

**One-pot synthesis of benzolactones and lactams via a
cobalt-catalyzed regioselective [2+2+2] cocyclotrimerization
of alkynyl alcohols and amines with propiolates**

Hong-Tai Chang, Masilamani Jeganmohan, and Chien-Hong Cheng*

Department of Chemistry, National Tsing Hua University, Hsinchu, 30013 Taiwan

Fax: 886-3-5724698; Tel: 886-3-5721454

E-mail: chcheng@mx.nthu.edu.tw

Electronic Supplementary Information

Experimental Section

General procedure for the cobalt-catalyzed [2+2+2] cocyclotrimerization of alkynyl alcohols and propiolates:

A 25-mL round-bottom flask containing $\text{CoI}_2(\text{dppe})$ (0.050 mmol), Zn (2.75 mmol), DMAD **2** or propiolate **4** (2.2 mmol) was evacuated and purged with nitrogen gas three times. Freshly distilled solvents CH_3CN and THF (2.0 + 2.0 mL) and alkynyl alcohol **1** (1.0 mmol) were added. The reaction mixture was initially stirred at room temperature for 1 h to reduce Co(II) (pale green) to active species (dark green) species and then at 80 °C for 12 h (For Scheme 1, 2, 4, and 5 heat at 80 °C for 12 h, and Scheme 3 and 6 heat at 80 °C for 16 h). The reaction mixture was then filtered through Celite and silica gel, and eluted with dichloromethane. The filtrate was concentrated, and the residue was purified on a silica gel column using hexanes-ethyl acetate as eluent to afford the desired products **3** or **5**. Products **7a-b**, and **9** were synthesized according to similar procedures using propargyl amines **6** and propiolate **4c**, and unsymmetrical diyne **8** and propargyl alcohol **1a** as the reagents, respectively. In addition, product **13** was synthesized based on a similar procedure using $\text{Ni}(\text{dppe})\text{Br}_2$ as the catalyst.

Product yields of these reactions are listed in Tables **1**, and **2**, while spectral data and the ^1H and ^{13}C NMR spectra of compounds **3a-g**, **5a-f**, **7a-b**, **9** and **13** are shown below.

Trimethyl 3-oxo-1,3-dihydro-4,5,6-isobenzofurantricarboxylate (3a): Colorless solid; m.p 152 °C; IR (neat); 1771, 1738, 1731, 1723, 1339, 1295, 1283, 1260 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3); δ 7.95 (s, 1 H), 5.34 (s, 2 H), 3.95 (s, 3 H), 3.89 (s, 3 H), 3.86 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3); δ 166.8, 165.8, 165.3, 164.6, 148.4, 135.7, 132.8, 131.9, 125.6, 124.8, 69.0, 53.3, 53.3, 53.3; HRMS calcd for $\text{C}_{14}\text{H}_{12}\text{O}_8$ 308.0532 found 308.0535.

3'-Oxo-3'H-spiro[cyclohexane-1,1'-isobenzofuran]-4',5',6'-tricarboxylic acid

trimethyl ester (3b): Pale yellow solid; m.p 122 °C; IR (neat): 1779, 1747, 1742, 1731, 1341, 1290, 1264, 1233 cm⁻¹; ¹H NMR (400 MHz, CDCl₃); δ 7.86 (s, 1 H), 3.96 (s, 3 H), 3.90 (s, 3 H), 3.87 (s, 3 H), 1.85-1.75 (m, 10 H); ¹³C NMR (100 MHz, CDCl₃); δ 165.8, 165.7, 165.5, 164.8, 156.5, 135.9, 132.6, 132.4, 125.4, 123.5, 86.8, 53.3, 53.2, 35.9, 24.4, 21.9; HRMS calcd for C₁₉H₂₀O₈ 376.1158 found 376.1158.

Trimethyl 7-methyl-3-oxo-1,3-dihydro-4,5,6-isobenzofurantricarboxylate (3c):

Pale yellow solid; m.p 102 °C; IR (neat): 1776, 1732, 1341, 1297, 1252, 1218 cm⁻¹; ¹H NMR (400 MHz, CDCl₃); δ 5.28 (s, 2 H), 3.97 (s, 3 H), 3.91 (s, 3 H), 3.86 (s, 3 H), 2.31 (s, 3 H); ¹³C NMR (100 MHz, CDCl₃); δ 167.4, 166.9, 165.2, 164.7, 149.4, 139.2, 132.4, 131.1, 128.7, 123.4, 68.7, 53.2, 53.2, 52.9, 15.0; HRMS calcd for C₁₅H₁₄O₈ 322.0689 found 322.0691.

Dimethyl 3,6-dioxo-1,3,6,8-tetrahydrofuro[3,4-e]isobenzofuran-4,5-dicarboxylate (3d): Colorless solid; m.p 130 °C; IR: 1791, 1766, 1744, 1733, 1310, 1221, 1210 cm⁻¹; ¹H NMR (400 MHz, CDCl₃); δ 5.42 (s, 4 H), 4.01 (s, 6 H); ¹³C NMR (100 MHz, d-acetone); δ 167.5, 165.1, 145.4, 131.7, 128.3, 69.4, 53.5; HRMS calcd for C₁₄H₁₀O₈ 306.0376 found 306.0376.

Trimethyl 1-oxo-3,4-dihydro-1H-6,7,8-isochromenetricarboxylate (3e): Colorless solid; m.p 158 °C; IR (neat): 1749, 1318, 1283 cm⁻¹; ¹H NMR (400 MHz, CDCl₃); δ 7.80 (s, 1 H), 4.54 (t, J = 6.0 Hz, 2 H), 3.90 (s, 3 H), 3.90 (s, 3 H), 3.87 (s, 3 H), 3.11 (t, J = 6.0Hz, 2 H); ¹³C NMR (100 MHz, CDCl₃); δ 166.8, 166.2, 165.2, 161.5, 142.2, 136.1, 133.9, 132.1, 129.6, 126.1, 66.8, 53.3, 53.2, 53.2, 28.2; HRMS calcd for C₁₅H₁₄O₈ 322.0689 found 322.0688.

