
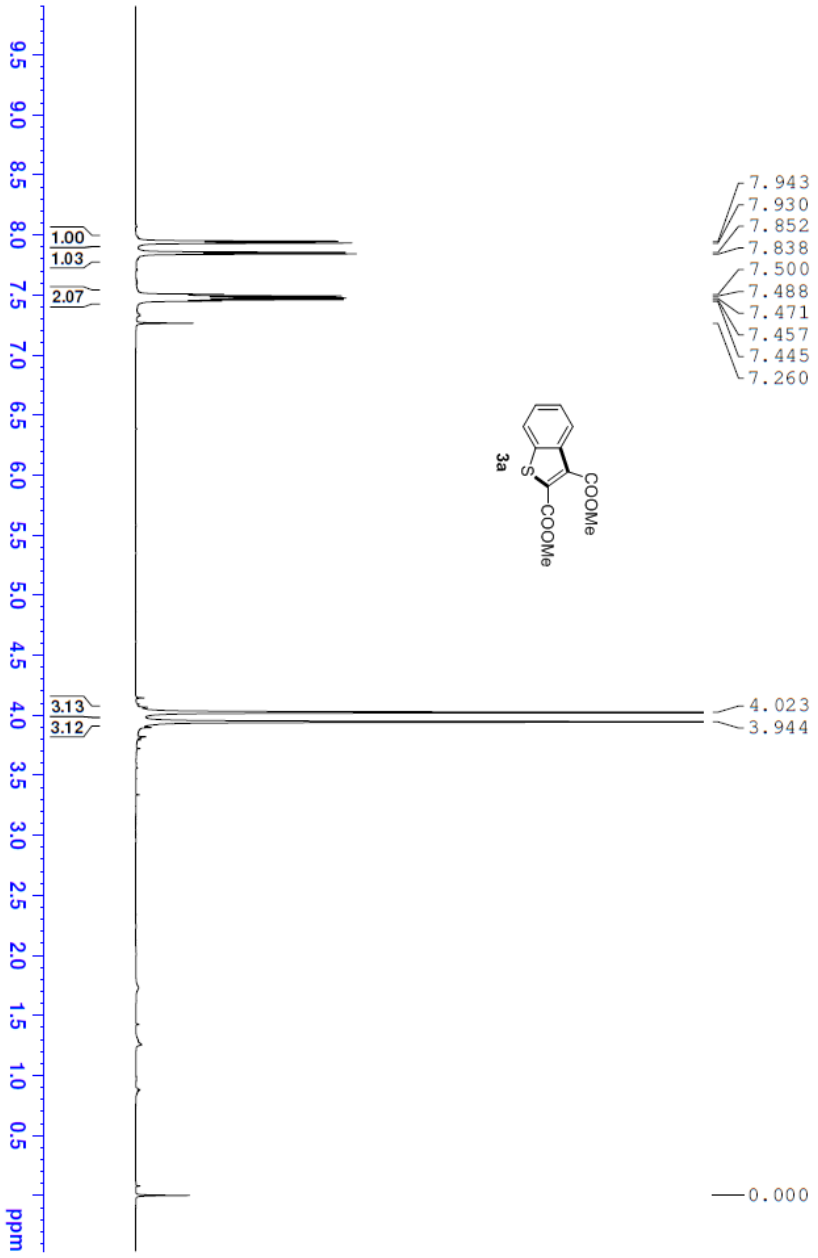


Figure 2. A packing view along the *a* direction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1.03
2.07
3.13
3.12
7.943
7.930
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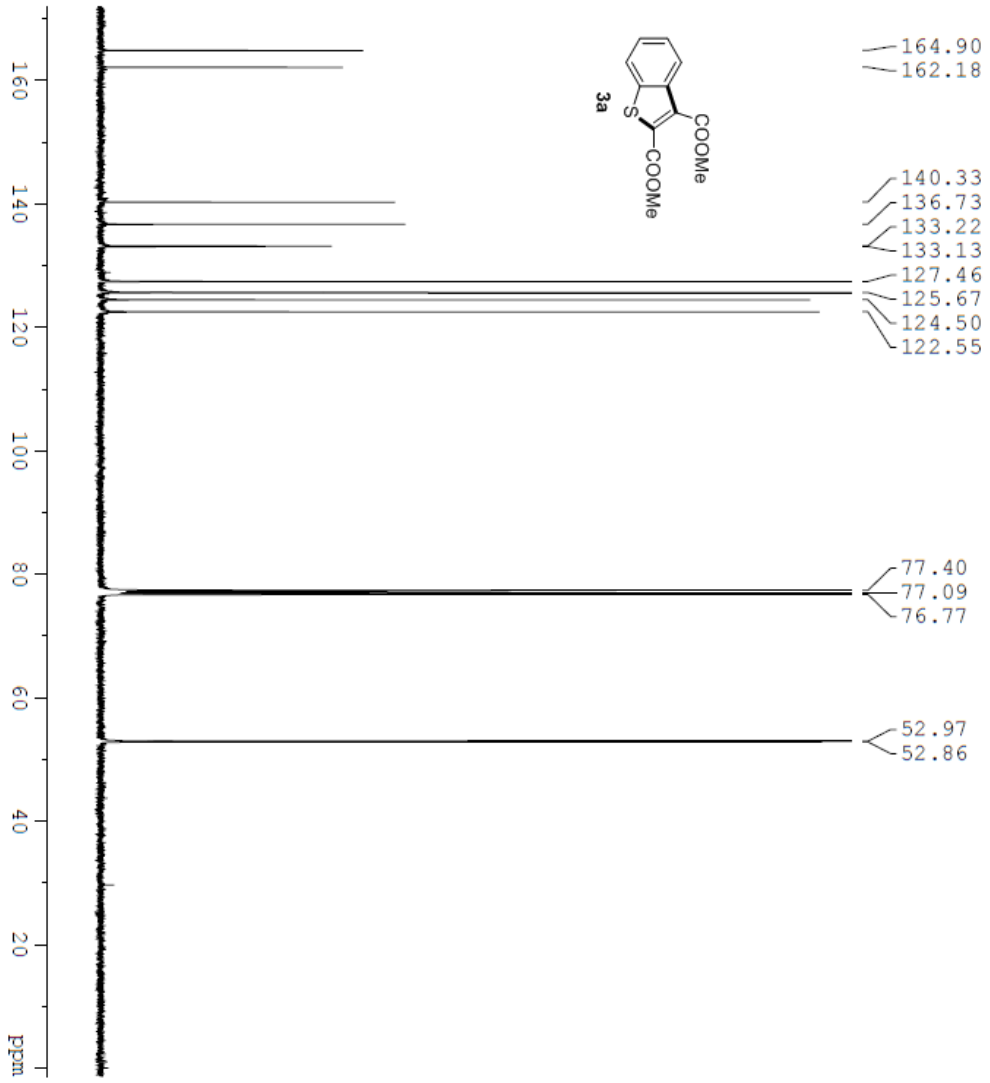
COOMe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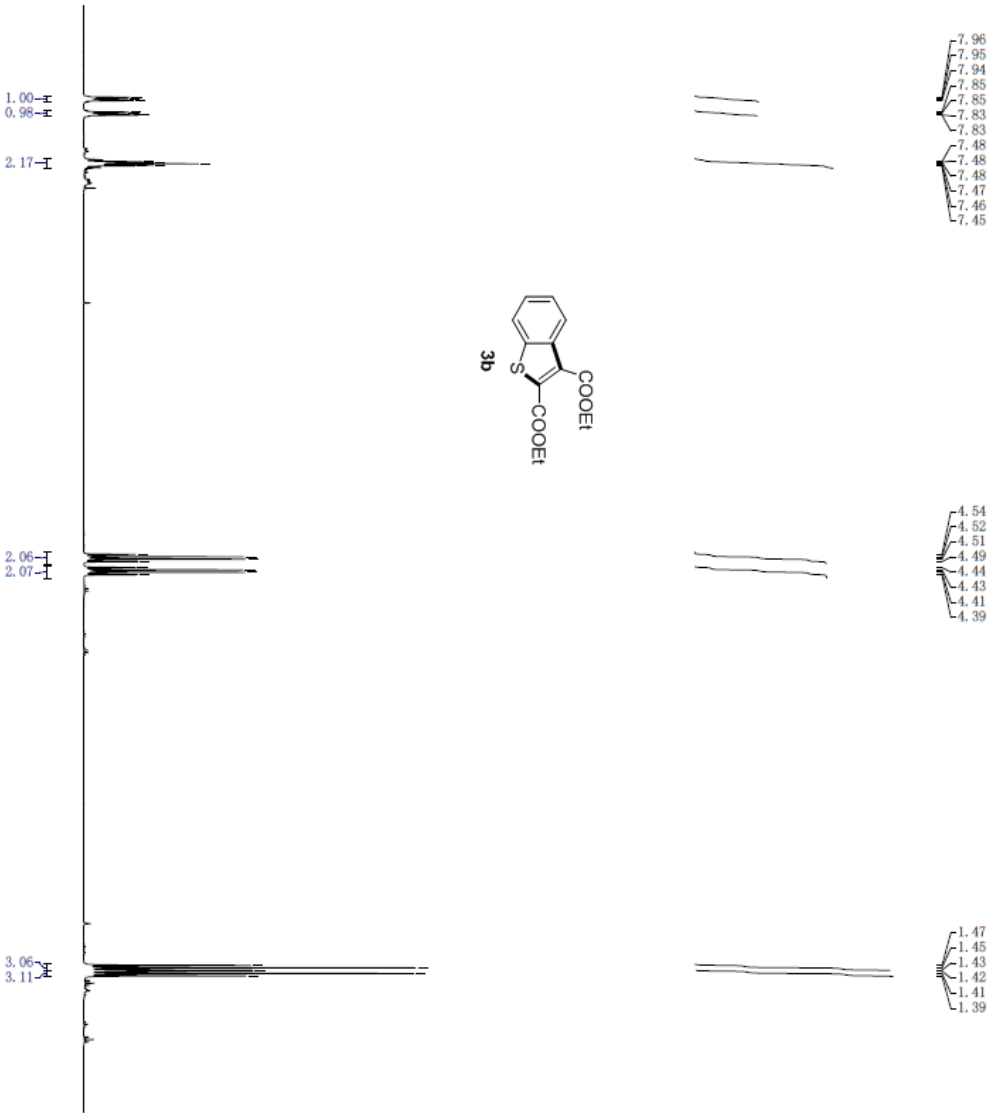
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4.49
4.44
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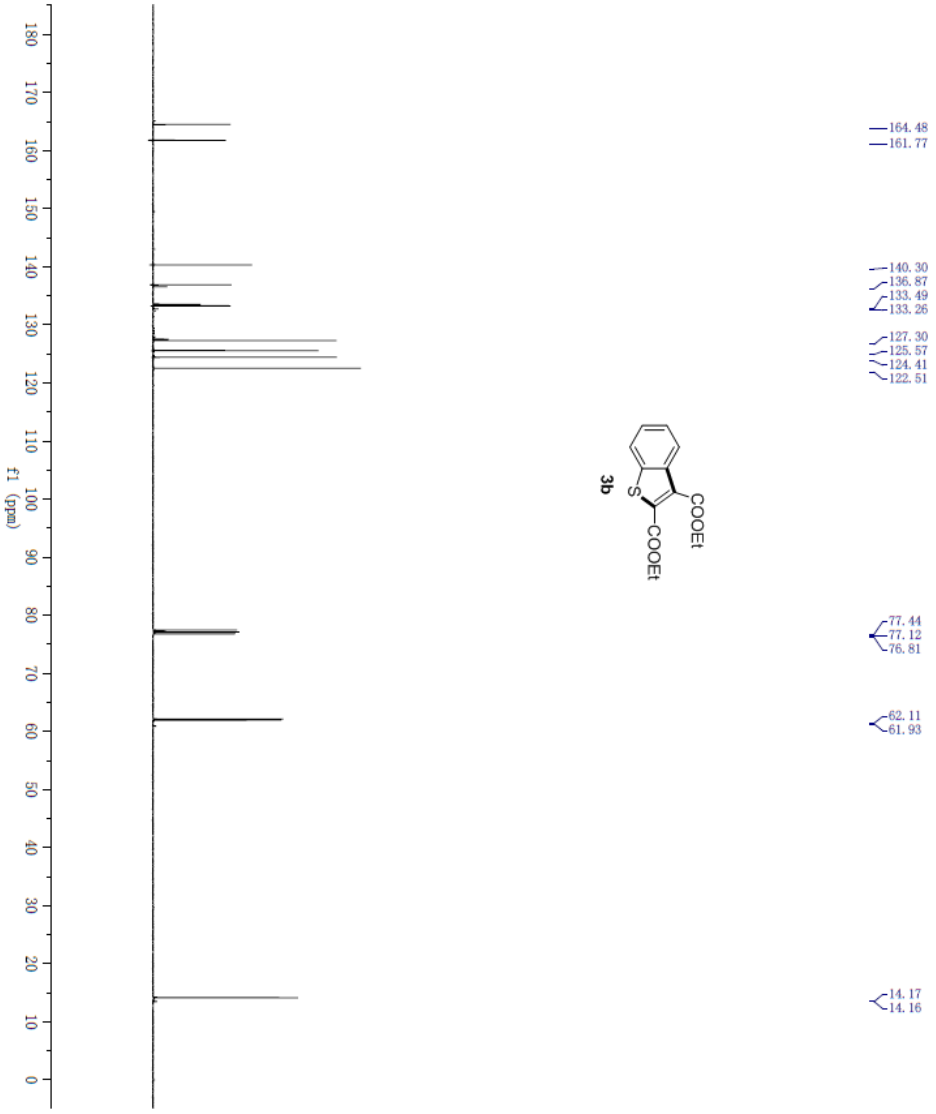
COOEt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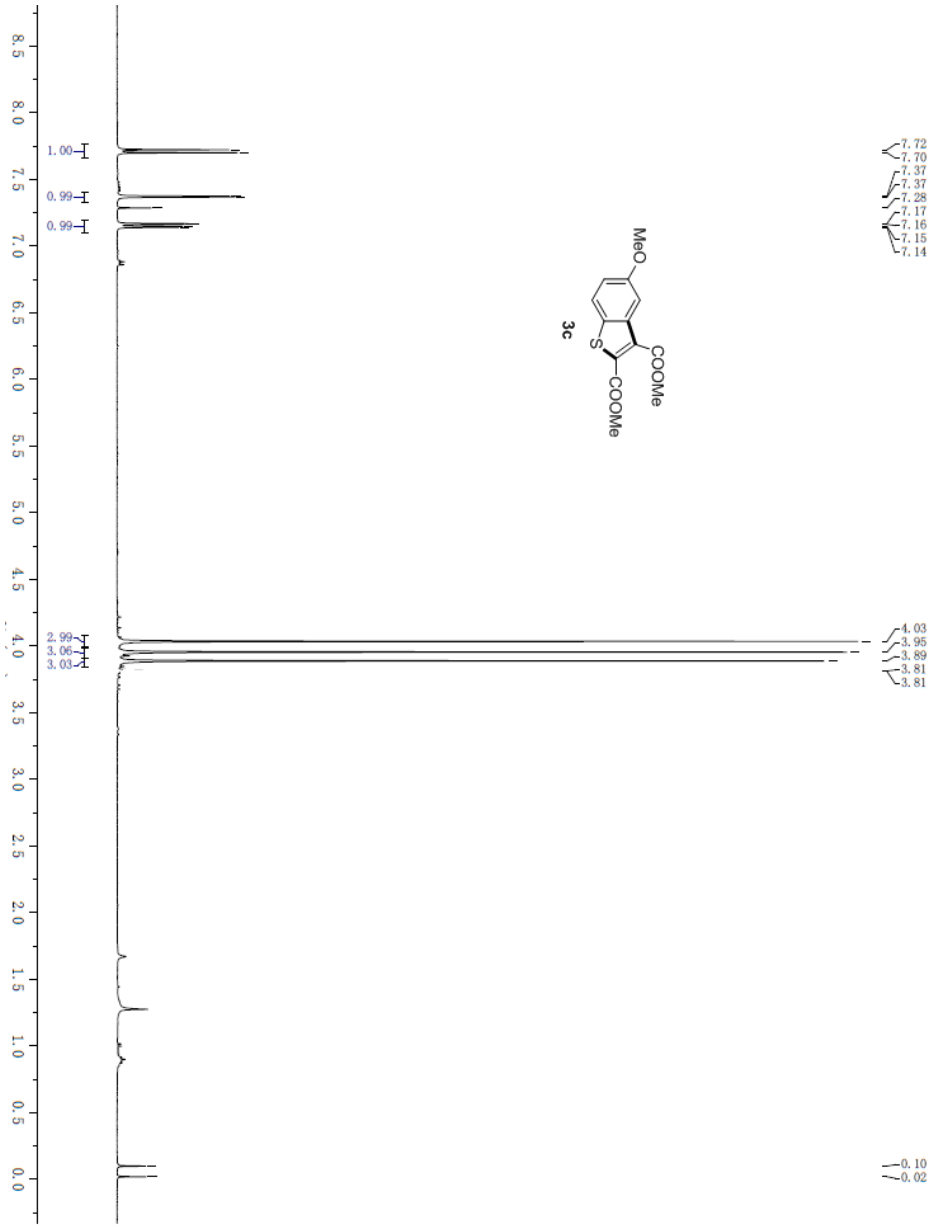
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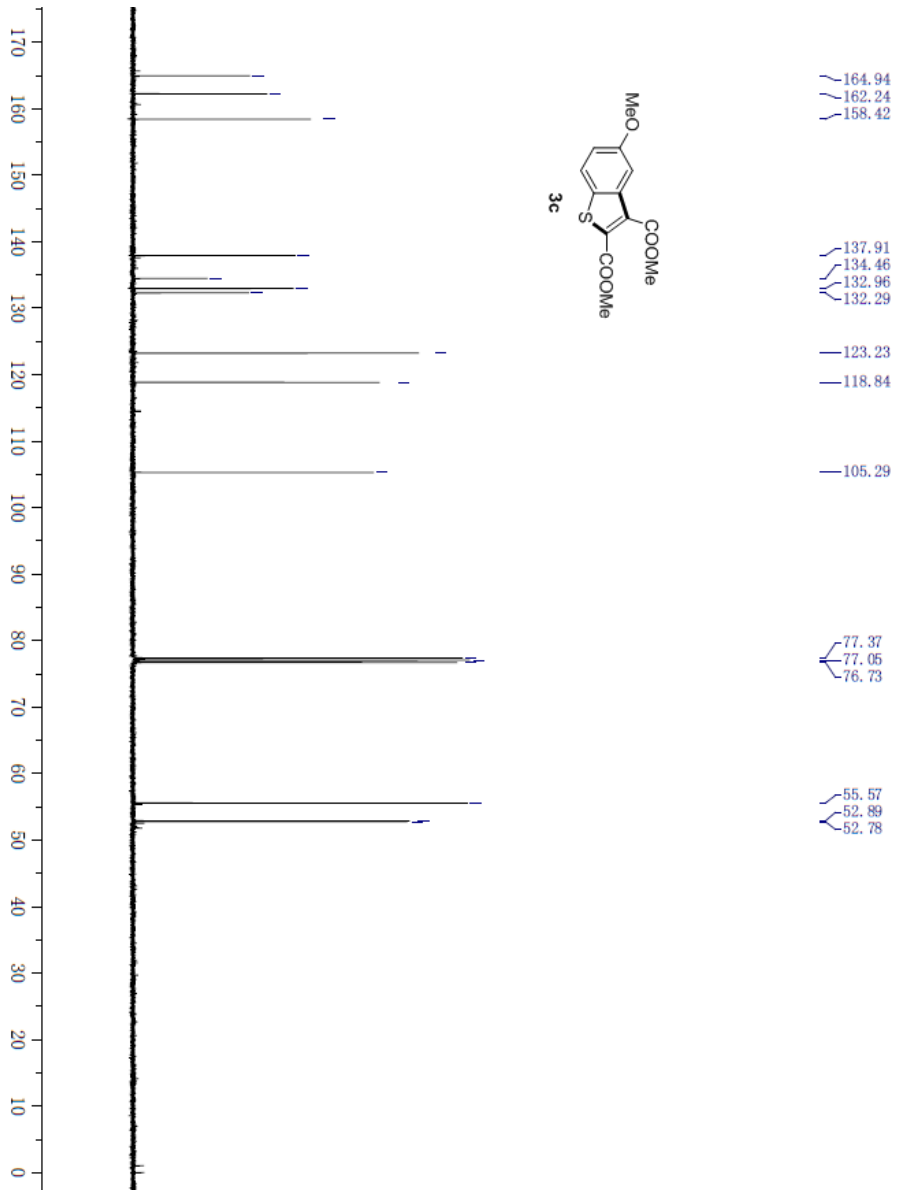
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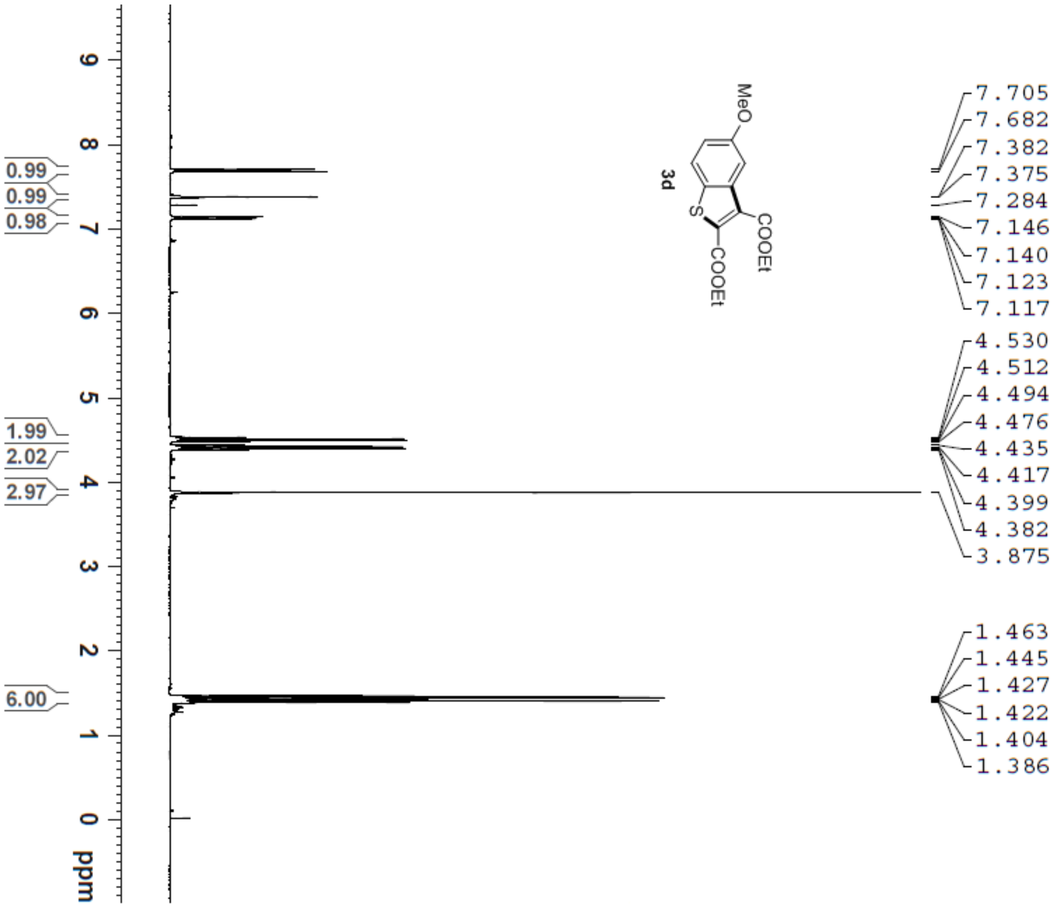
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1.427
1.422
1.404
1.386
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0.99
0.98
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9
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7
6
5
4
3
2
1
0
ppm