Trimethyl 1-oxo-1,3,4,5-tetrahydro-2-benzoxepine-7,8,9-tricarboxylate (3f):

Colorless solid; m.p 126 °C; IR (neat): 1740, 173, 1328, 1283, 1248 cm⁻¹; ¹H NMR

(400 MHz, CDCl₃); δ 7.84 (s, 1 H), 4.20 (t, *J* = 6.4 Hz, 2 H), 3.88 (s, 3 H), 3.82 (s, 3 H), 2.90 (t, *J* = 6.8 Hz, 2 H), 2.09 (m, 2 H); ¹³C NMR (100 MHz, CDCl₃); δ 168.6, 167.0, 165.9, 164.9, 138.2, 135.4, 133.6, 132.7, 131.8, 131.7, 60.0, 53.3, 53.0, 53.0, 28.9, 26.8; HRMS calcd for C₁₆H₁₆O₈ 336.0845 found 336.0853.

Tetramethyl 5-(4-hydroxybutyl)-1,2,3,4-benzenetetracarboxylate (3g): Pale yellow viscous oil; IR (neat): 1735, 1326, 1299, 1263, 1247, 1209 cm⁻¹; ¹H NMR (400 MHz, CDCl₃); δ 7.92 (s, 1 H), 3.86 (s, 3 H), 3.86 (s, 3 H), 3.84 (s, 3 H), 3.81 (s, 3 H), 3.59 (t, *J* = 6.4 Hz, 2 H), 2.67 (t, *J* = 7.2 Hz, 2 H), 1.66-1.62 (m, 2 H), 1.56-1.54 (m, 2 H); ¹³C NMR (100 MHz, CDCl₃); δ 167.5, 167.5, 165.7, 165.0, 142.5, 136.6, 133.8, 133.0, 129.9, 129.7, 61.9, 53.0, 52.9, 52.7, 52.7, 32.8, 31.9, 27.1; HRMS calcd for C₁₈H₂₂O₉ 382.1264 found 382.1266.

Methyl 1-oxo-6,7-dipentyl-1,3-dihydro-5-isobenzofurancarboxylate (5a): Pale yellow viscous oil; IR (neat): 1755, 1730, 1312, 1218 cm⁻¹; ¹H NMR (400 MHz, CDCl₃); δ 7.54 (s, 1 H), 5.18 (s, 2 H), 3.91 (s, 3 H), 3.13 (t, *J* = 8.0 Hz, 2 H), 2.86 (t, *J* = 8.0 Hz, 2 H), 1.50-1.32 (m, 16 H), 0.91-0.87 (m, 6 H); ¹³C NMR (100 MHz, CDCl₃); δ 170.3, 168.6, 144.4, 144.1, 142.2, 136.9, 125.1, 120.4, 68.6, 52.5, 32.2, 32.2, 31.5, 31.2, 28.9, 26.9, 22.4, 22.3, 13.9, 13.9; HRMS calcd for C₂₀H₂₈O₄ 332.1988 found 332.1985.

Methyl 1-oxo-6,7-diphenyl-1,3-dihydro-5-isobenzofurancarboxylate (5b): Pale yellow solid; IR (neat): 1752, 1710, 1330, 1241 cm⁻¹; ¹H NMR (400 MHz, CDCl₃); δ 7.81 (s, 1 H), 7.20-7.17 (m, 3 H), 7.15-7.13 (m, 3 H), 7.00-6.98 (m, 2 H), 6.95-6.93 (m, 2 H), 5.36 (s, 2 H), 3.55 (s, 3 H); ¹³C NMR (100 MHz, CDCl₃); δ 168.7, 168.4, 145.8, 142.5, 142.1, 138.4, 137.5, 134.1, 129.9, 129.7, 127.6, 127.5, 127.3, 127.2, 124.9, 121.3, 67.9, 52.4; HRMS calcd for C₂₂H₁₆O₄ 344.1049 found 344.1050.

Methyl 3-methyl-1-oxo-6,7-diphenyl-1,3-dihydro-5-isobenzofurancarboxylate (5c): Pale yellow solid; m.p 141 °C; IR: 1763, 1737, 1327, 1222 cm⁻¹; ¹H NMR (400

MHz, CDCl₃); δ 7.73 (s, 1 H), 7.18-7.17 (m, 3 H), 7.15-7.13 (m, 3 H), 7.00-6.98 (m, 2 H), 6.98-6.94 (m, 2 H), 5.56 (q, *J* = 6.8 Hz, 1 H), 3.53 (s, 3 H), 1.70 (d, *J* = 6.8 Hz, 3 H); ¹³C NMR (100 MHz, CDCl₃); δ 168.5, 168.1, 150.5, 142.4, 142.0, 138.4, 137.5, 134.2, 129.9, 129.7, 127.5, 127.3, 127.1, 124.9, 120.8, 75.8, 52.4, 20.5; HRMS calcd for C₂₃H₁₈O₄ 358.1205 found 358.1204.

Methyl 1-oxo-1,3-dihydro-5-isobenzofurancarboxylate (5d): Colorless solid; m.p 151 °C; IR (neat): 1755, 1719, 1285, 1208 cm⁻¹; ¹H NMR (400 MHz, CDCl₃); δ 8.17 (d, *J* = 8.0 Hz, 1 H), 8.15 (s, 1 H), 7.95 (d, *J* = 8.0 Hz, 1 H), 5.35 (s, 2 H), 3.95 (s, 3 H); ¹³C NMR (100 MHz, CDCl₃); δ 169.9, 165.7, 146.4, 135.3, 130.3, 129.3, 125.7, 123.5, 69.6, 52.7; HRMS calcd for C₁₀H₈O₄ 192.0423 found 192.0421.

Methyl 3-methyl-1-oxo-1,3-dihydro-5-isobenzofurancarboxylate (5e): Pale yellow solid; m.p 117 °C; IR (neat): 1764, 1724, 1284, 1209 cm⁻¹; ¹H NMR (400 MHz, CDCl₃); δ 8.15 (d, *J* = 7.6 Hz, 1 H), 8.08 (s, 1 H), 7.91 (d, *J* = 8.0 Hz, 1 H), 5.58 (q, *J* = 6.8 Hz, 1 H), 3.94 (s, 3 H), 1.64 (d, *J* = 6.8 Hz, 3 H); ¹³C NMR (100 MHz, CDCl₃); δ 169.3, 165.7, 151.0, 135.3, 130.3, 129.4, 125.7, 122.9, 77.8, 52.7, 20.2; HRMS calcd for C₁₁H₁₀O₄ 206.0579 found 206.0580.

Methyl 1-oxo-3,4-dihydro-1*H*-6-isochromenecarboxylate (5f): Colorless solid; m.p 112 °C; IR (neat): 1764, 1724, 1284, 1209 cm⁻¹; ¹H NMR (400 MHz, CDCl₃); δ 8.12 (d, *J* = 8.0 Hz, 1 H), 7.99 (d, *J* = 8.0 Hz, 1 H), 7.91 (s, 1 H), 4.53 (t, *J* = 6.0 Hz, 2 H), 3.92 (s, 3 H), 3.09 (t, *J* = 6.0 Hz, 2 H); ¹³C NMR (100 MHz, CDCl₃); δ 165.9, 164.1, 139.5, 134.4, 130.5, 128.8, 128.4, 128.4, 67.3, 52.5, 27.7; HRMS calcd for C₁₁H₁₀O₄ 206.0579 found 206.0575.

Methyl 2-[(4-methylphenyl)sulfonyl]-1-oxo-5-isoindolinecarboxylate (7a): Colorless solid; m.p 172 °C; IR (neat): 1739, 1707, 1357, 1291, 1212 cm⁻¹; ¹H NMR (400 MHz, CDCl₃); δ 8.12 (s, 1 H), 8.11 (d, *J* = 9.6 Hz, 1 H), 8.00 (d, *J* = 8.4 Hz, 2 H), 7.83 (d, *J* = 8.0 Hz, 1 H), 7.32 (d, *J* = 8.0 Hz, 2 H), 4.92 (s, 2 H), 3.93 (s, 3 H), 2.39 (s,

3 H); ^{13}C NMR (100 MHz, CDCl_3); δ 165.7, 165.1, 145.5, 140.9, 135.1, 134.7, 130.0, 129.8, 128.2, 125.0, 124.7, 77.9, 52.7, 49.7, 21.7; HRMS calcd for $\text{C}_{17}\text{H}_{15}\text{NO}_5\text{S}$ 345.0671 found 345.0672.

Methyl 2-[*(E*)-3-methoxy-3-oxo-1-propenyl]-1-oxo-5-isoindolinecarboxylate (7b**):**

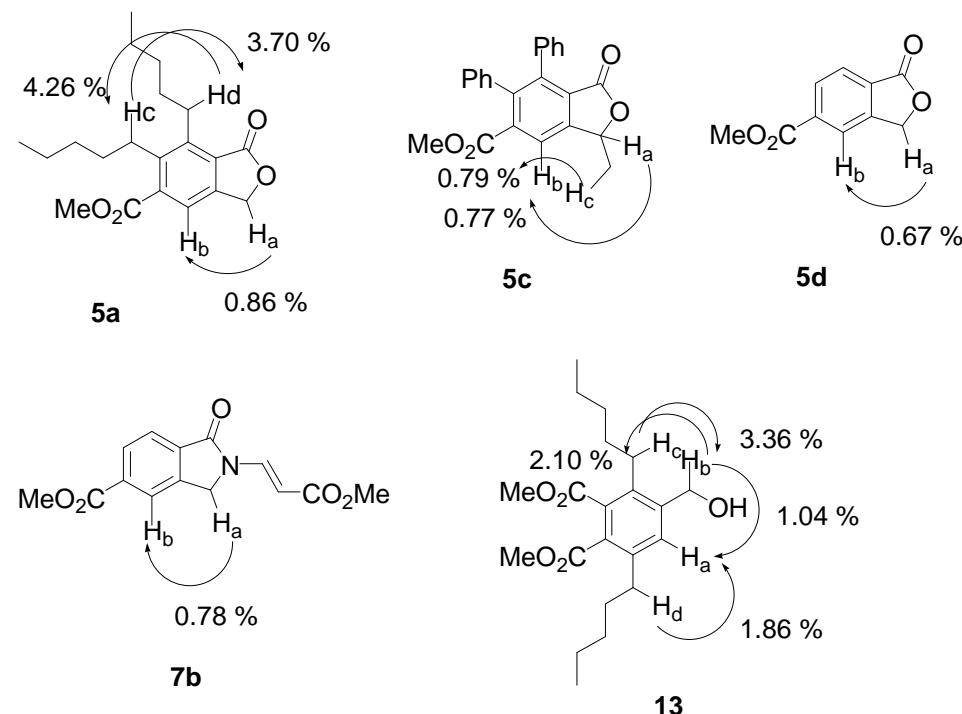
Colorless solid; m.p 177 °C; IR (neat): 1726, 1708, 1637, 1295, 1266 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3); δ 8.34 (d, $J = 14.4$ Hz, 1 H), 8.19 (s, 1 H), 8.18 (d, $J = 6.5$ Hz, 1 H), 7.97 (d, $J = 8.4$ Hz, 1 H), 5.48 (d, $J = 14.4$ Hz, 1 H), 4.62 (s, 2 H), 3.96 (s, 3 H), 3.77 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3) ; δ 167.3, 165.9, 140.6, 137.0, 134.8, 134.5, 130.2, 124.8, 124.5, 100.5, 52.7, 51.6, 47.9; HRMS calcd for $\text{C}_{14}\text{H}_{13}\text{NO}_5$ 275.0794 found 275.0793.