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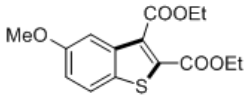

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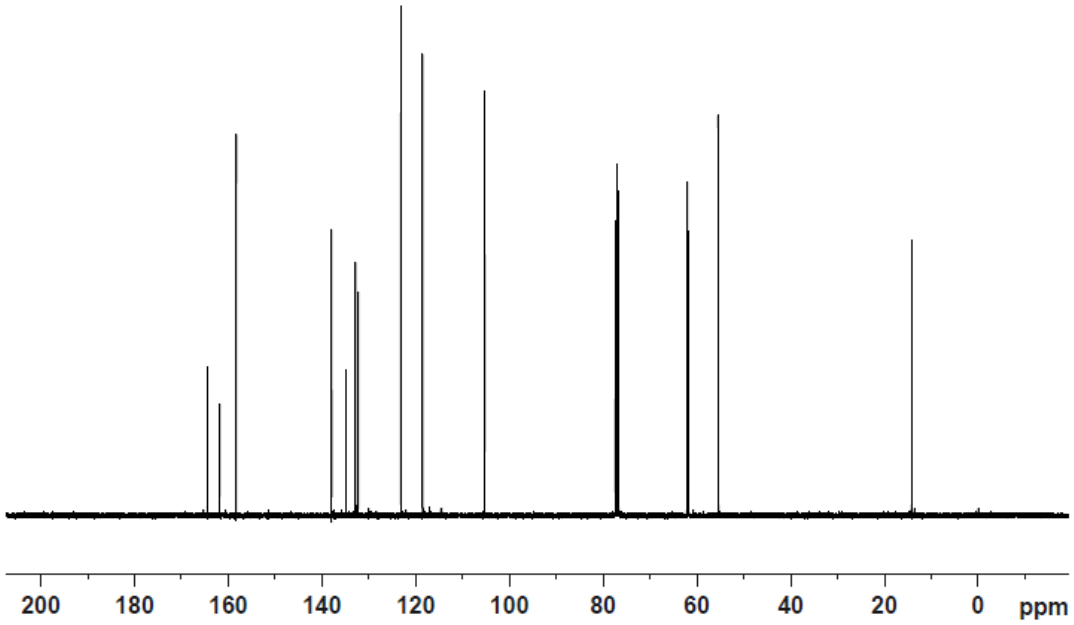

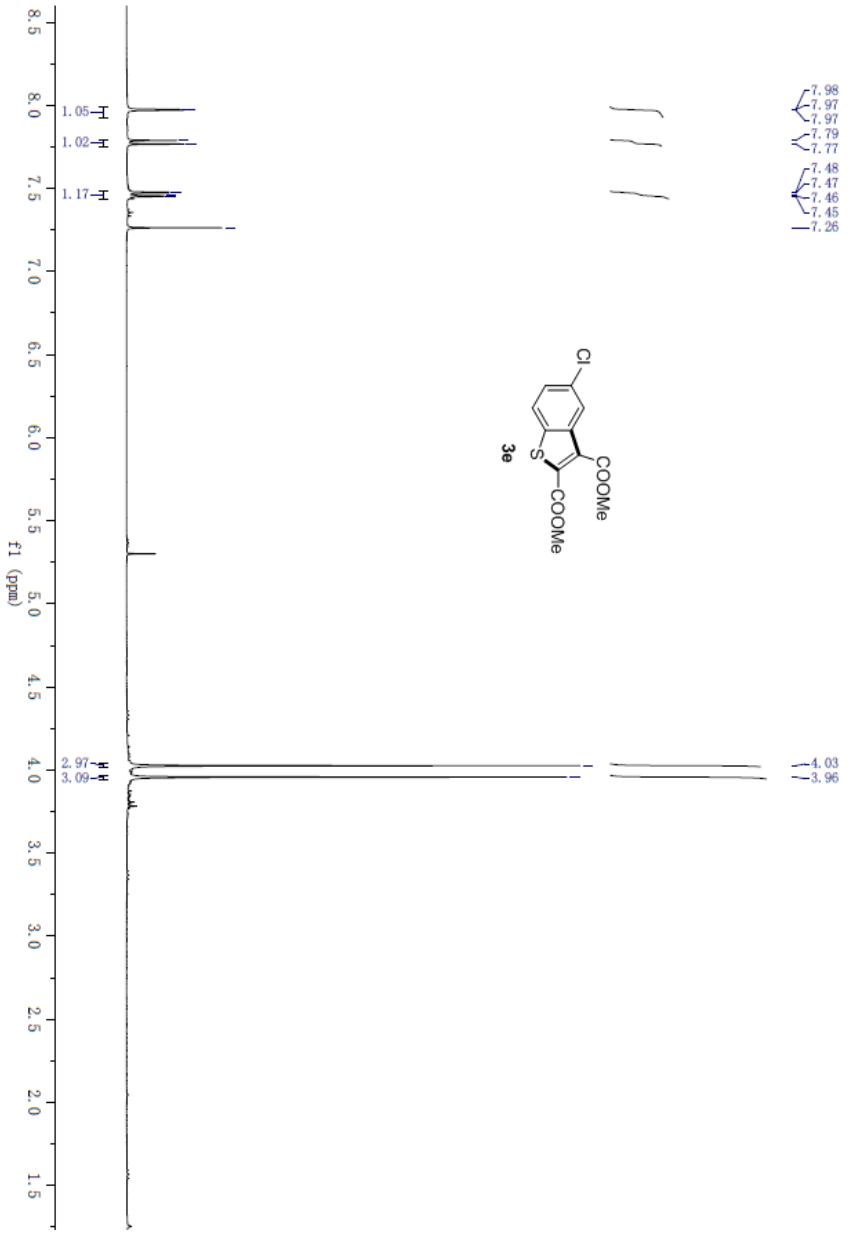
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1.5
f1 (ppm)
1.05
1.02
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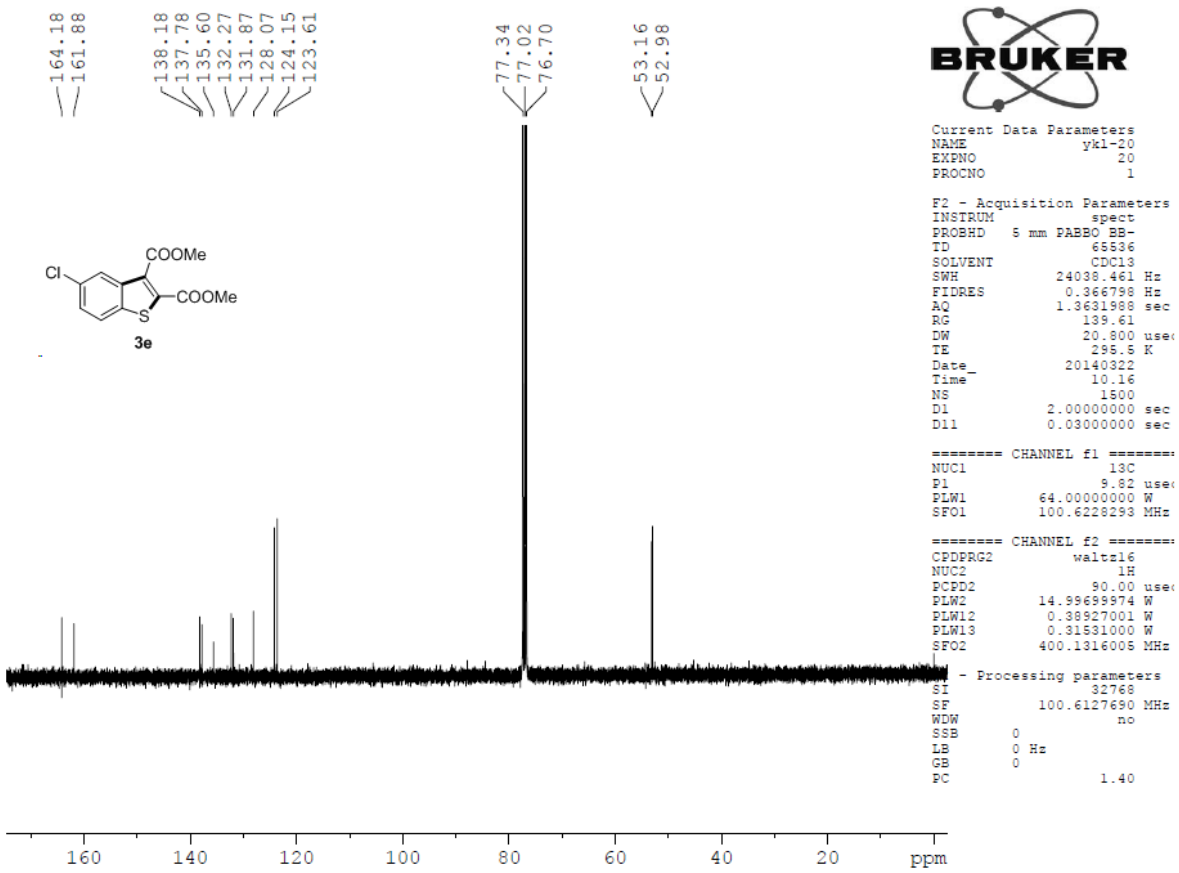
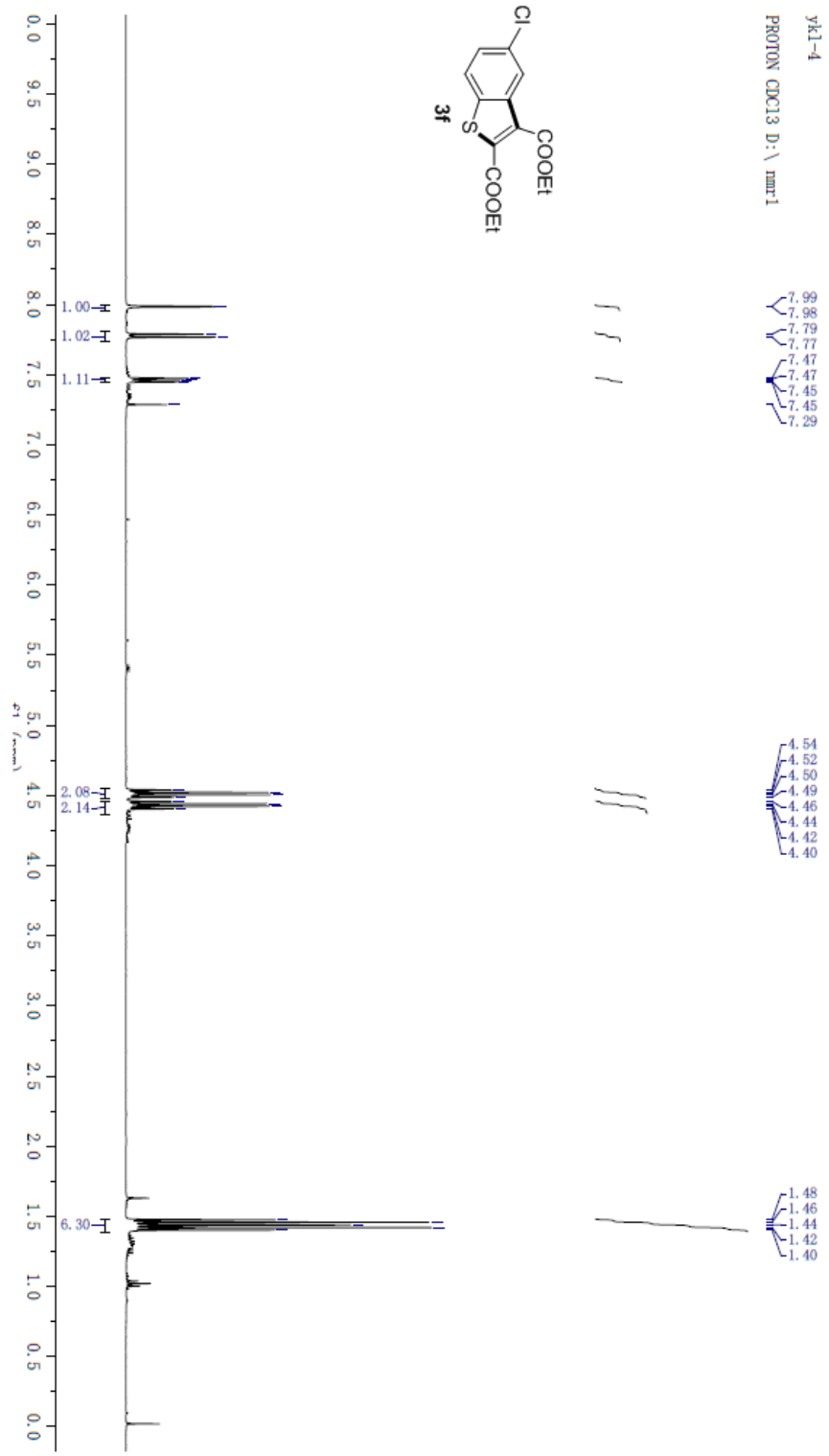
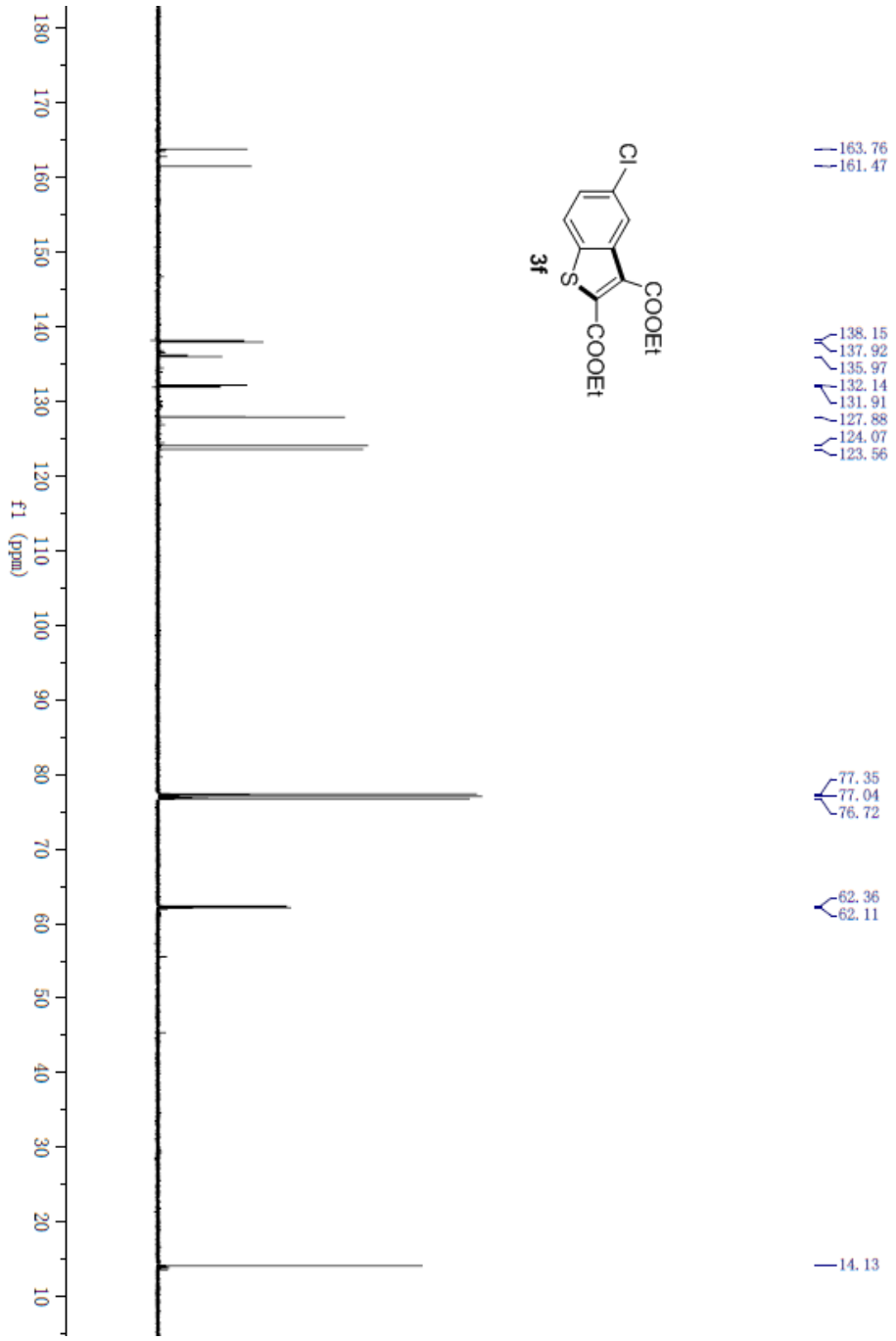
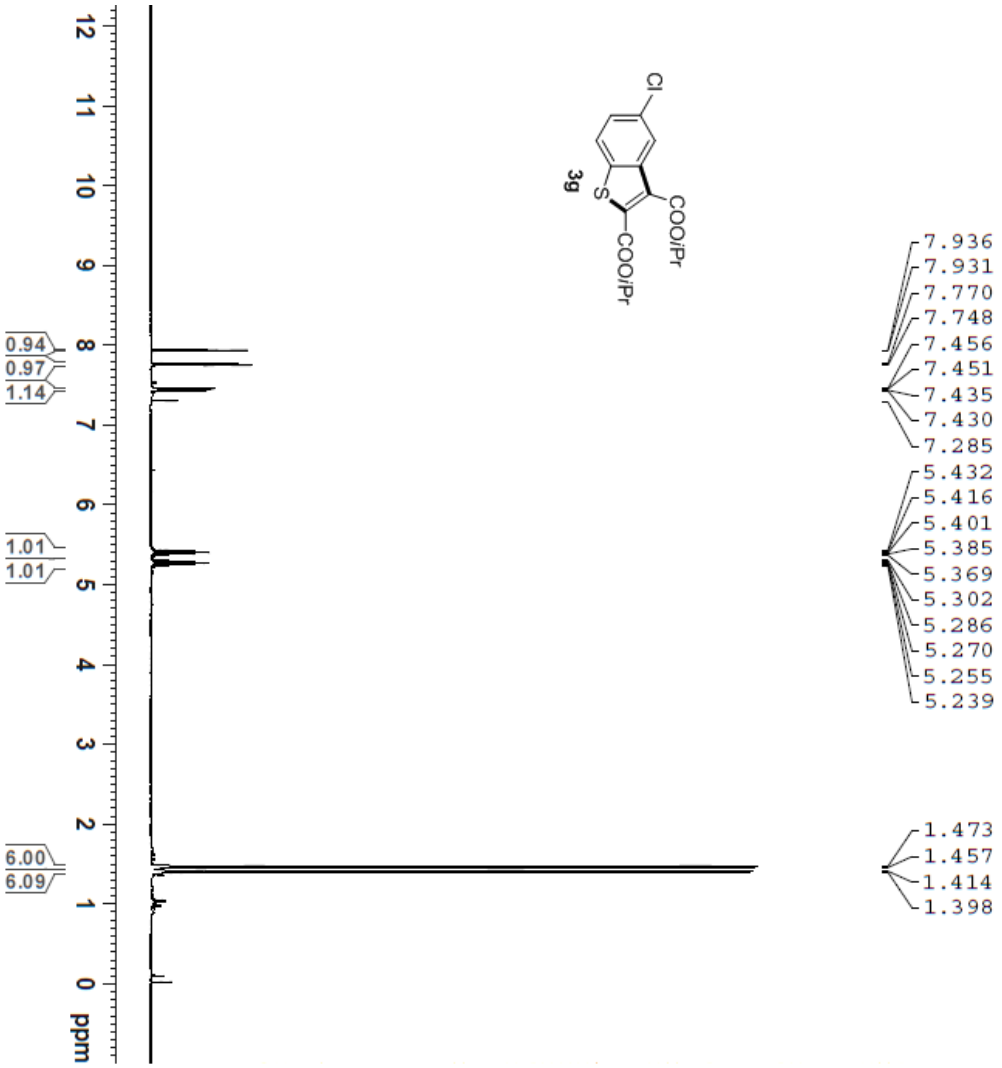
3.09
7.98
7.97
7.97
7.79
7.77
7.48
7.47
7.46
7.45
7.26
4.03
3.96
Cl
S
COOMe
COOMe
3e