1,3,6,8-Tetrahydrofuro[3,4-*e*]isobenzofuran-1-one (9**):** Colorless solid; m.p 73 °C; IR (neat): 1748, 1347, 1237, 1146 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3); δ 7.52 (d, $J = 8.0$ Hz, 1 H), 7.38 (d, $J = 7.6$ Hz, 1 H), 5.38 (s, 2 H), 5.35 (s, 2 H), 5.14 (s, 2 H); ^{13}C NMR (100 MHz, CDCl_3); δ 170.2, 146.0, 141.3, 138.4, 126.6, 121.2, 120.2, 72.8, 72.2, 70.4; HRMS calcd for $\text{C}_{10}\text{H}_8\text{O}_3$ 176.0473 found 176.0473.

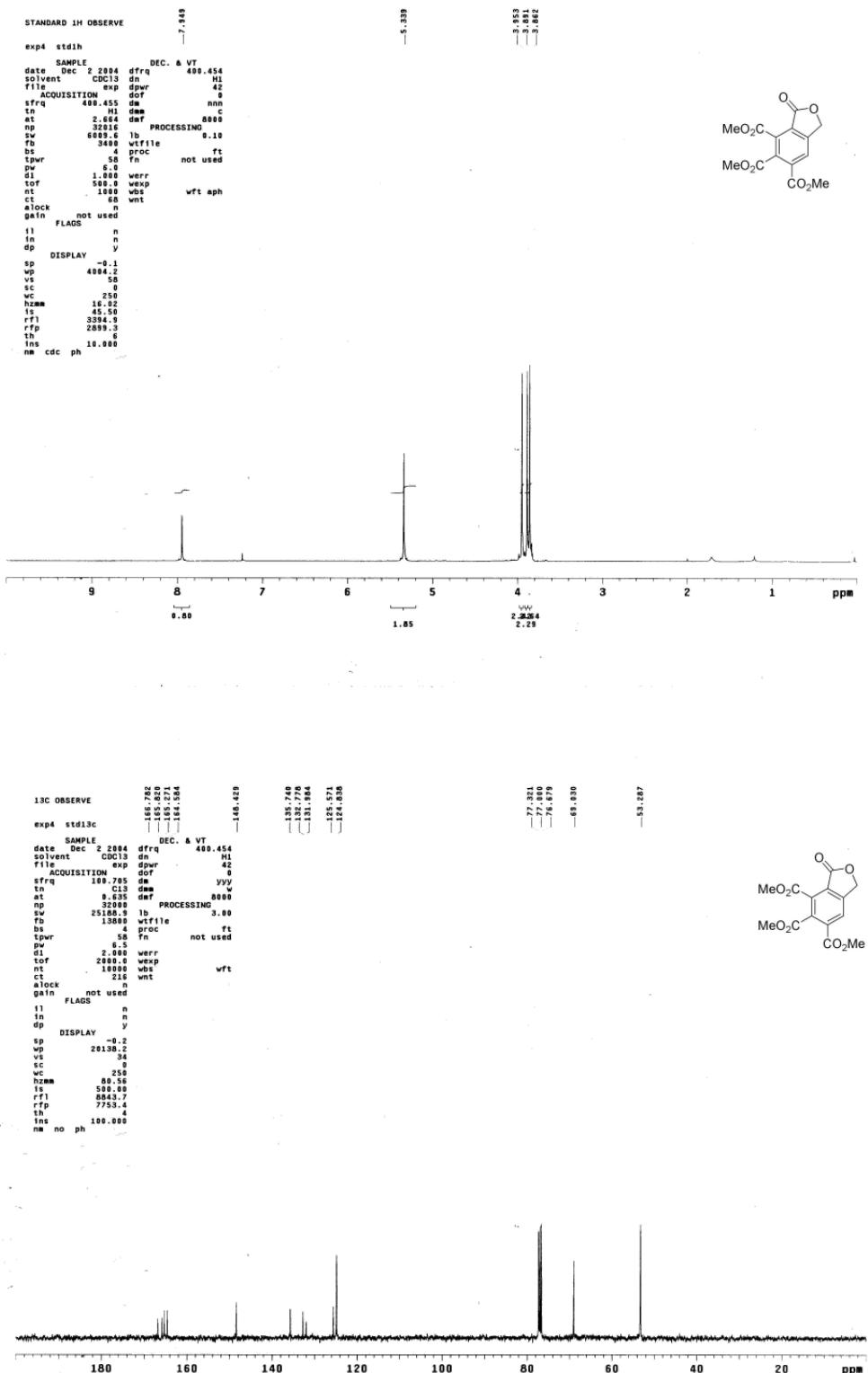
Dimethyl 4-(hydroxymethyl)-3,6-dipentylphthalate (13**):** Pale yellow viscous oil; IR (neat): 1735, 1283, 1208, 1173 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3); δ 7.39 (s, 1 H), 4.73 (s, 2 H), 3.83 (s, 3 H), 3.83 (s, 3 H), 2.67-2.63 (m, 2 H), 2.62-2.59 (m, 2 H), 1.54-1.48 (m, 4 H), 1.32-1.22 (m, 8 H), 0.88-0.84 (m, 6 H); ^{13}C NMR (100 MHz, CDCl_3); δ 169.4, 168.8, 141.5, 139.7, 136.1, 132.9, 130.5, 130.1, 62.0, 52.2, 52.2, 33.6, 32.1, 31.7, 31.2, 30.8, 29.2, 22.4, 22.3, 13.9; HRMS calcd for $\text{C}_{21}\text{H}_{32}\text{O}_5$ 364.225 found 364.225.