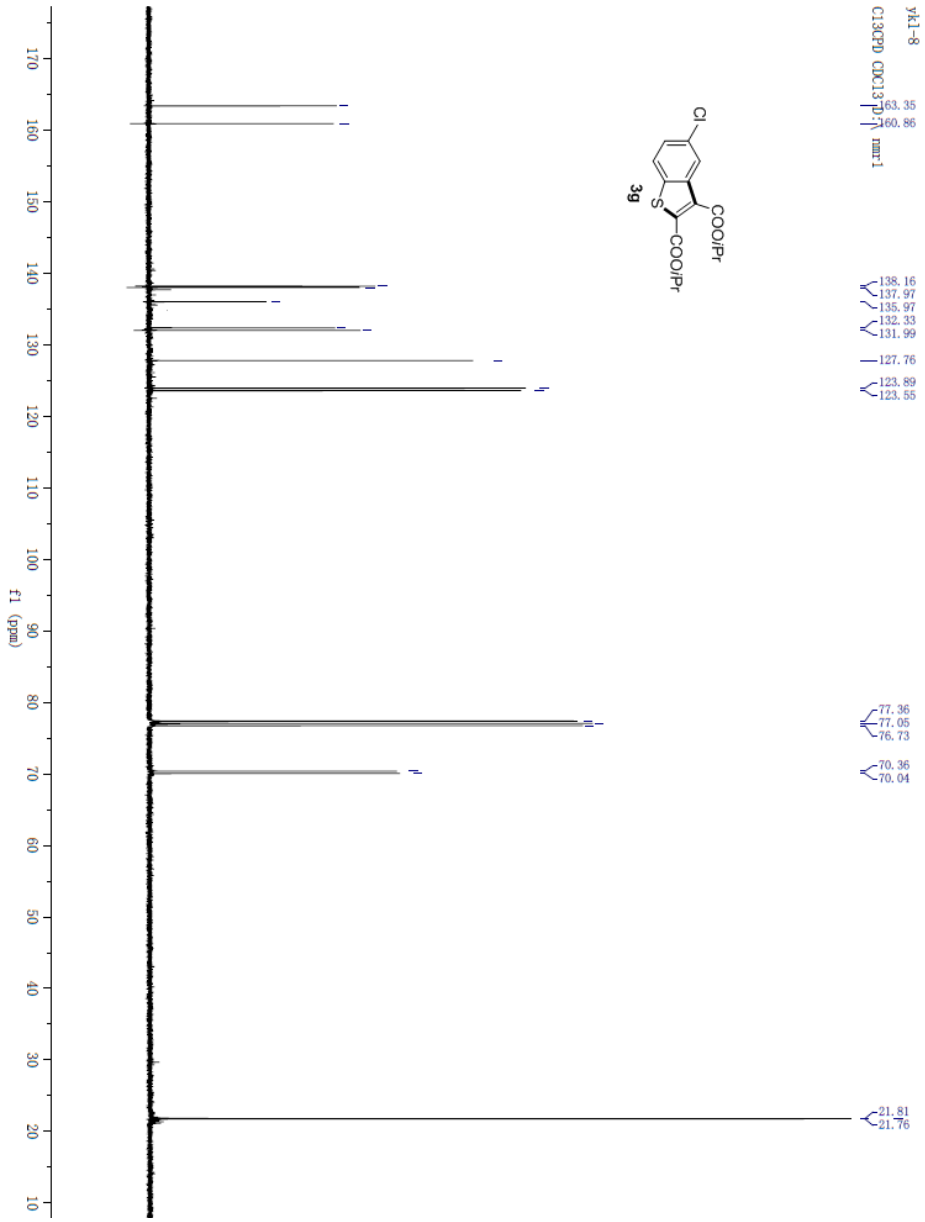


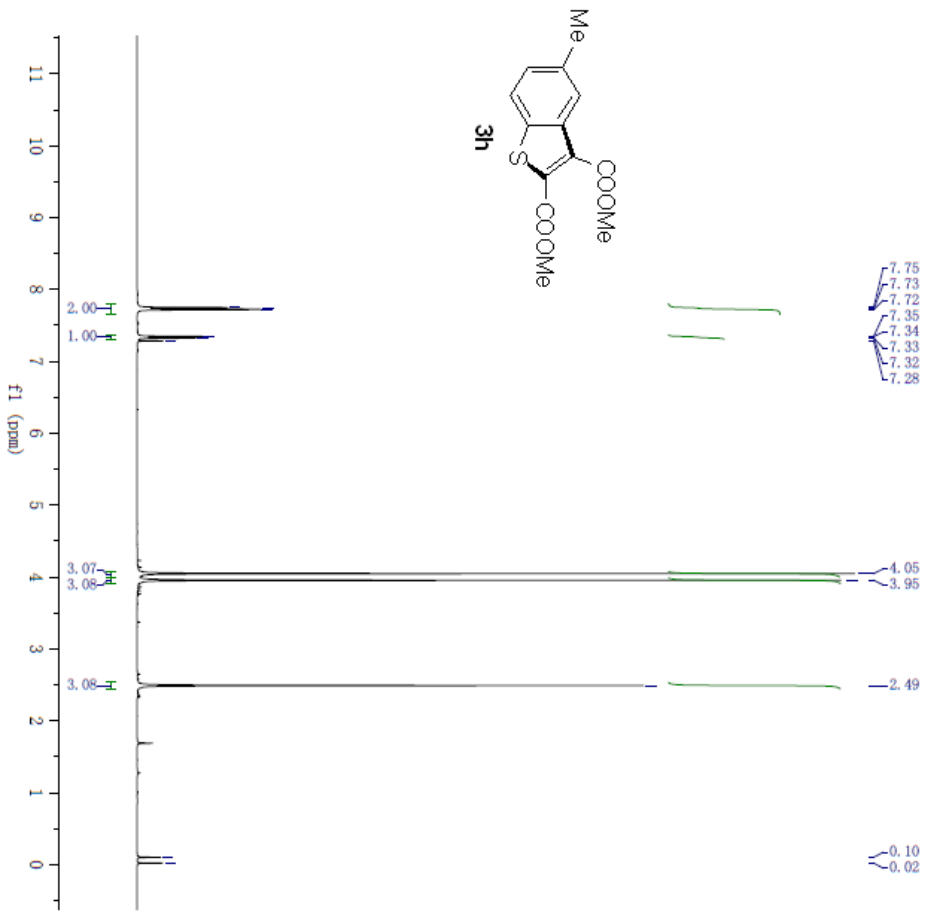

```
Current Data Parameters
NAME                  ykl-20
EXPNO                     20
PROCNO                     1

F2 - Acquisition Parameters
INSTRUM                 spect
PROBHD    5 mm PABBO BB-
TD                      65536
SOLVENT                 CDCl3
SWH                24038.461 Hz
FIDRES                 0.366798 Hz
AQ                     1.3631988 sec
RG                       139.61
DW                        20.800 use
TE                       295.5 K
Date_                 20140322
Time                      10.16
NS                         1500
D1                 2.00000000 sec
D11                0.03000000 sec

======== CHANNEL f1 ========
NUC1                       13C
P1                          9.82 use
PLW1              64.00000000 W
SFO1             100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                     90.00 use
PLW2              14.99699974 W
PLW12              0.38927001 W
PLW13              0.31531000 W
SFO2             400.1316005 MHz

F2 - Processing parameters
SI                         32768
SF               100.6127690 MHz
WDW                           no
SSB          0
LB           0 Hz
GB           0
PC                         1.40
```

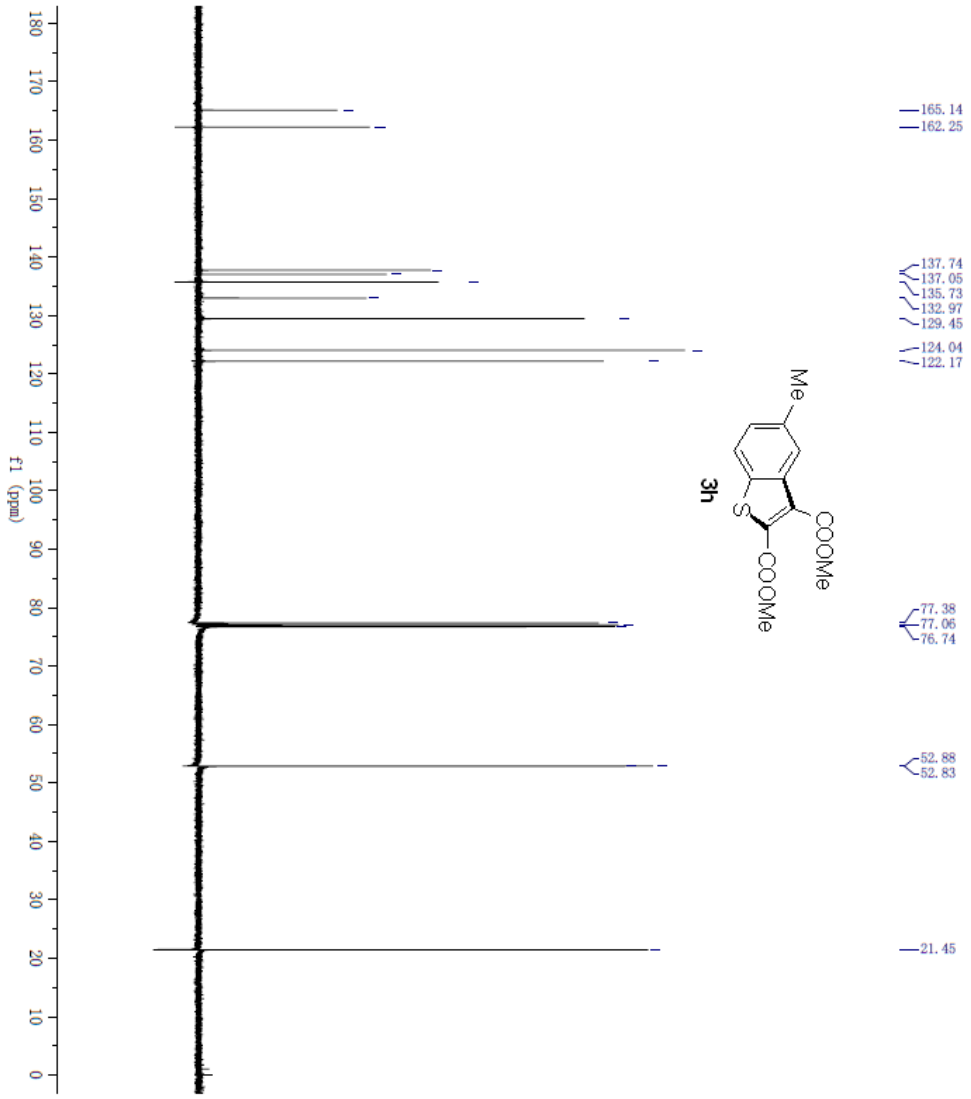

yk1-4
PROTON CDC13 D:\ nmr1
3f
Cl
COOEt
COOEt
7.99
7.98
7.79
7.77
7.47
7.47
7.45
7.45
7.29
4.54
4.52
4.50
4.49
4.46
4.44
4.42
4.40
1.48
1.46
1.44
1.42
1.40
1.00
1.02
1.11
2.08
2.14
6.30
f1 (ppm)

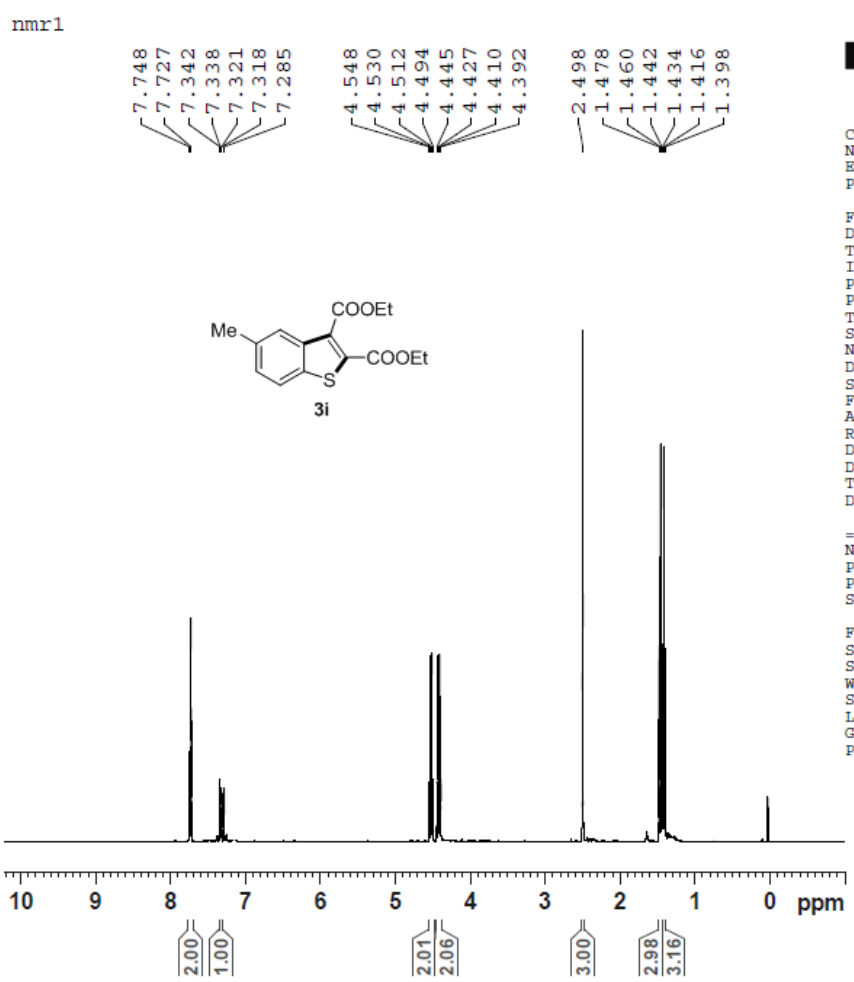

163.76
161.47
138.15
137.92
135.97
132.14
131.91
127.88
124.07
123.56
77.35
77.04
76.72
62.36
62.11
14.13
Cl
COOEt
COOEt
S
3f
f1 (ppm)


3g
Cl
COOiPr
COOiPr
7.936
7.931
7.770
7.748
7.456
7.451
7.435
7.430
7.285
5.432
5.416
5.401
5.385
5.369
5.302
5.286
5.270
5.255
5.239
1.473
1.457
1.414
1.398
0.94
0.97
1.14
1.01
1.01
6.00
6.09
ppm


yk1-8
C13CPD CDCl3 25.
1H nmr1
163.35
160.86
138.16
137.97
135.97
132.33
131.99
127.76
123.89
123.55
77.36
77.05
76.73
70.36
70.04
21.81
21.76
Cl
S
COOiPr
COOiPr
3g
f1 (ppm)


Me
3h
S
COOMe
COOMe
f1 (ppm)
2.00
1.00
3.07
3.08
3.08
7.75
7.73
7.72
7.35
7.34
7.33
7.32
7.28
4.05
3.95
2.49
0.10
0.02


165.14
162.25
137.74
137.05
135.73
132.97
129.45
124.04
122.17
77.38
77.06
76.74
52.88
52.83
21.45
f1 (ppm)
3h
Me
COOMe
COOMe
S



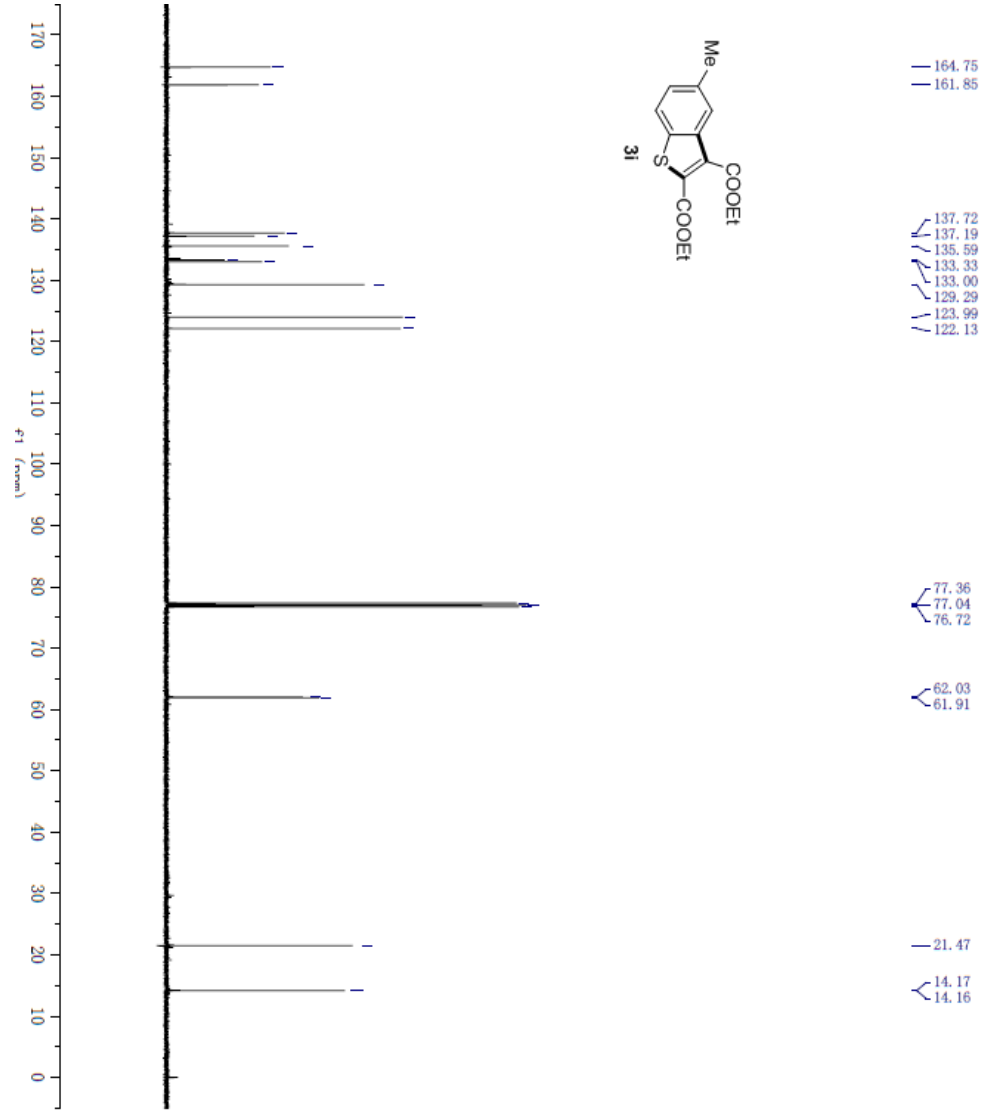
BRUKER

Current Data Parameters
NAME ykl-6
EXPNO 21
PROCNO 1

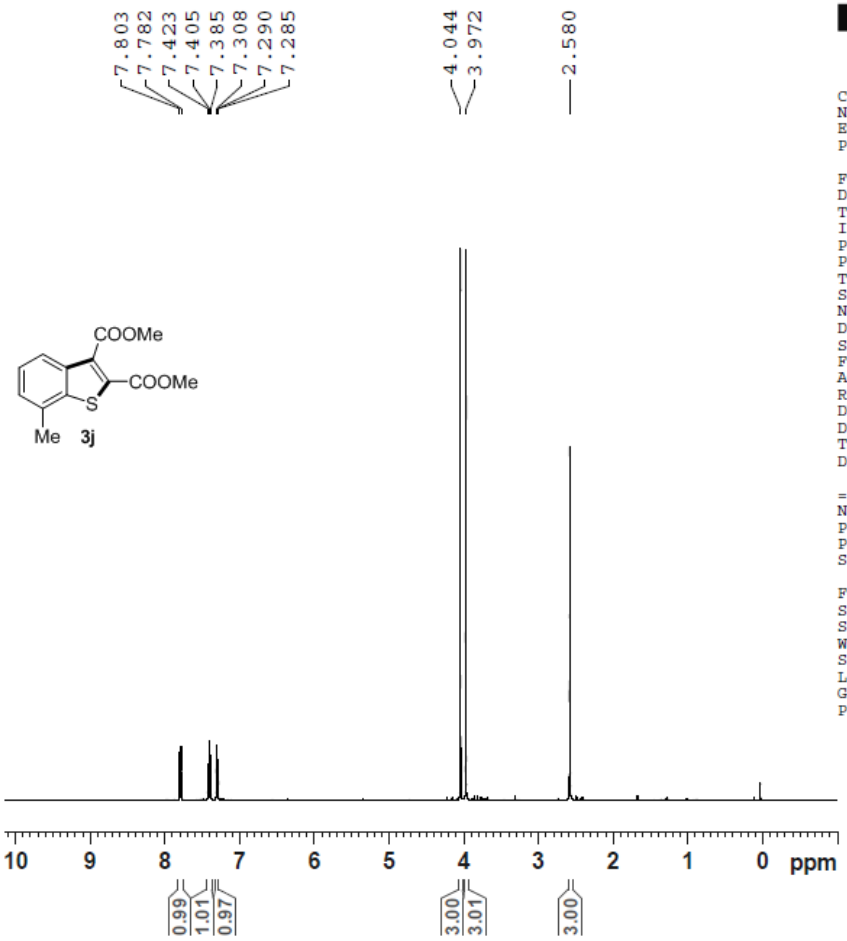
F2 - Acquisition Parameters
Date_ 20140225
Time 2.05
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 128
DW 60.800 usec
DE 6.50 usec
TE 303.0 K
D1 1.00000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 14.94 usec
PLW1 14.10299969 W
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 65536
SF 400.1300000 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

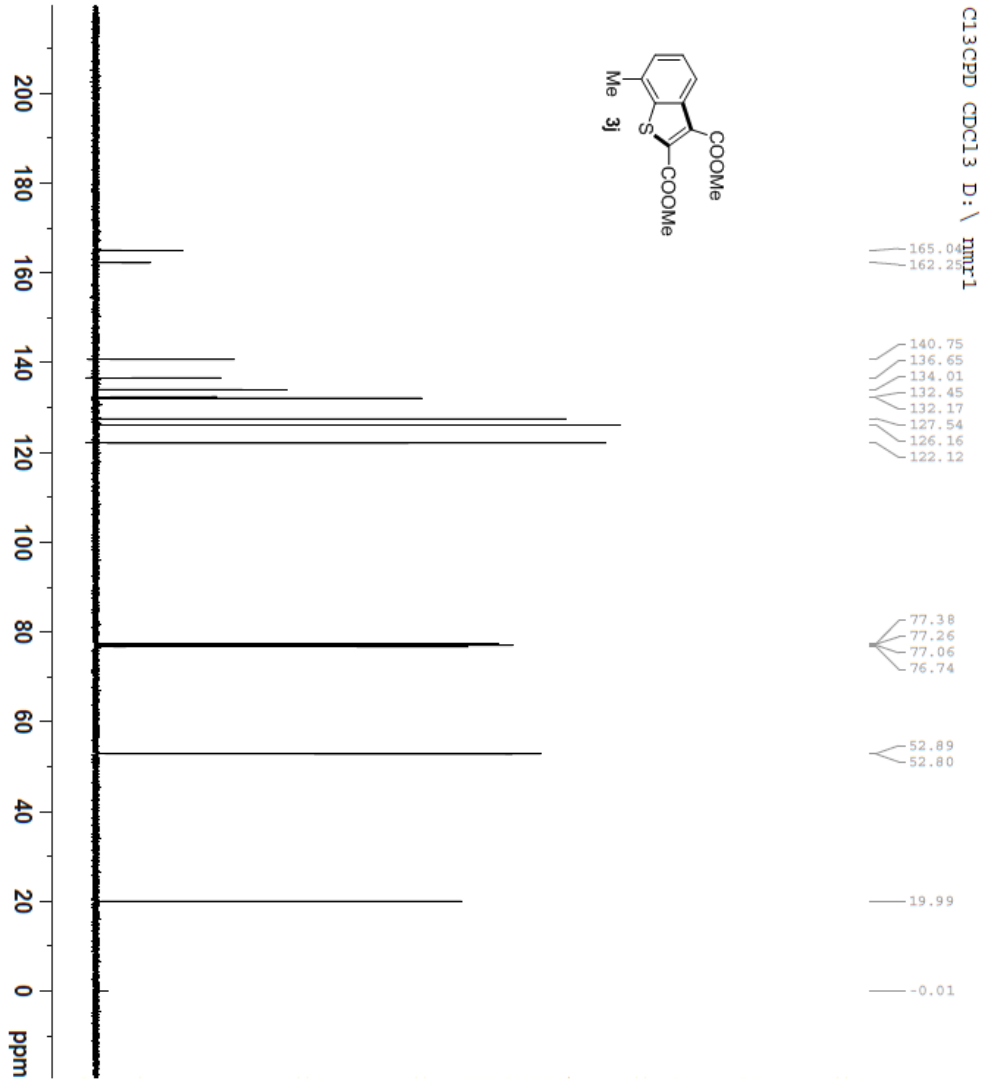

Me
S
COOEt
COOEt
3i
164.75
161.85
137.72
137.19
135.59
133.33
133.00
129.29
123.99
122.13
77.36
77.04
76.72
62.03
61.91
21.47
14.17
14.16
f1 (ppm)

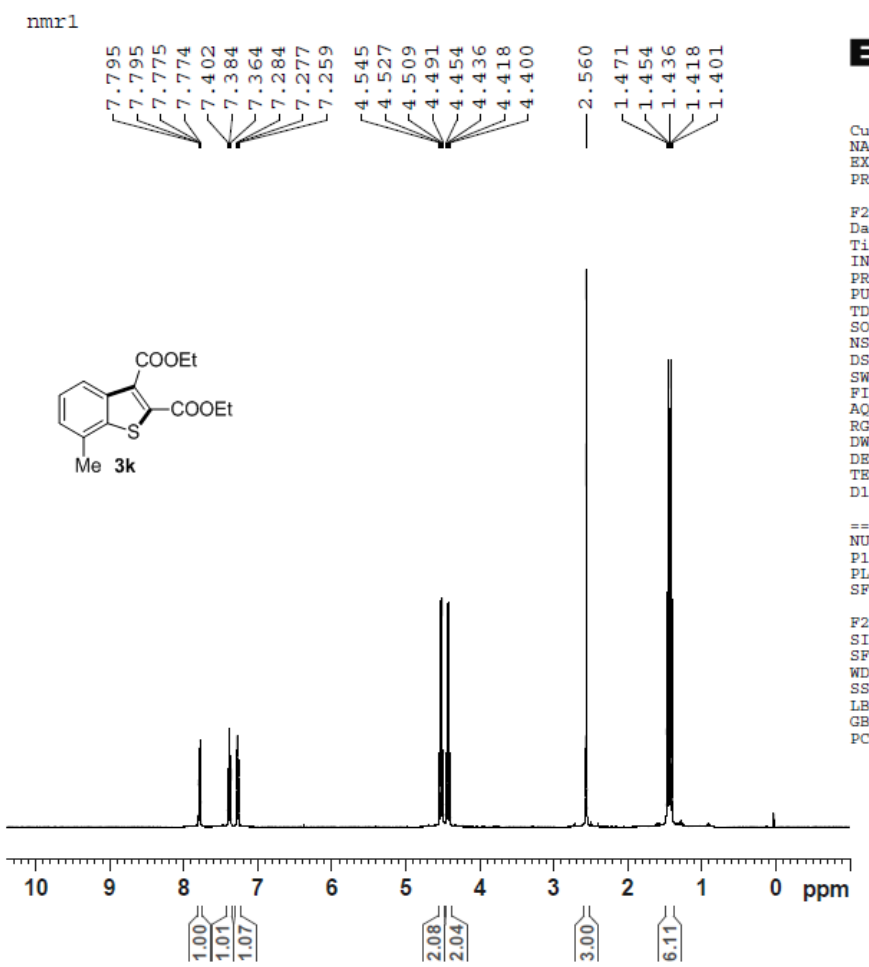
nmr1



BRUKER

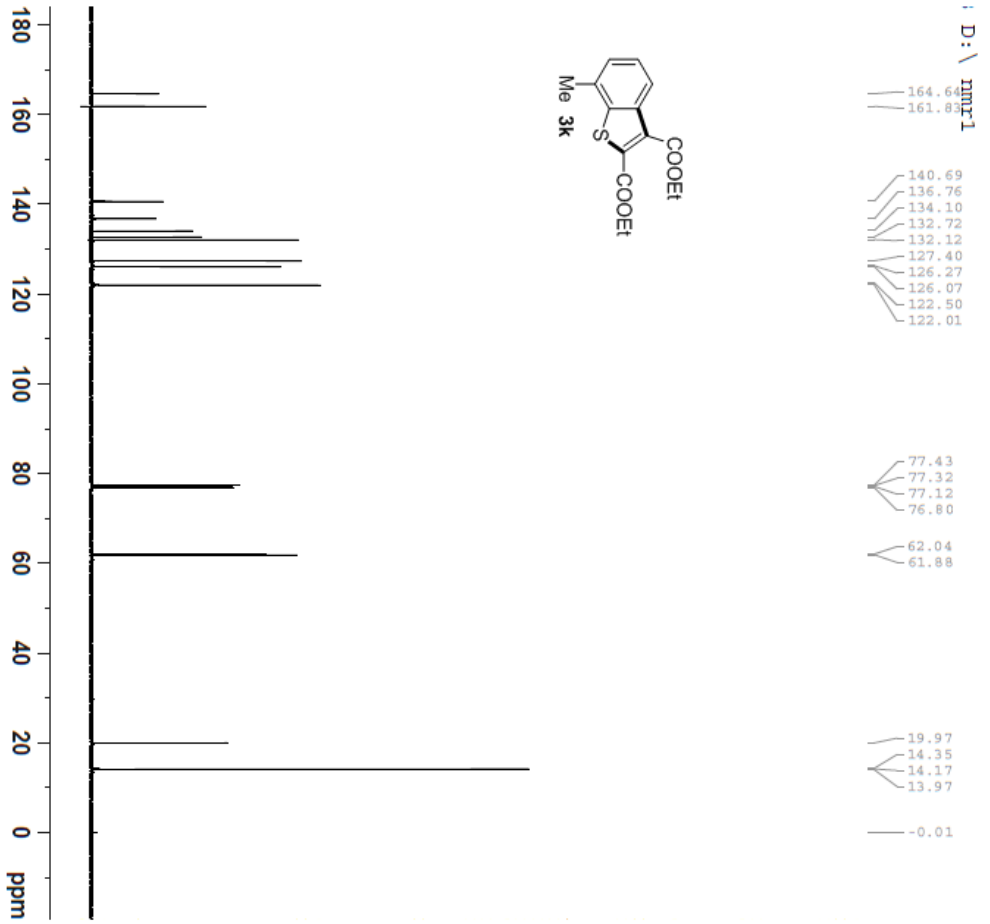
```
Current Data Parameters
NAME                  ykl-9
EXPNO                    20
PROCNO                    1

F2 - Acquisition Parameters
Date_              20140311
Time                   5.58
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT               CDC13
NS                       16
DS                        2
SWH                8223.685 Hz
FIDRES             0.125483 Hz
AQ                 3.9846387 sec
RG                       114
DW                  60.800 usec
DE                   6.50 usec
TE                   303.0 K
D1                1.00000000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                     14.94 usec
PLW1            14.10299969 W
SFO1           400.1324710 MHz

F2 - Processing parameters
SI                        65536
SF             400.1300000 MHz
WDW                        no
SSB       0
LB        0 Hz
GB        0
PC                      1.00
```

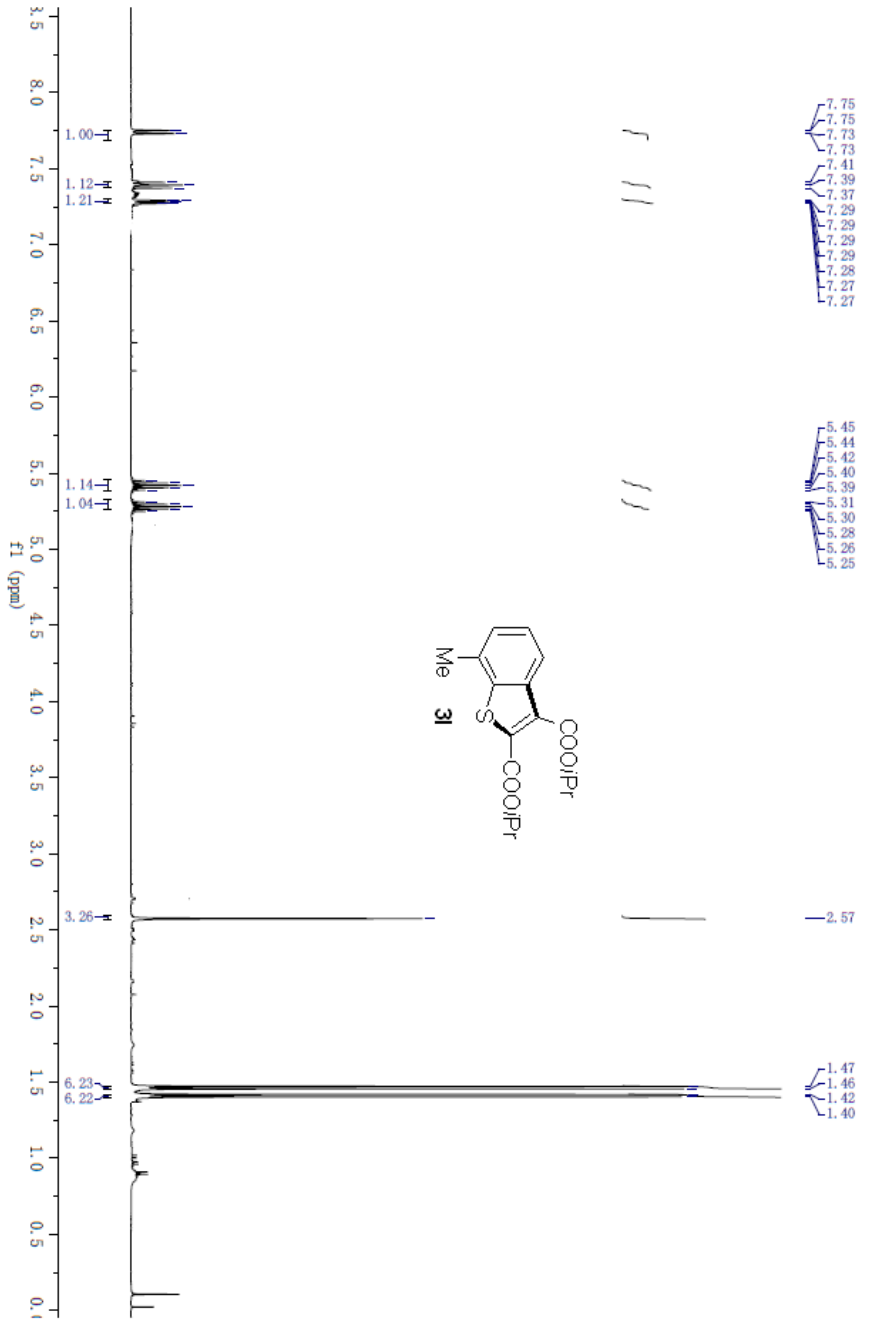

C13CPD CDCl3 D:\ nmr1
165.04
162.25
140.75
136.65
134.01
132.45
132.17
127.54
126.16
122.12
77.38
77.26
77.06
76.74
52.89
52.80
19.99
-0.01
Me
3j
S
COOMe
COOMe
200
180
160
140
120
100
80
60
40
20
0
ppm



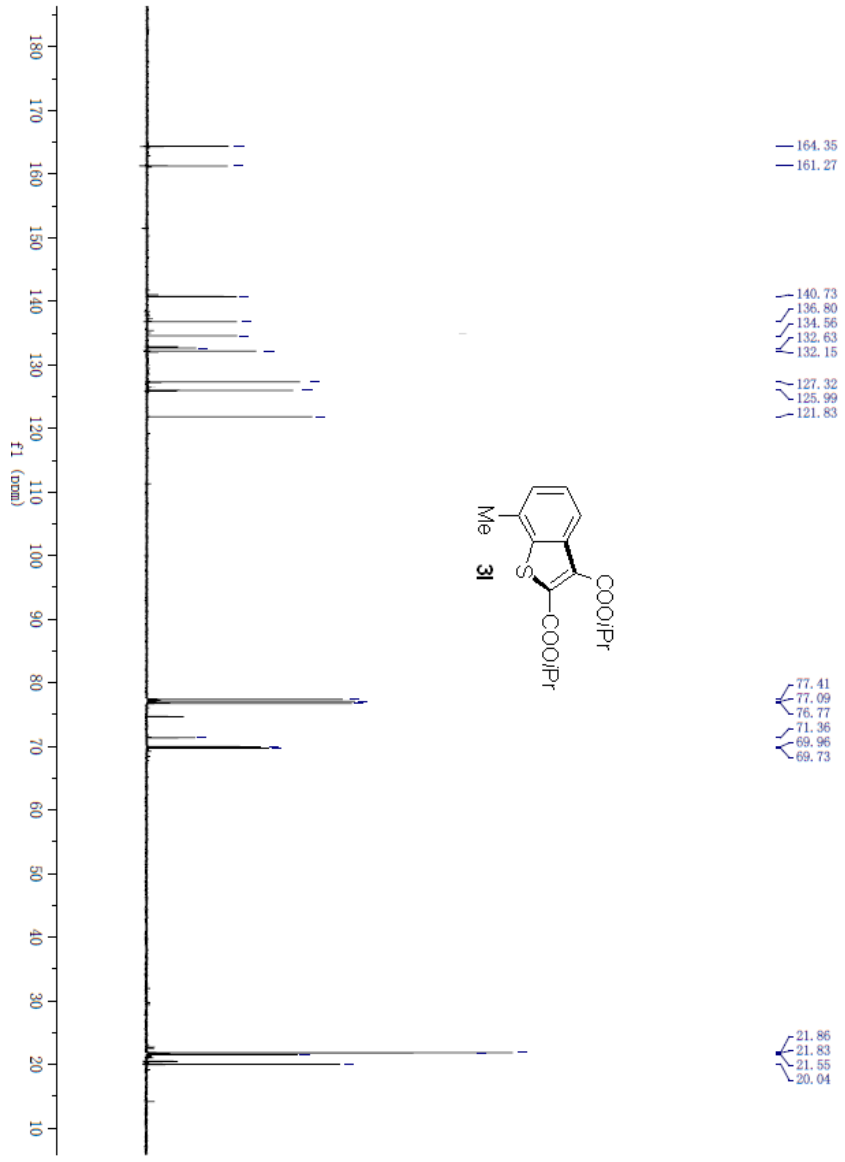
BRUKER

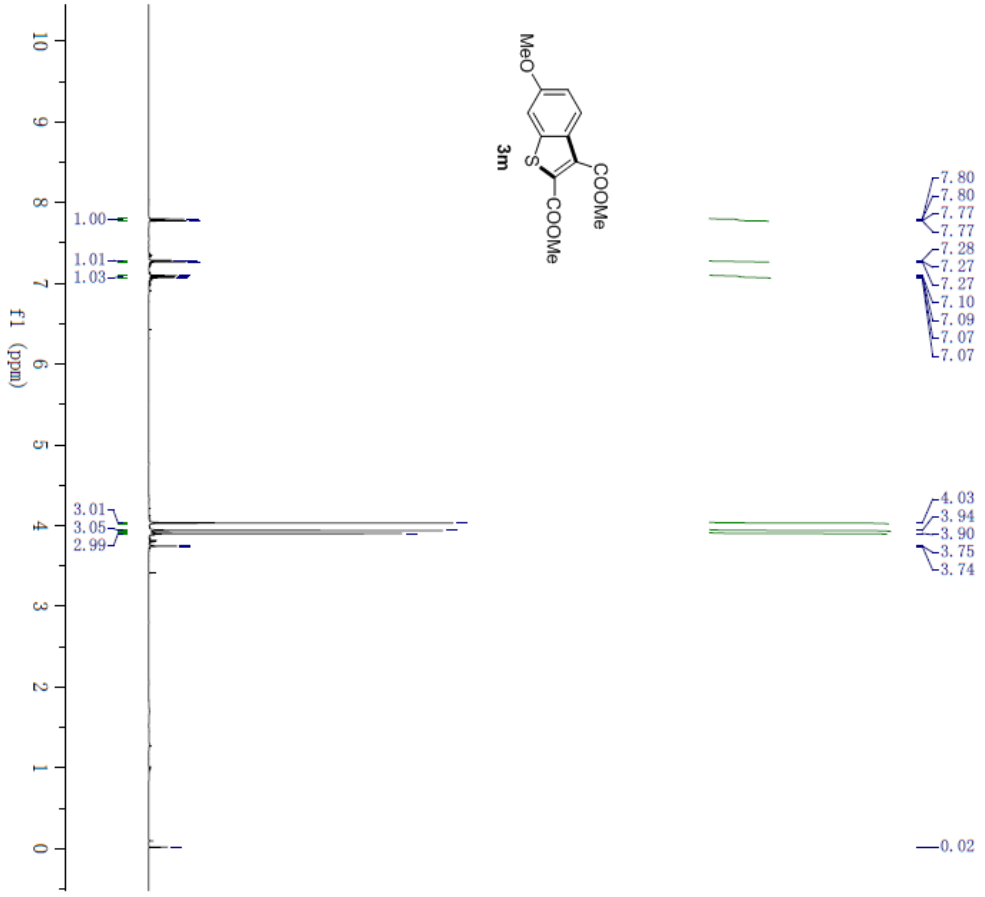
```
Current Data Parameters
NAME                  ykl-10
EXPNO                     22
PROCNO                     1

F2 - Acquisition Parameters
Date_              20140311
Time                   7.50
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8223.685 Hz
FIDRES            0.125483 Hz
AQ                3.9846387 sec
RG                       57
DW                  60.800 usec
DE                   6.50 usec
TE                   303.0 K
D1              1.00000000 sec

======== CHANNEL f1 ========
NUC1                     1H
P1                    14.94 usec
PLW1          14.10299969 W
SFO1          400.1324710 MHz

F2 - Processing parameters
SI                    65536
SF            400.1300000 MHz
WDW                      no
SSB       0
LB        0 Hz
GB        0
PC                    1.00
```