The structures of [2+2+2] coccyclotrimerization products **5a-f**, **7a-b**, and **13** were established by ^1H NMR NOE experiments. For example compound **5a**, selective irradiation of the methylene protons H_a in the lactone ring at δ 5.30 led to an enhancement of the signal H_b at δ 7.04 by 0.86 % in the aromatic region, irradiation

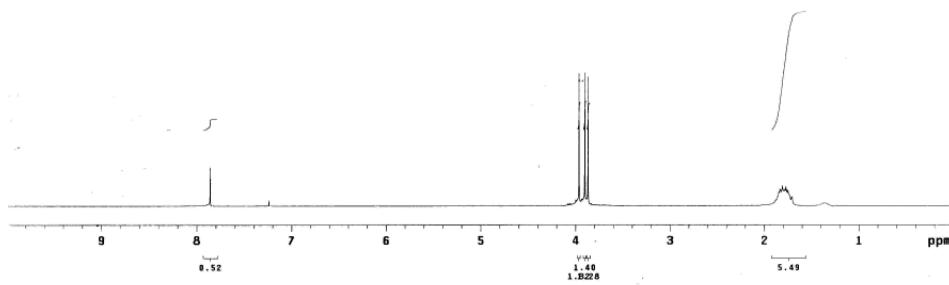
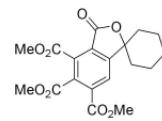
of the H_d signal at δ 2.84 caused an enhancement of the H_c signal at δ 2.64 by 4.26 % and irradiation of the H_c signal at δ 2.64 caused an enhancement of the H_d signal at δ 2.84 by 3.70 % for the methylene protons directly attached to aromatic ring. Similar NOE results were obtained for **5c**, **d**, **7b** and **13** and were shown below. These NOE results strongly confirm the proposed structures of these compounds.



¹H and ¹³C NMR Spectra of compound 3a:



¹H and ¹³C NMR Spectra of compound 3b:

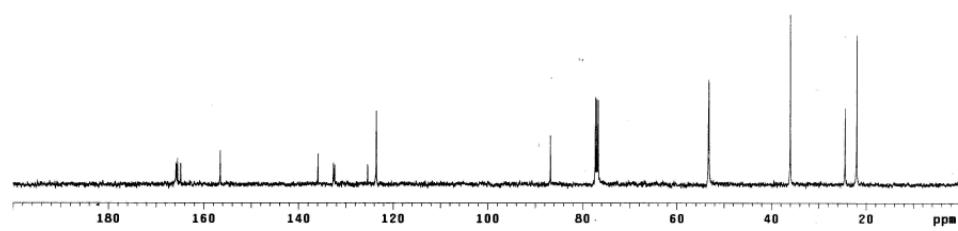
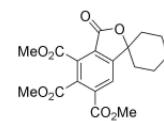


```

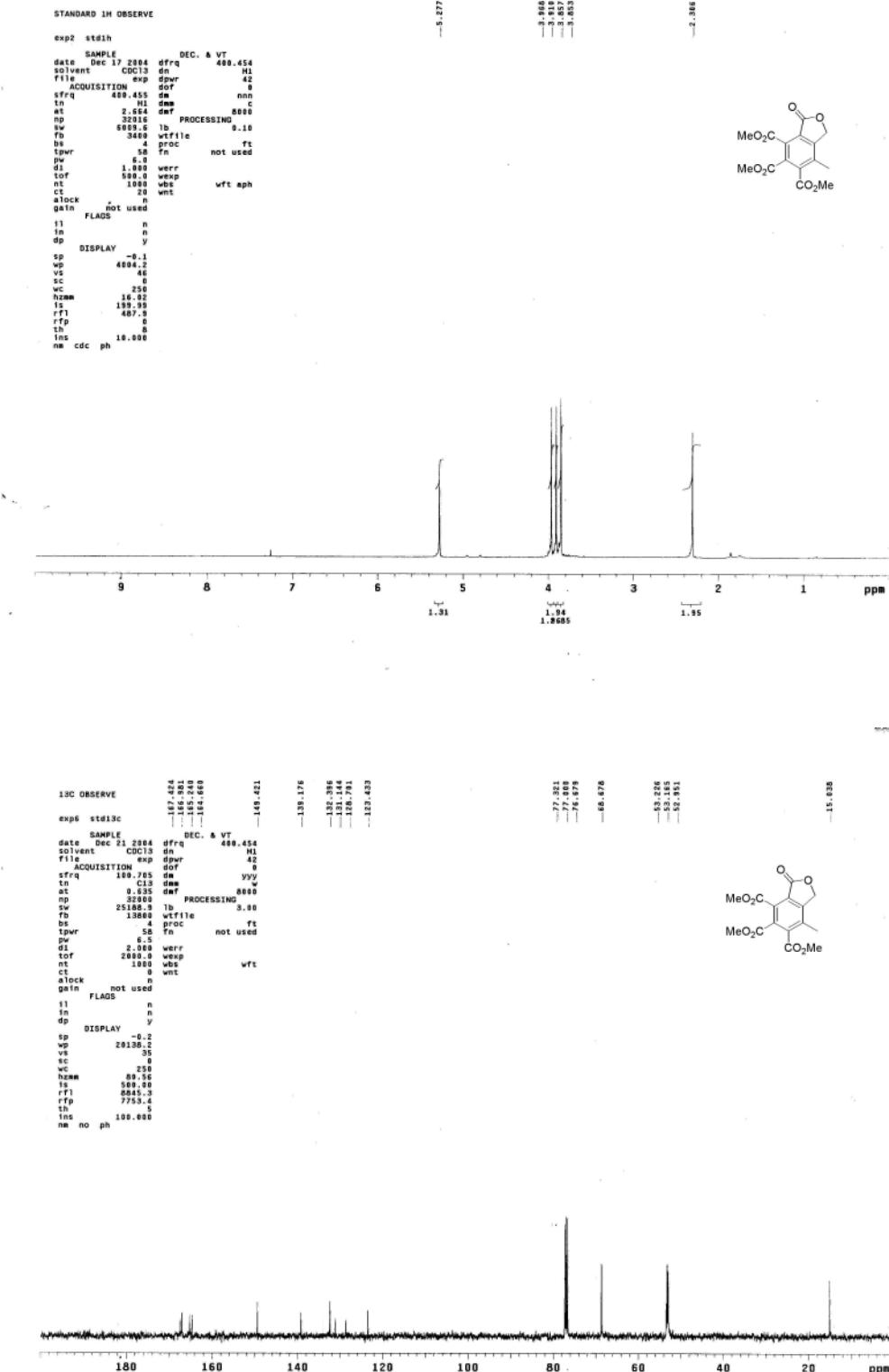
13C OBSERVE

exp8 std13c          SAMPLE           DEC. & VT
                                dfrq      400.45
solvent Dec 8 2006      CUDC      165.785
file      dprw      165.484
ACQUISITION      dprw      165.488
sfreq      100.745      C13      165.517
at        833      daf      800.000
sw        1000      daf      800.000
sw2      25108.5      Ib      PROCESSING 3.01
t1        13880      Ia      100.000
be        1000      proc      f
tpw      5.0      fn      NOT USED
di        2.000      werr
t0f      2000.0      wexp
t1f      1000.0      wexp
ct        0          wnt
clock      n
gain      not used
FLAGS      n
in        n
dp        y
DISPLAY      -9.2
sp        2613.8
wp        100.000
sc        0
scs       256
hwm      88.000
is        500.000
r1        100.000
r2p      7753.4
th        3
nm no ph      100.000

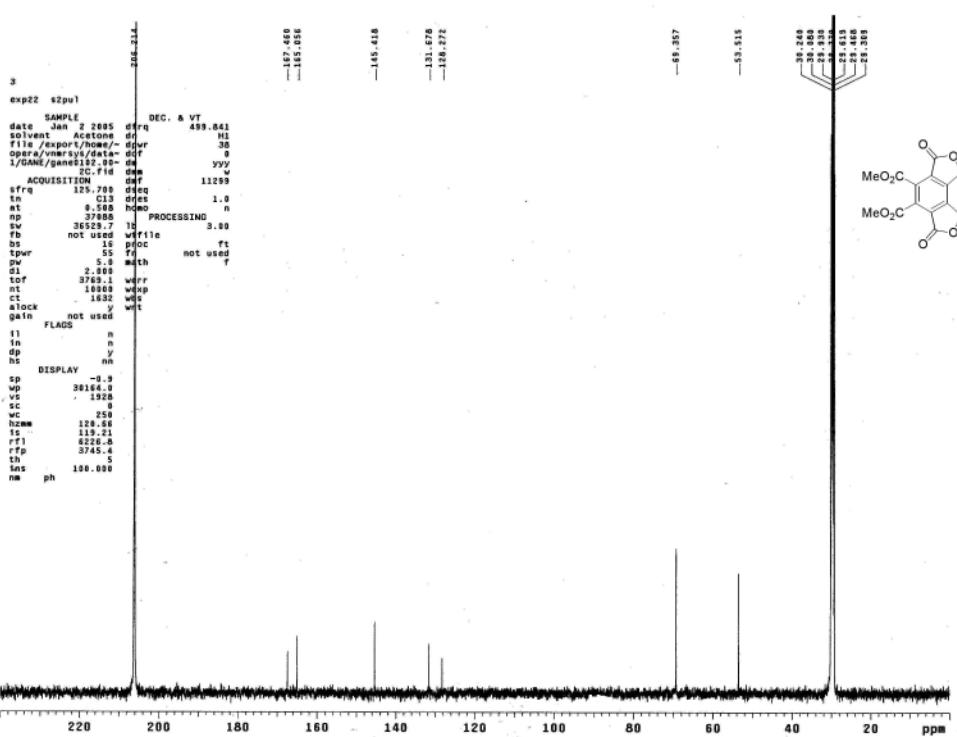
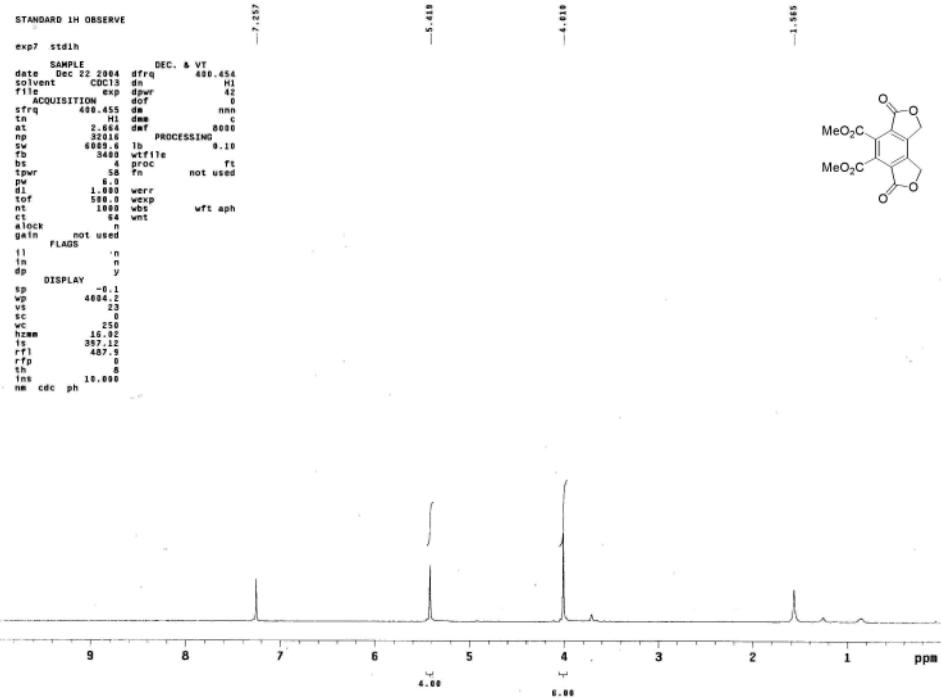
```