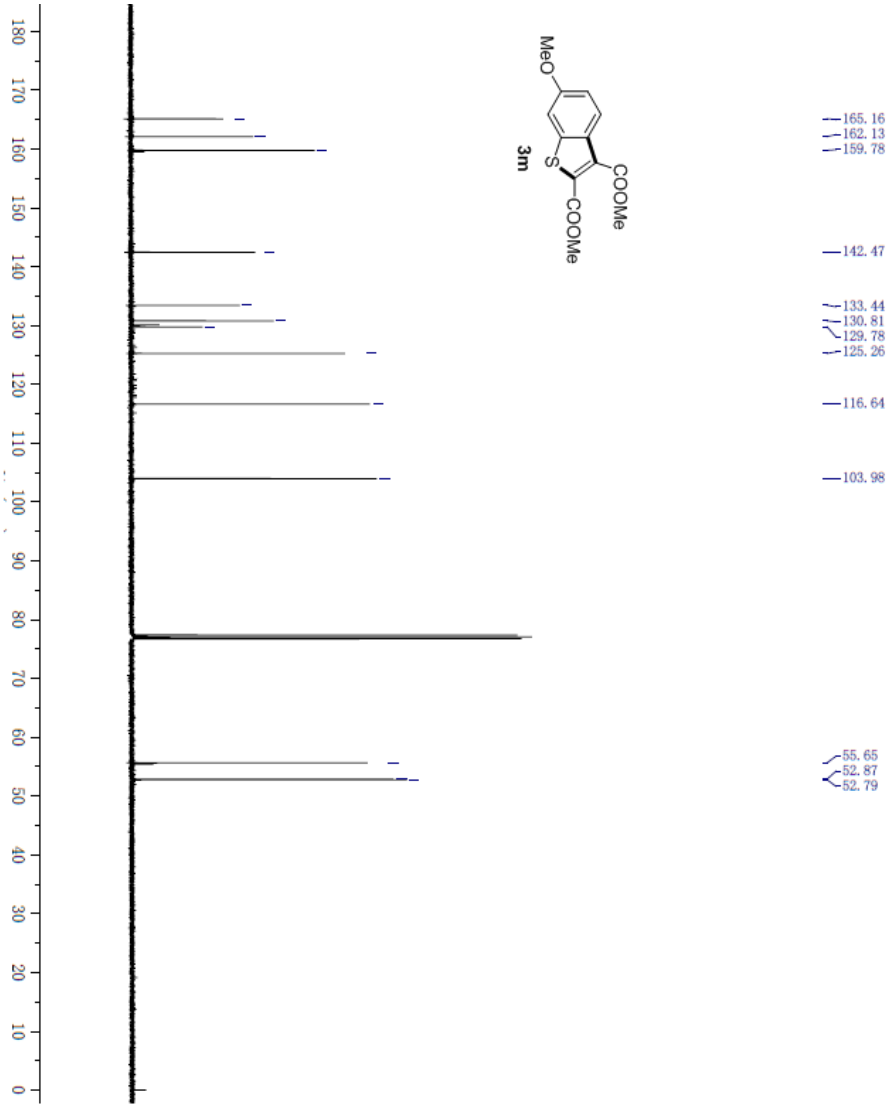

D:\ nmr1
164.64
161.83
140.69
136.76
134.10
132.72
132.12
127.40
126.27
126.07
122.50
122.01
77.43
77.32
77.12
76.80
62.04
61.88
19.97
14.35
14.17
13.97
-0.01
Me
3k
S
COOEt
COOEt
180
160
140
120
100
80
60
40
20
0
ppm

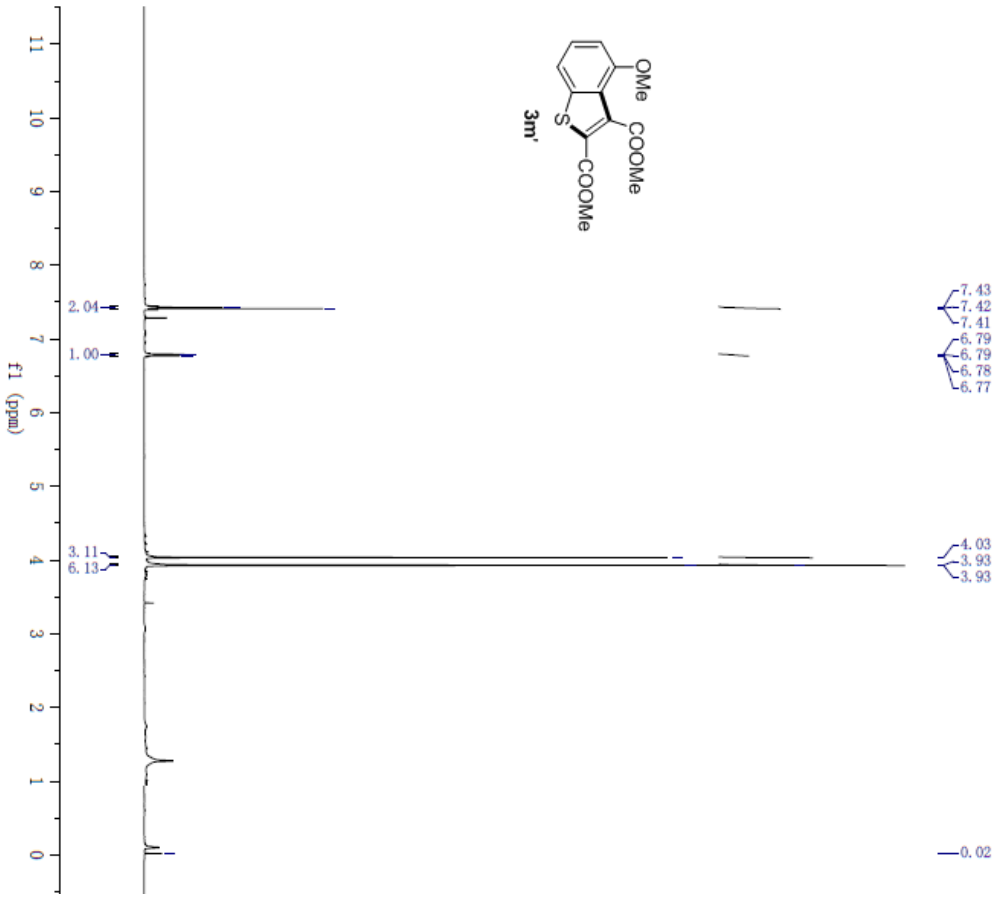

f1 (ppm)
1.00
1.12
1.21
1.14
1.04
3.26
6.23
6.22
7.75
7.75
7.73
7.73
7.41
7.39
7.37
7.29
7.29
7.29
7.29
7.28
7.27
7.27
5.45
5.44
5.42
5.40
5.39
5.31
5.30
5.28
5.26
5.25
2.57
1.47
1.46
1.42
1.40
Me
S
COOiPr
COOiPr

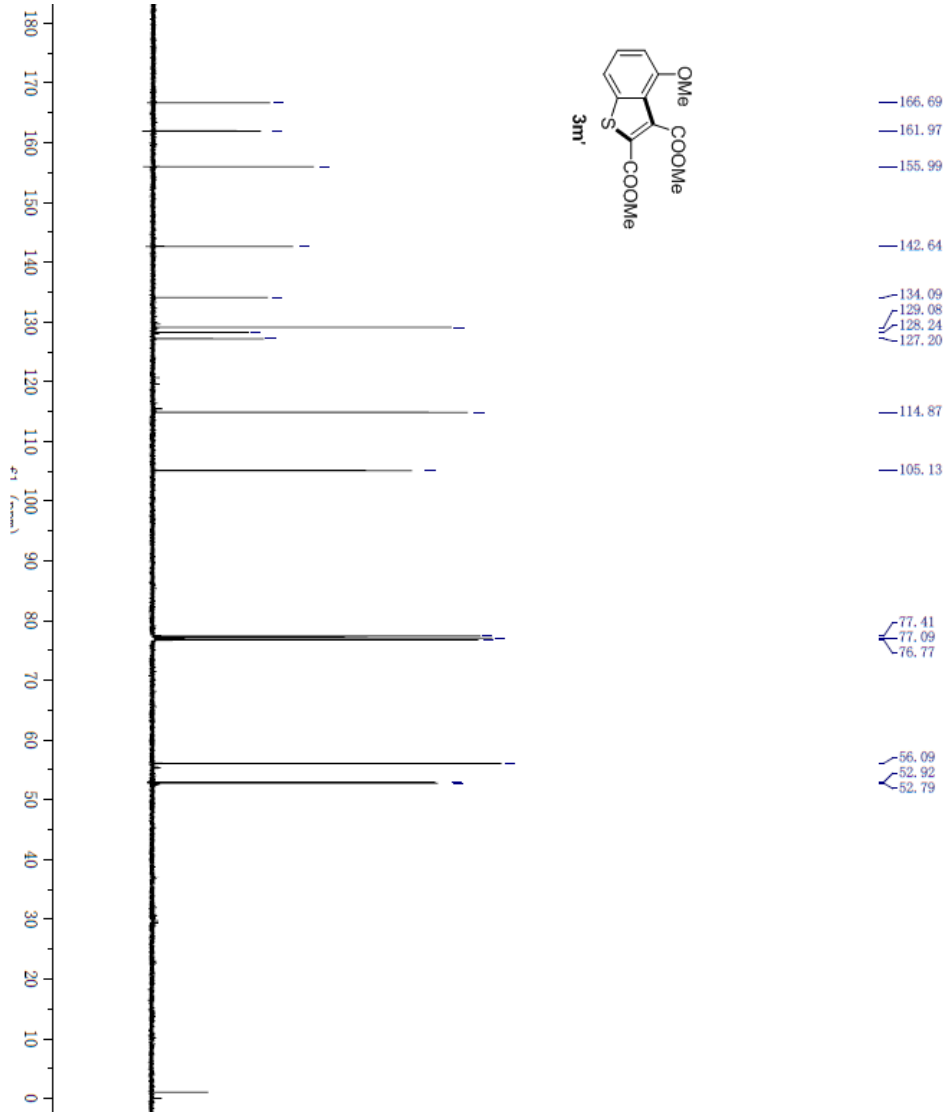
3l

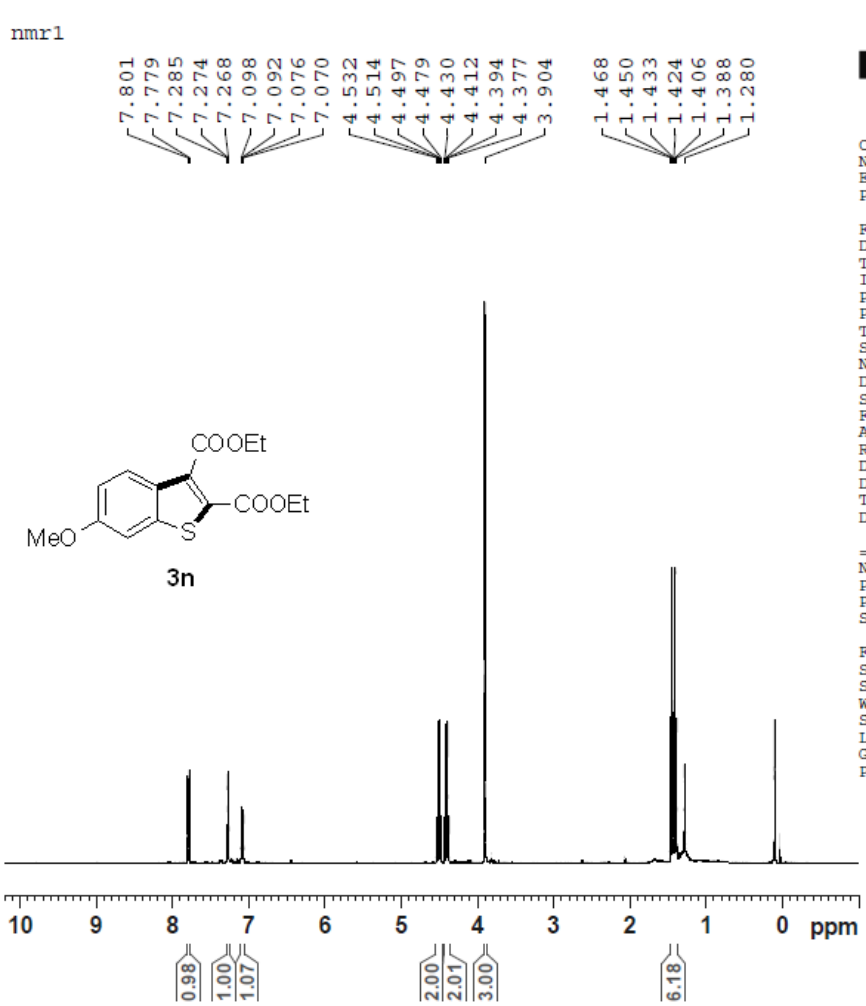

f1 (ppm)
164.35
161.27
140.73
136.80
134.56
132.63
132.15
127.32
125.99
121.83
77.41
77.09
76.77
71.36
69.96
69.73
21.86
21.83
21.55
20.04
Me
S
COOiPr
COOiPr
3l


MeO
S
COOMe
COOMe
3m
f1 (ppm)
1.00
1.01
1.03
3.01
3.05
2.99
7.80
7.80
7.77
7.77
7.28
7.27
7.27
7.10
7.09
7.07
7.07
4.03
3.94
3.90
3.75
3.74
0.02


MeO
S
COOMe
COOMe
3m
165.16
162.13
159.78
142.47
133.44
130.81
129.78
125.26
116.64
103.98
55.65
52.87
52.79

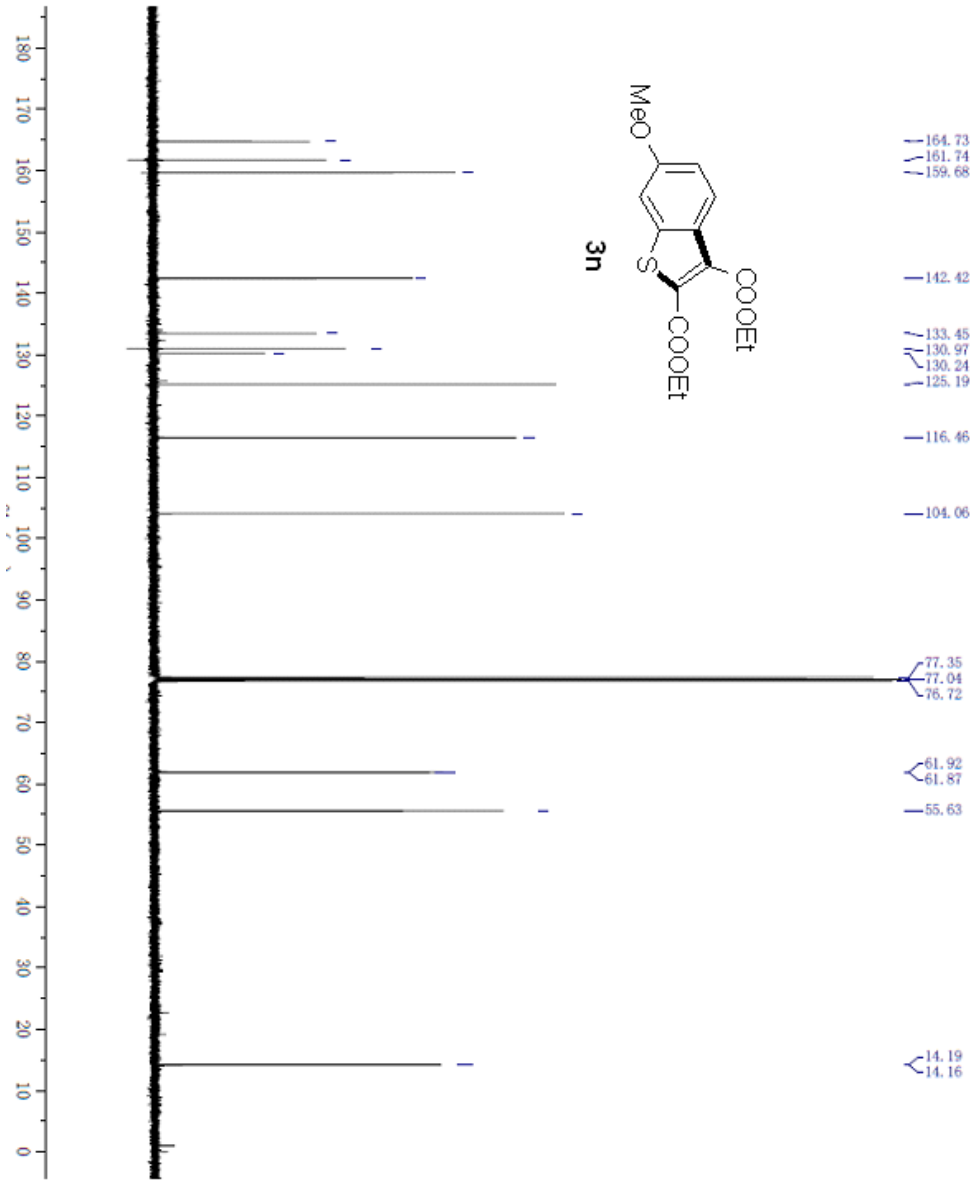

OMe
COOMe
COOMe
S
3m'
f1 (ppm)
2.04
1.00
3.11
6.13
7.43
7.42
7.41
6.79
6.79
6.78
6.77
4.03
3.93
3.93
0.02

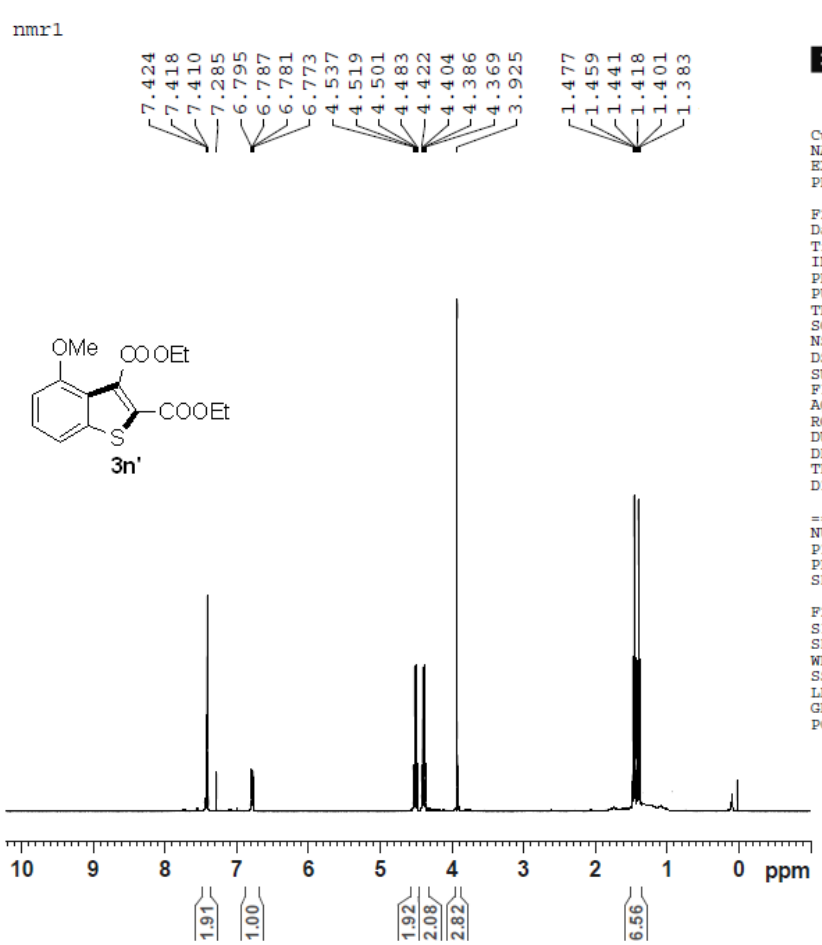

OMe
COOMe
COOMe
S
3m'
166.69
161.97
155.99
142.64
134.09
129.08
128.24
127.20
114.87
105.13
77.41
77.09
76.77
56.09
52.92
52.79



BRUKER

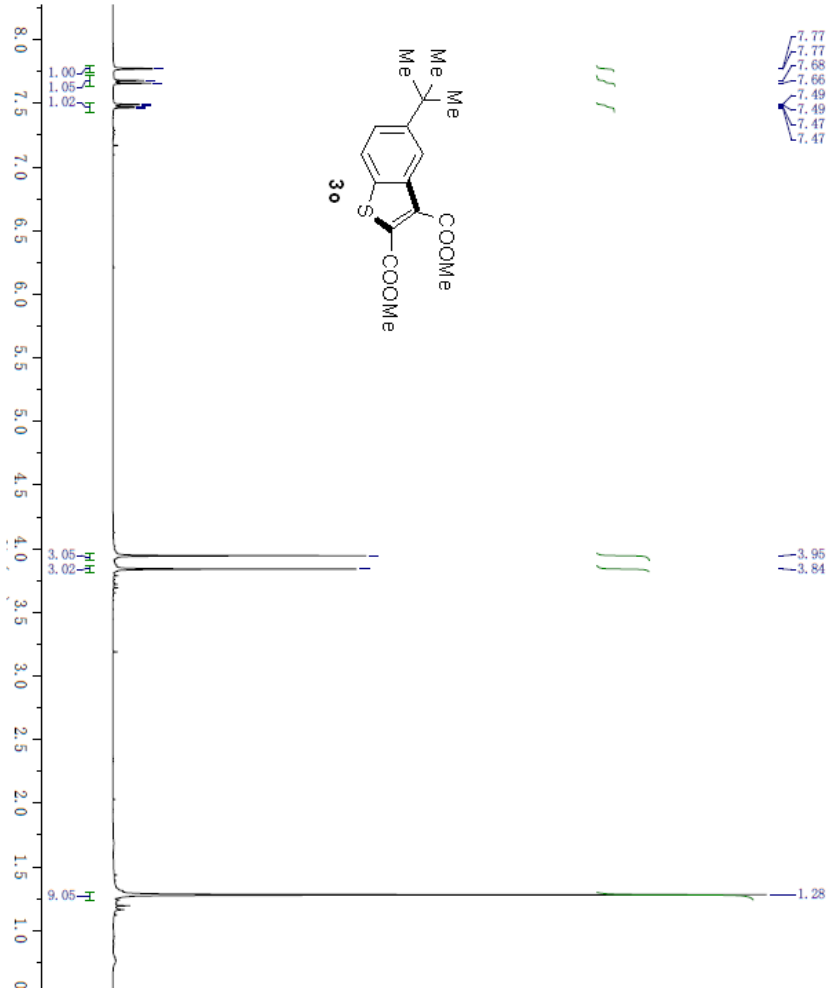
```
Current Data Parameters
NAME                  ykl-15a
EXPNO                      10
PROCNO                      1

F2 - Acquisition Parameters
Date_               20140311
Time                    4.53
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG                 zg30
TD                     65536
SOLVENT                CDC13
NS                        16
DS                         2
SWH                 8223.685 Hz
FIDRES              0.125483 Hz
AQ                3.9846387 sec
RG                       128
DW                  60.800 usec
DE                   6.50 usec
TE                   303.0 K
D1                1.00000000 sec

======== CHANNEL f1 ========
NUC1                       1H
P1                      14.94 usec
PLW1             14.10299969 W
SFO1             400.1324710 MHz

F2 - Processing parameters
SI                         65536
SF               400.1300000 MHz
WDW                           no
SSB          0
LB           0 Hz
GB           0
PC                        1.00
```

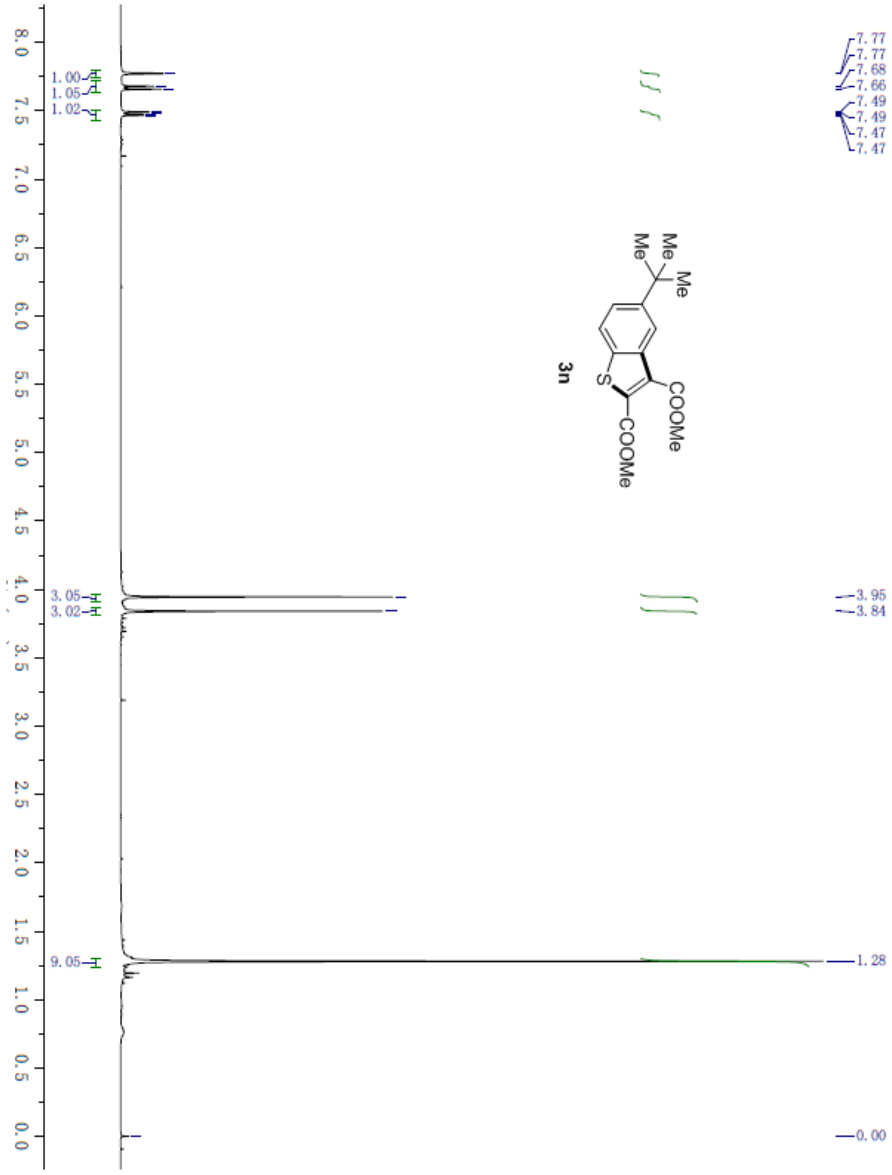

MeO
S
COOEt
COOEt
3n
164.73
161.74
159.68
142.42
133.45
130.97
130.24
125.19
116.46
104.06
77.35
77.04
76.72
61.92
61.87
55.63
14.19
14.16

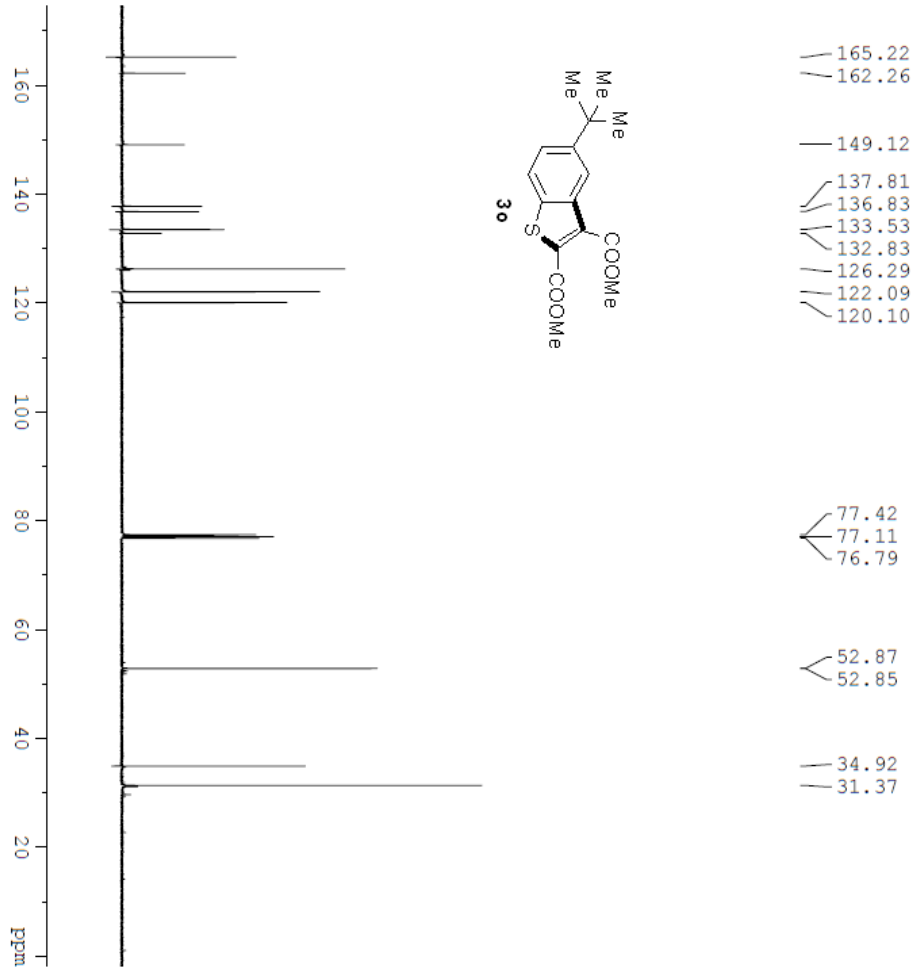


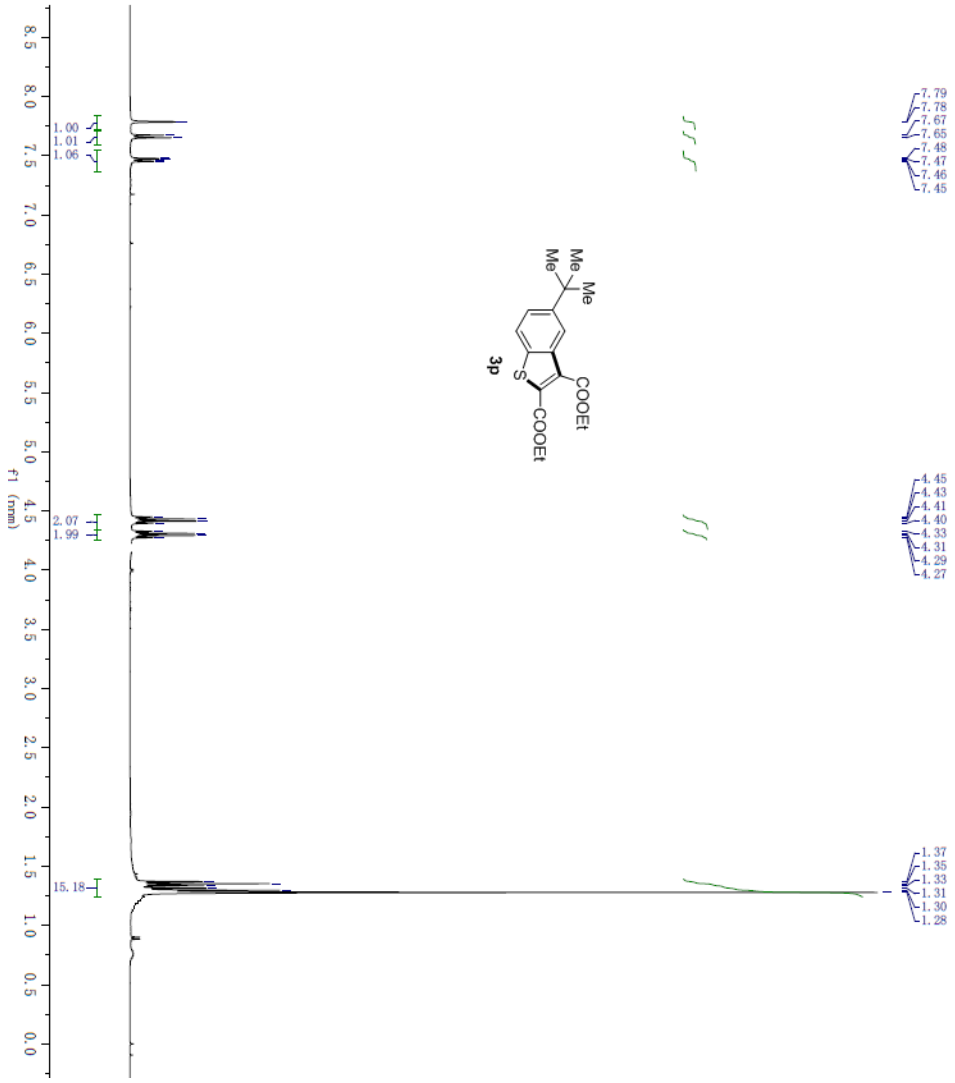
BRUKER

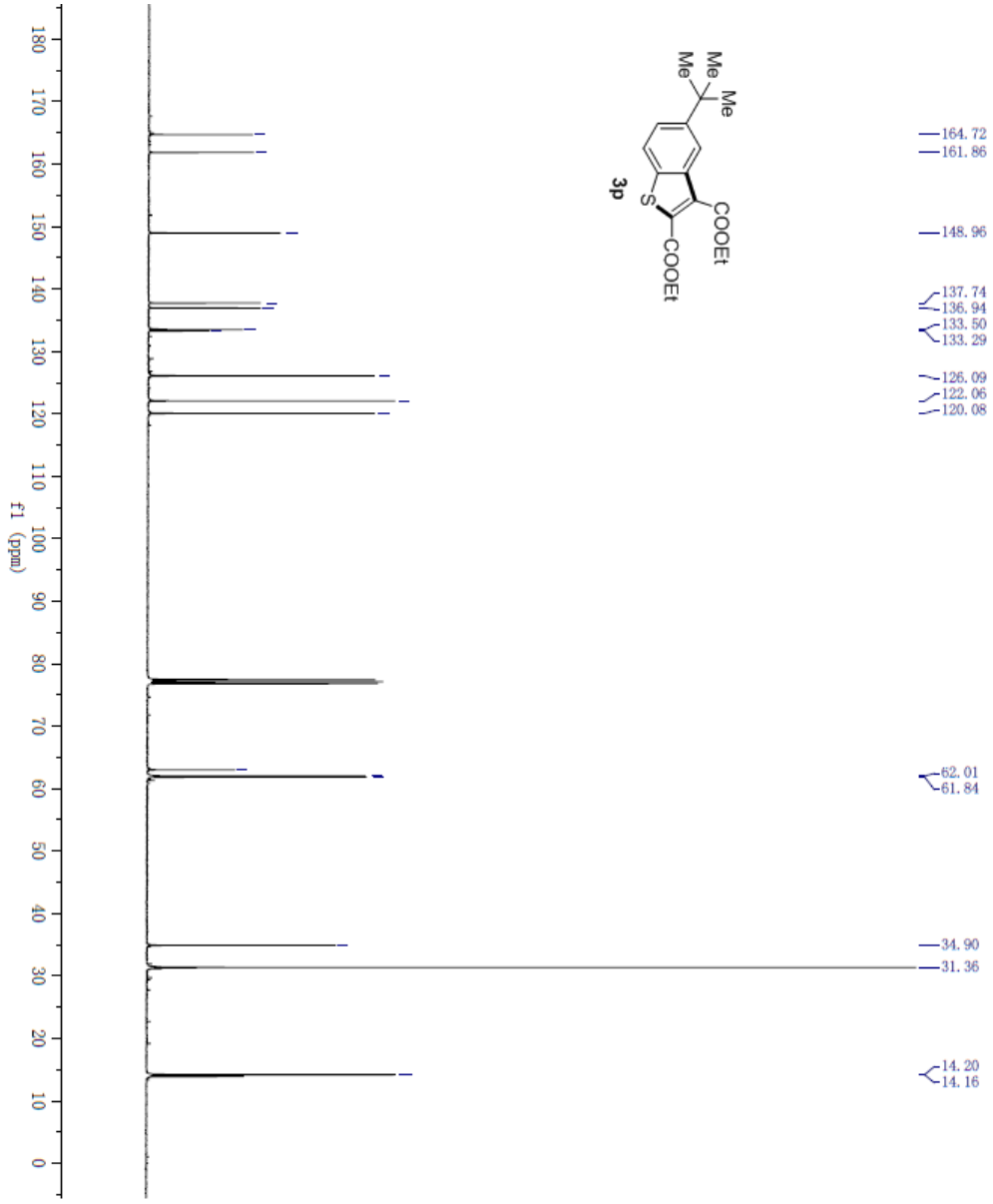
```
Current Data Parameters
NAME                  ykl-15b
EXPNO                      10
PROCNO                      1

F2 - Acquisition Parameters
Date_               20140312
Time                    8.59
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                 8223.685 Hz
FIDRES              0.125483 Hz
AQ                  3.9846387 sec
RG                       80.6
DW                    60.800 usec
DE                     6.50 usec
TE                     641.0 K
D1                  1.00000000 sec

======== CHANNEL f1 ========
NUC1                       1H
P1                      14.94 usec
PLW1             14.10299969 W
SFO1            400.1324710 MHz

F2 - Processing parameters
SI                     65536
SF              400.1300000 MHz
WDW                       no
SSB          0
LB           0 Hz
GB           0
PC                      1.00
```

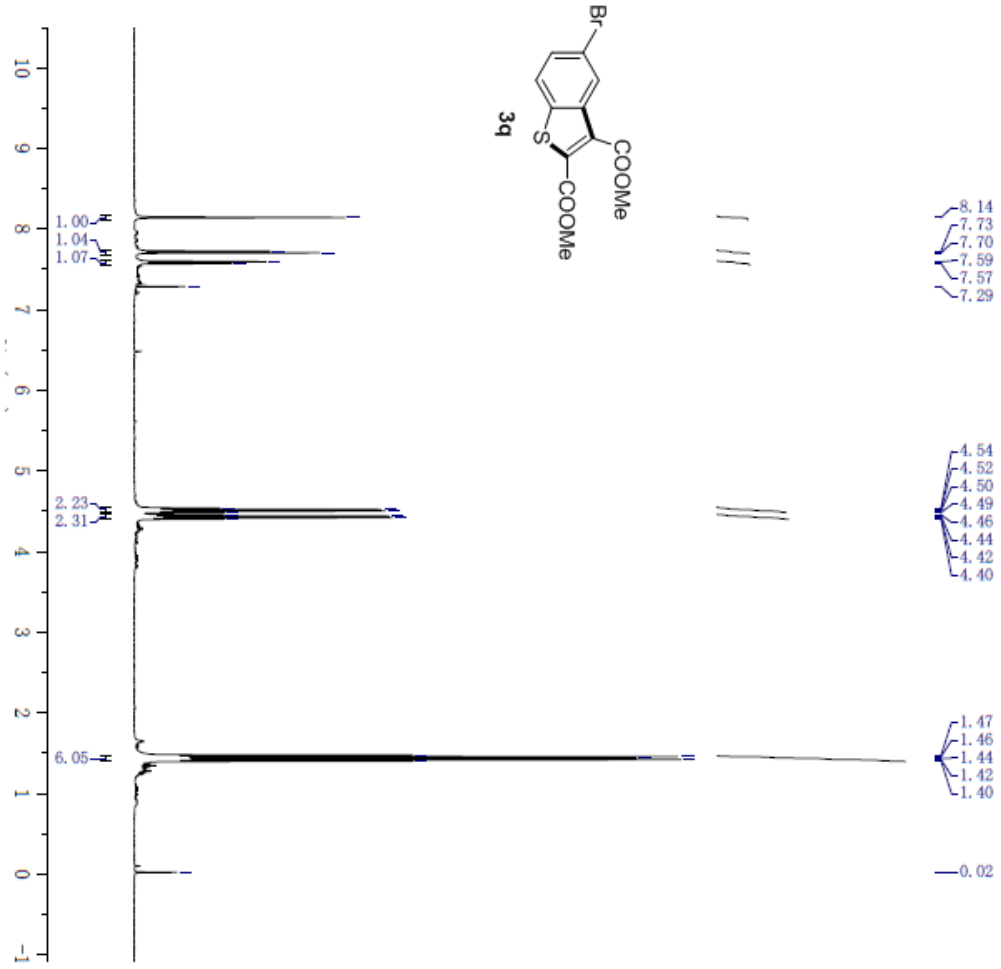

3o
Me
Me
Me
COOMe
COOMe
S
7.77
7.77
7.68
7.66
7.49
7.49
7.47
7.47
3.95
3.84
1.28
1.00
1.05
1.02
3.05
3.02
9.05

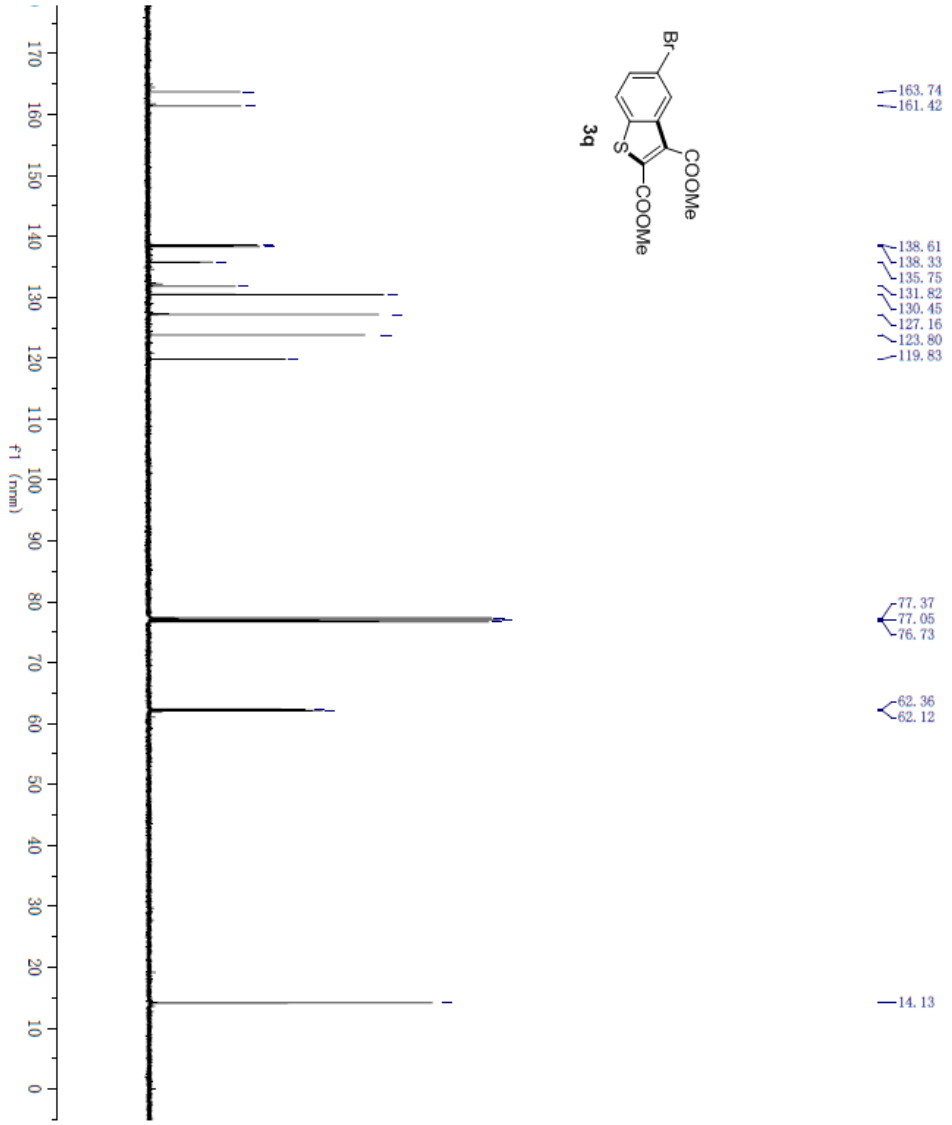

3n
Me
Me
Me
COOMe
COOMe
S
7.77
7.77
7.68
7.66
7.49
7.49
7.47
7.47
3.95
3.84
1.28
0.00
1.00
1.05
1.02
3.05
3.02
9.05

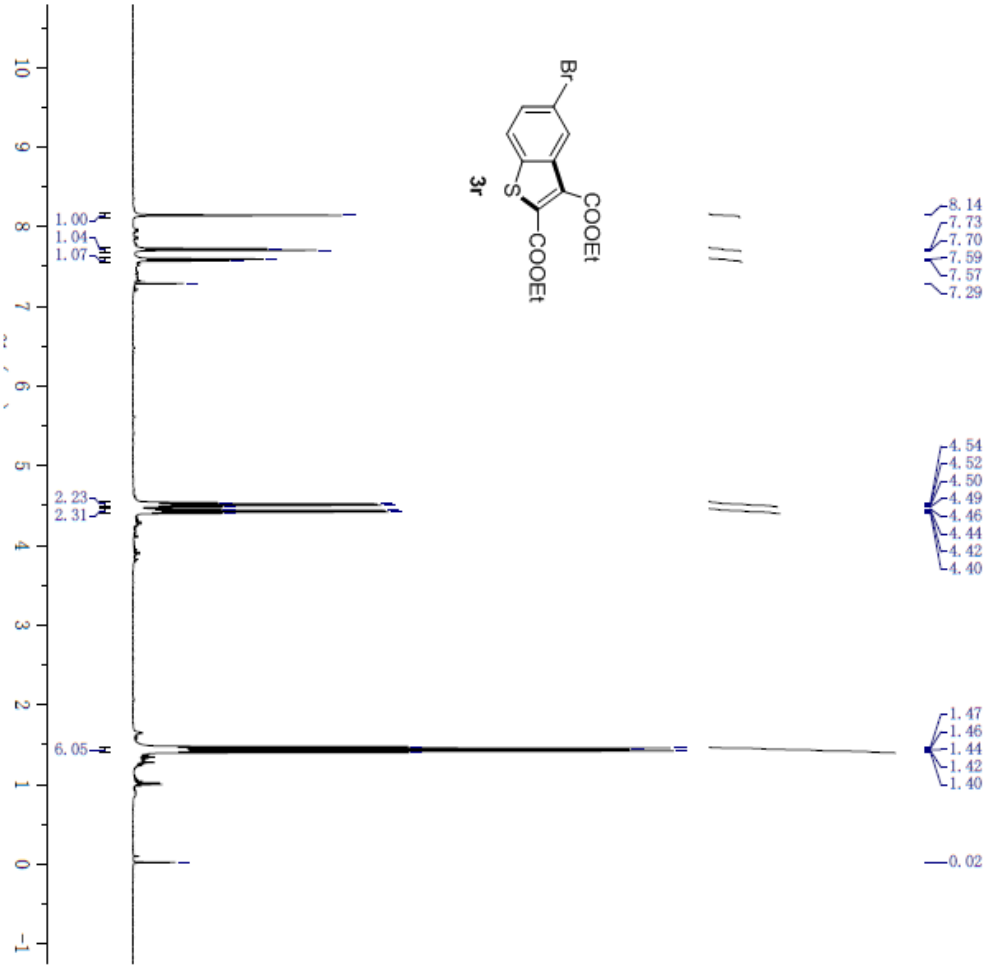

165.22
162.26
149.12
137.81
136.83
133.53
132.83
126.29
122.09
120.10
77.42
77.11
76.79
52.87
52.85
34.92
31.37
Me
Me
Me
S
COOMe
COOMe
3o
160
140
120
100
80
60
40
20
ppm


Me
Me
Me
S
COOEt
COOEt
3p
f1 (ppm)
1.00
1.01
1.06
2.07
1.99
15.18
7.79
7.78
7.67
7.65
7.48
7.47
7.46
7.45
4.45
4.43
4.41
4.40
4.33
4.31
4.29
4.27
1.37
1.35
1.33
1.31
1.30
1.28


Me
Me
Me
COOEt
COOEt
S
3p
164.72
161.86
148.96
137.74
136.94
133.50
133.29
126.09
122.06
120.08
62.01
61.84
34.90
31.36
14.20
14.16
f1 (ppm)


Br
S
COOMe
COOMe
3q
1.00
1.04
1.07
2.23
2.31
6.05
8.14
7.73
7.70
7.59
7.57
7.29
4.54
4.52
4.50
4.49
4.46
4.44
4.42
4.40
1.47
1.46
1.44
1.42
1.40
0.02


Br
COOMe
COOMe
S
3q
163.74
161.42
138.61
138.33
135.75
131.82
130.45
127.16
123.80
119.83
77.37
77.05
76.73
62.36
62.12
14.13
f1 (ppm)


Br
S
COOEt
COOEt
3r
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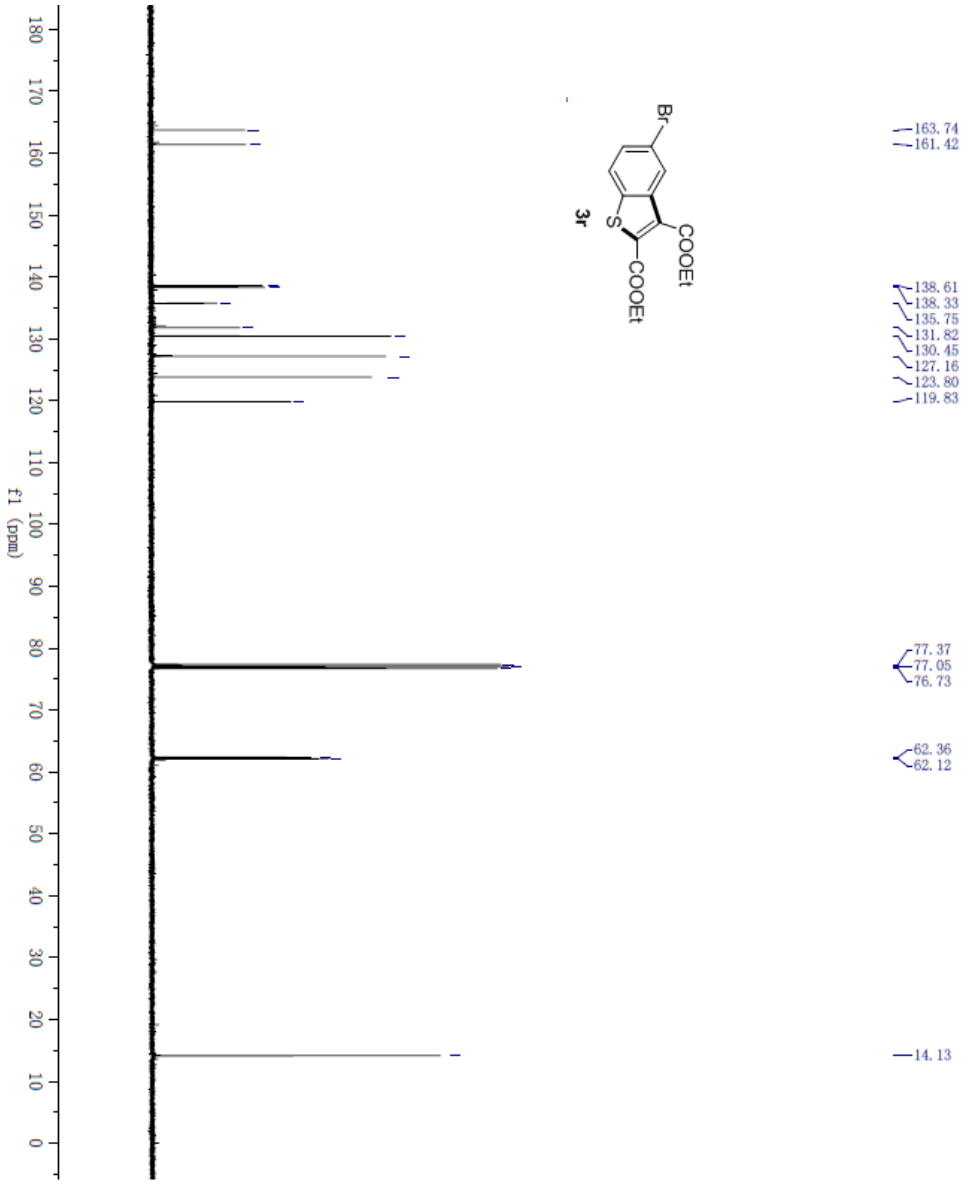
1.04
1.07
2.23
2.31
6.05
8.14
7.73
7.70
7.59
7.57
7.29
4.54
4.52
4.50
4.49
4.46
4.44
4.42
4.40
1.47
1.46
1.44
1.42
1.40
0.02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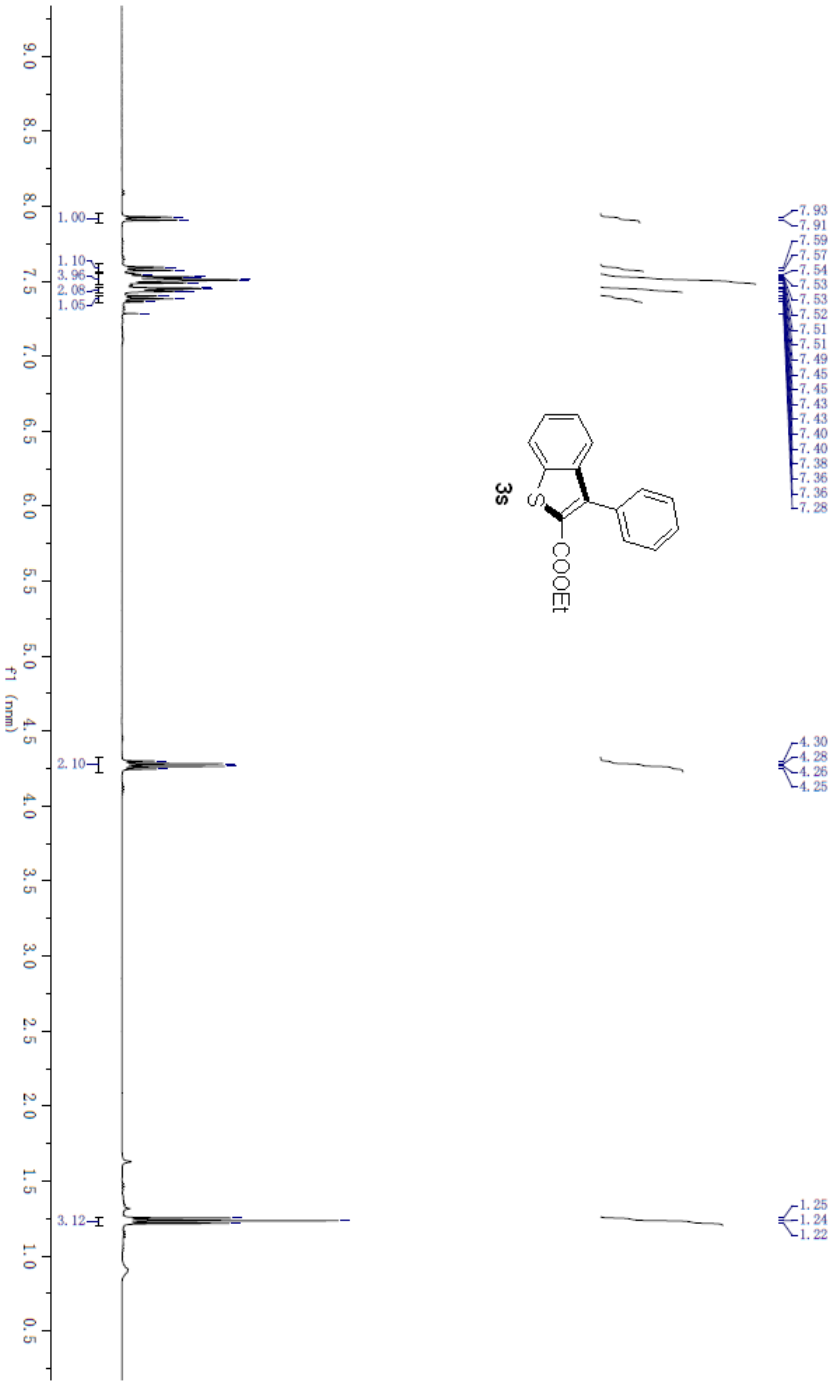
COOEt
COOEt
3r
163.74
161.42
138.61
138.33
135.75
131.82
130.45
127.16
123.80
119.83
77.37
77.05
76.73
62.36
62.12
14.13
f1 (ppm)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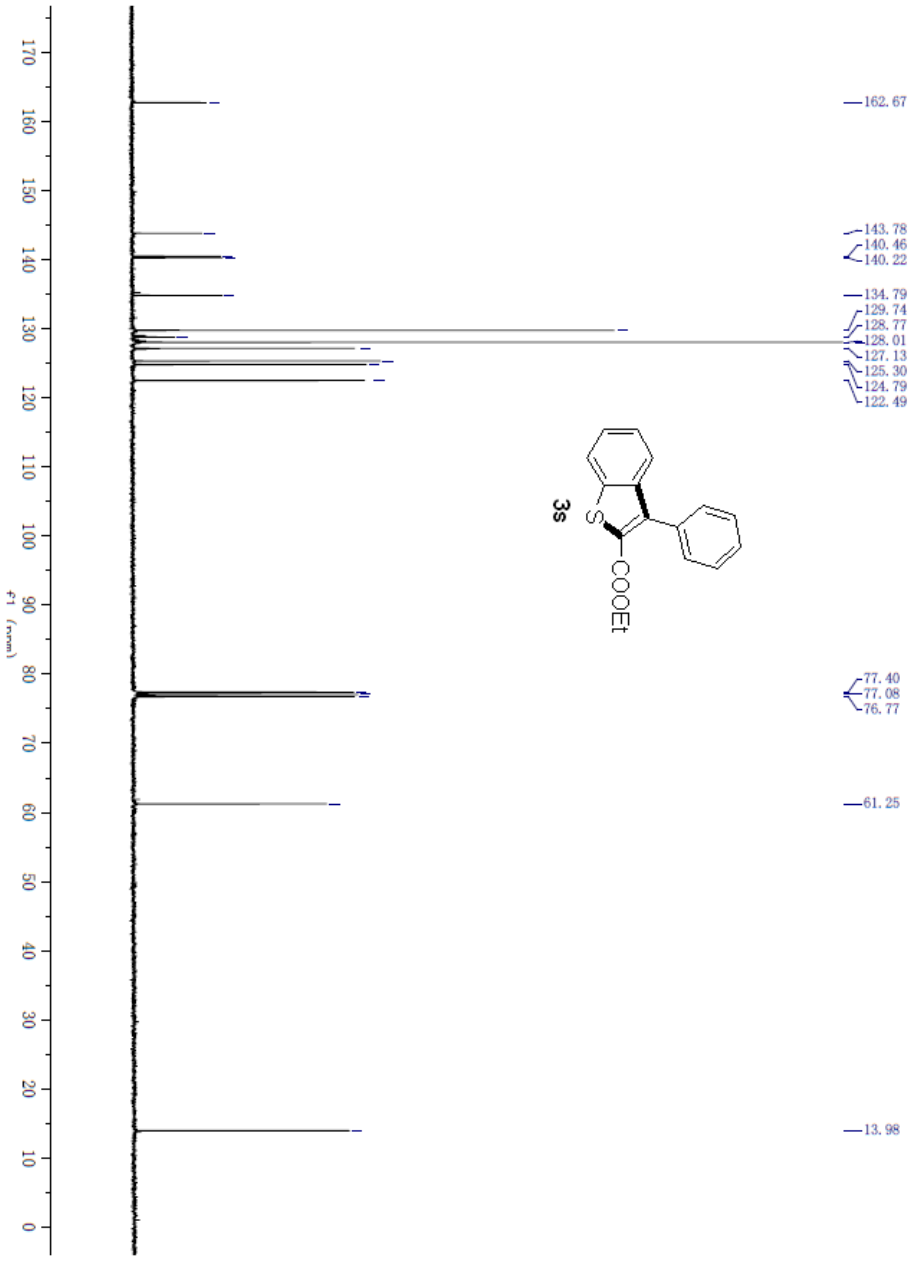
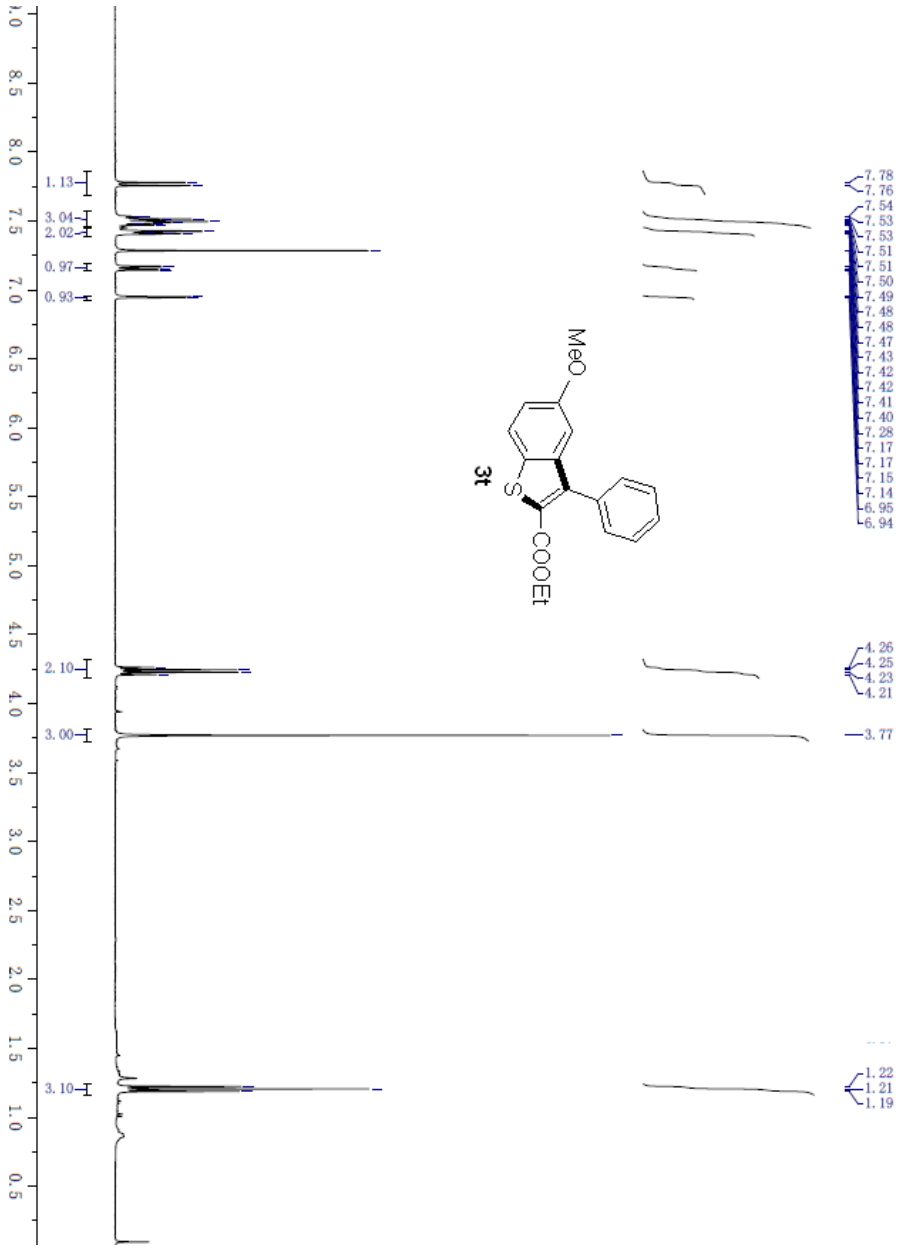
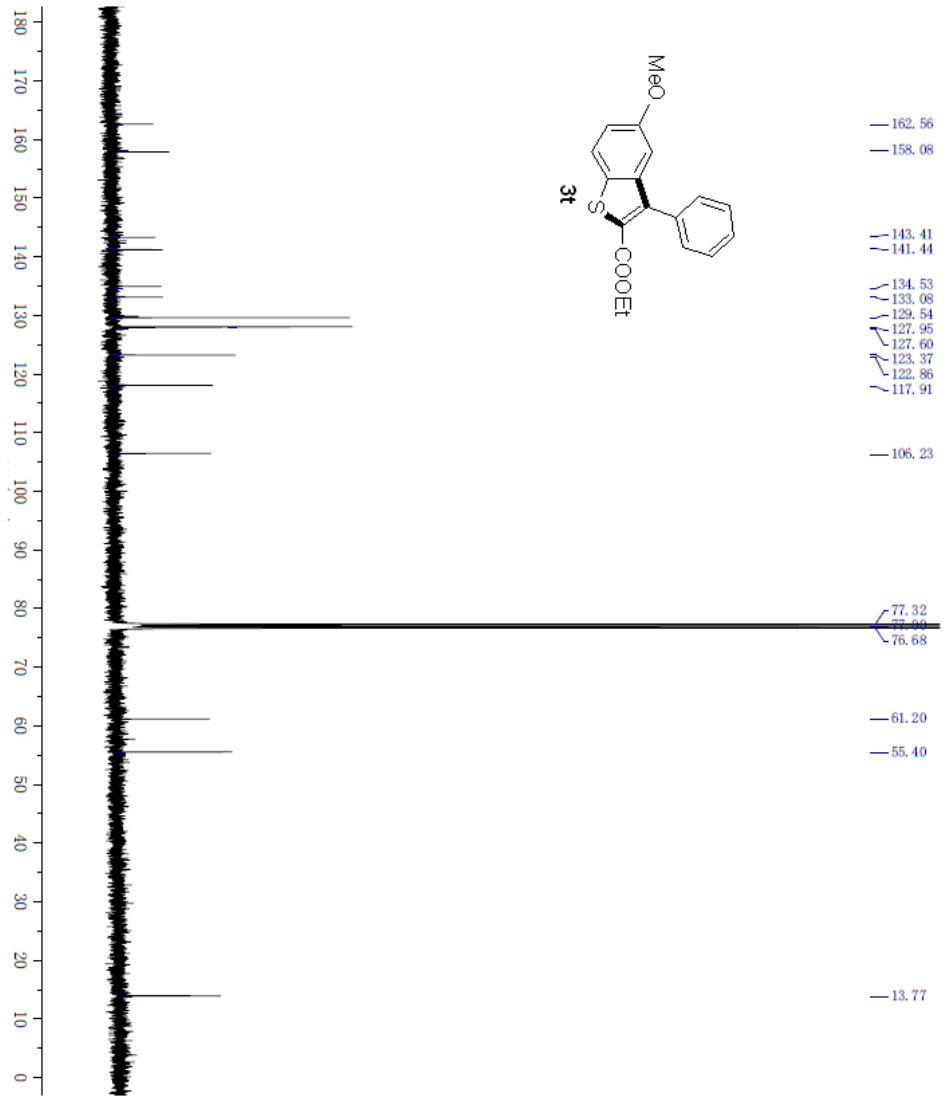
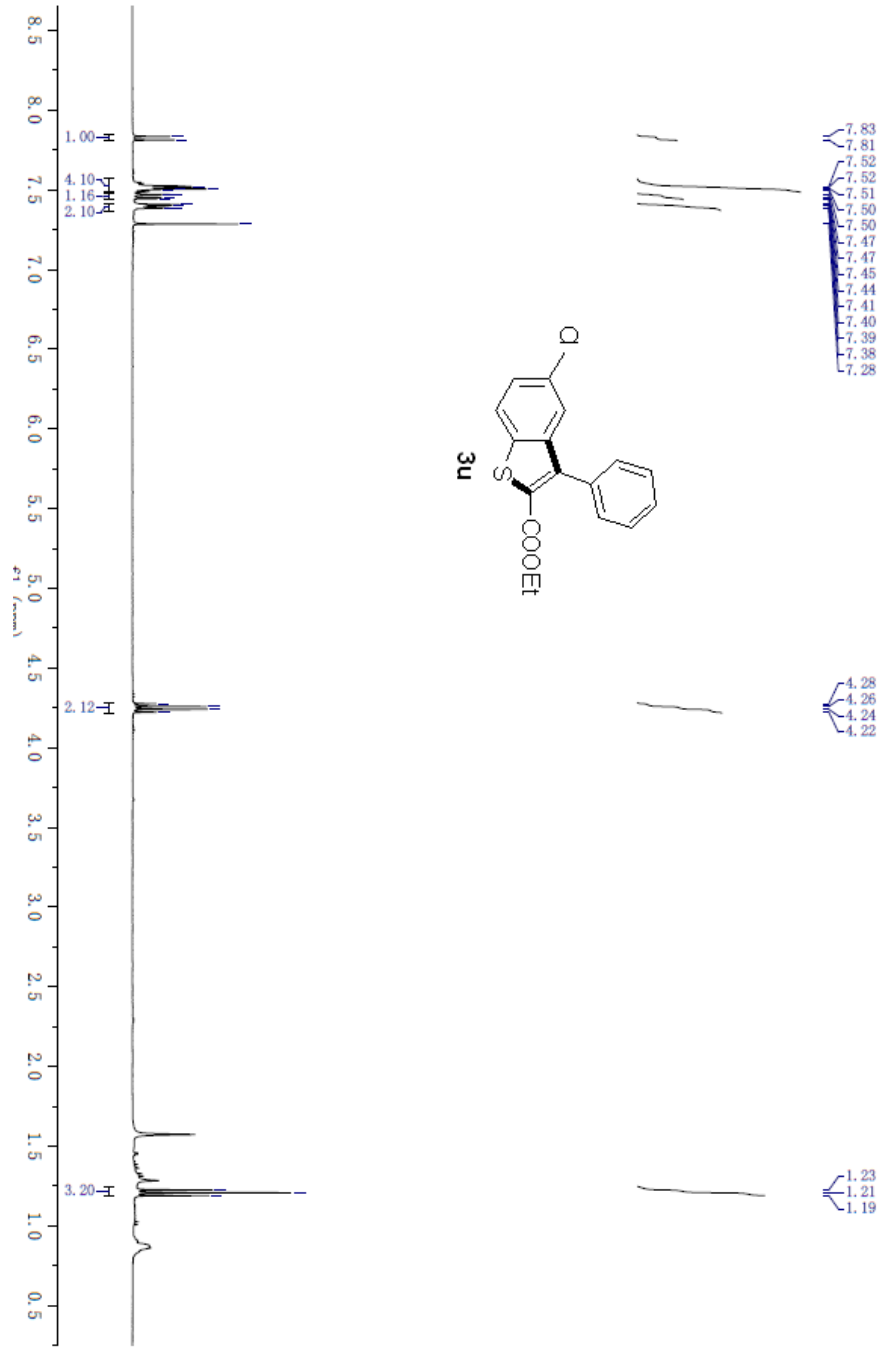
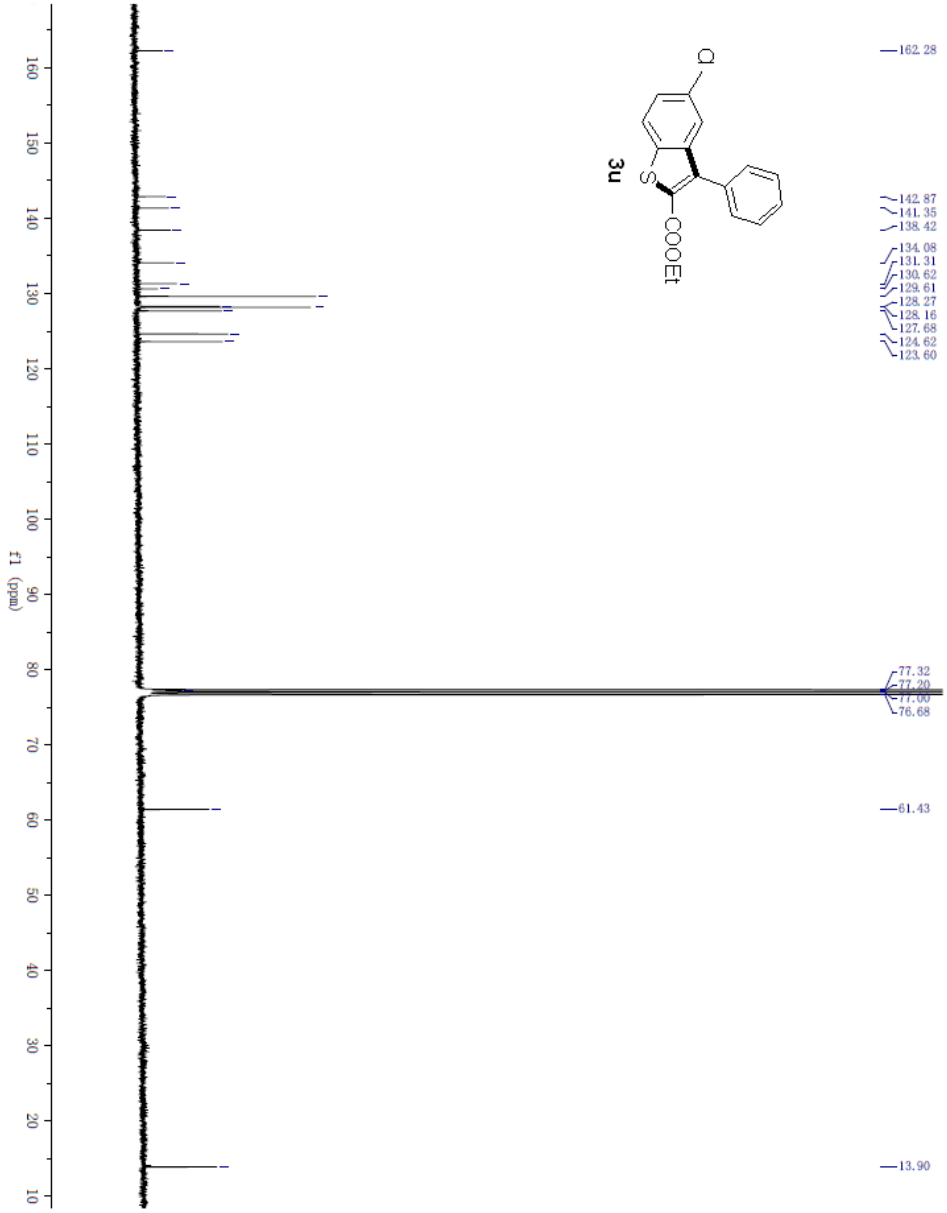
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)
1.00
1.10
3.96
2.08
1.05
2.10
3.12
7.93
7.91
7.59
7.57
7.54
7.53
7.53
7.52
7.51
7.51
7.49
7.45
7.45
7.43
7.43
7.40
7.40
7.38
7.36
7.36
7.28
4.30
4.28
4.26
4.25
1.25
1.24
1.22
3s
COOEt


162.67
143.78
140.46
140.22
134.79
129.74
128.77
128.01
127.13
125.30
124.79
122.49
77.40
77.08
76.77
61.25
13.98
3s
S
COOEt
f1 (ppm)


MeO
S
COOEt
3t
1.13
3.04
2.02
0.97
0.93
2.10
3.00
3.10
7.78
7.76
7.54
7.53
7.53
7.51
7.51
7.50
7.49
7.48
7.48
7.47
7.43
7.42
7.42
7.41
7.40
7.28
7.17
7.17
7.15
7.14
6.95
6.94
4.26
4.25
4.23
4.21
3.77
1.22
1.21
1.19


MeO
S
COOEt
3t
162.56
158.08
143.41
141.44
134.53
133.08
129.54
127.95
127.60
123.37
122.86
117.91
106.23
77.32
76.68
61.20
55.40
13.77


3u
Cl
S
COOEt
7.83
7.81
7.52
7.52
7.51
7.50
7.50
7.47
7.47
7.45
7.44
7.41
7.40
7.39
7.38
7.28
4.28
4.26
4.24
4.22
1.23
1.21
1.19
1.00
4.10
1.16
2.10
2.12
3.20
f1 (ppm)


3u
Cl
S
COOEt
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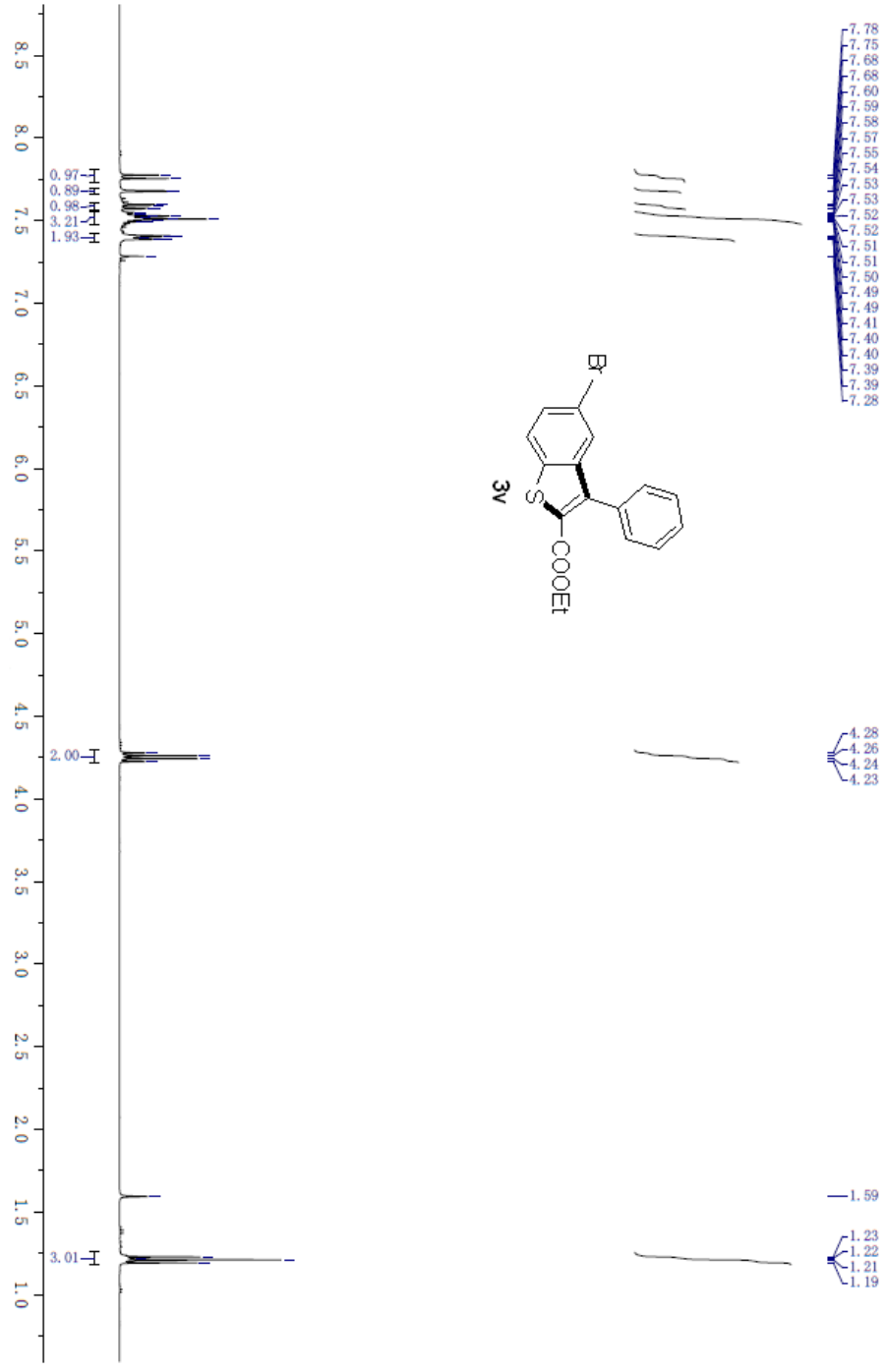
142.87
141.35
138.42
134.08
131.31
130.62
129.61
128.27
128.16
127.68
124.62
123.60
77.32
77.20
77.00
76.68
61.43
13.90
f1 (ppm)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5.0
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4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.97
0.89
0.98
3.21
1.93
2.00
3.01
7.78
7.75
7.68
7.68
7.60
7.59
7.58
7.57
7.55
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7.39
7.28
4.28
4.26
4.24
4.23
1.59
1.23
1.22
1.21
1.19
Br
S
COOEt
3v