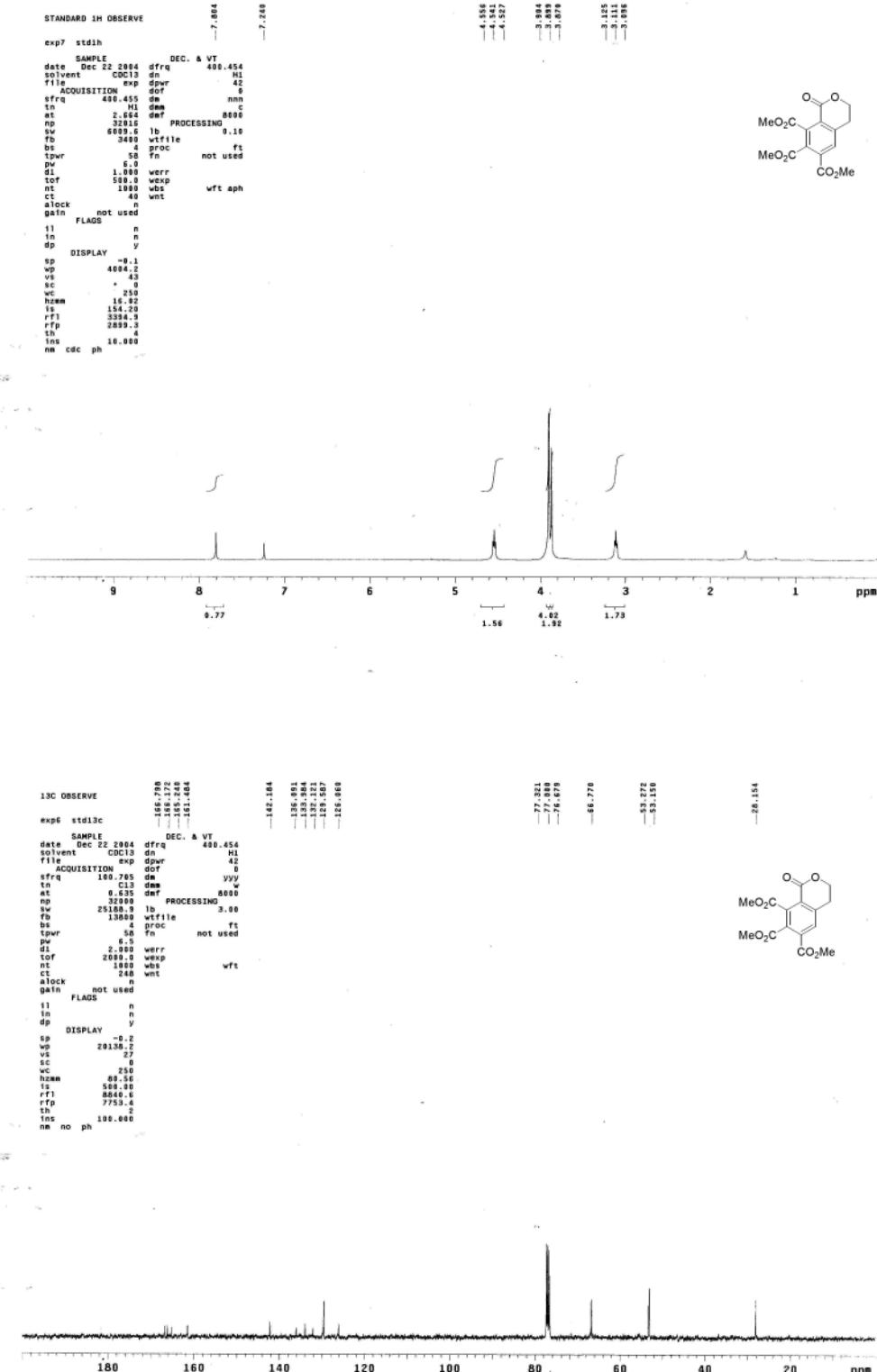
¹H and ¹³C NMR Spectra of compound 3c:



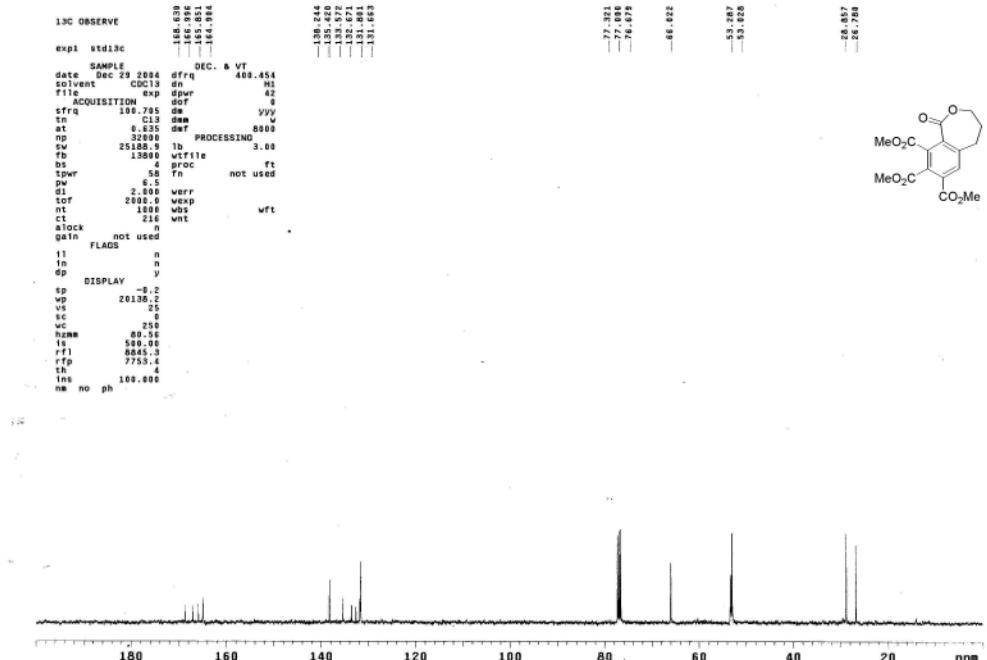
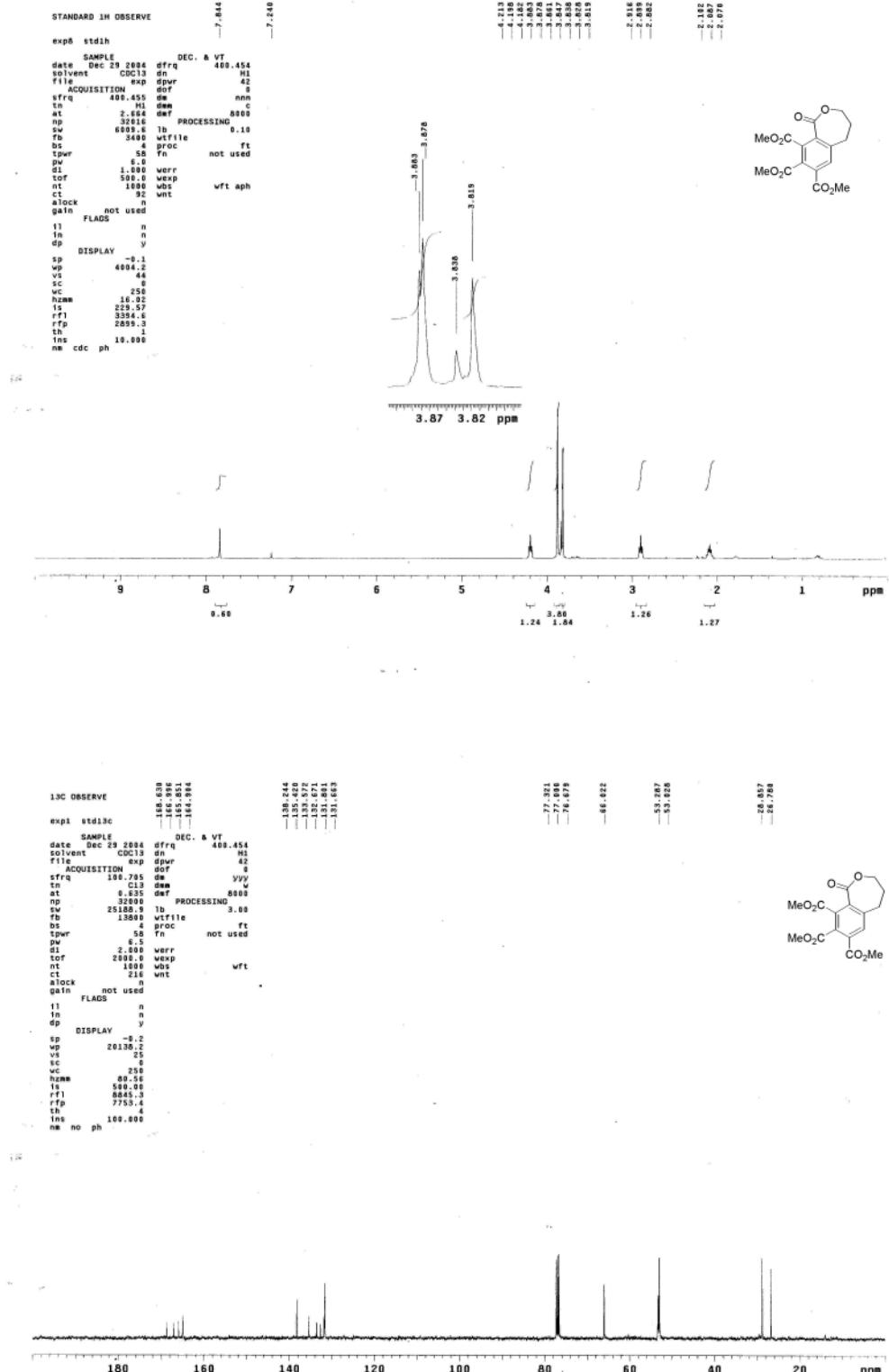
¹H and ¹³C NMR Spectra of compound 3d:



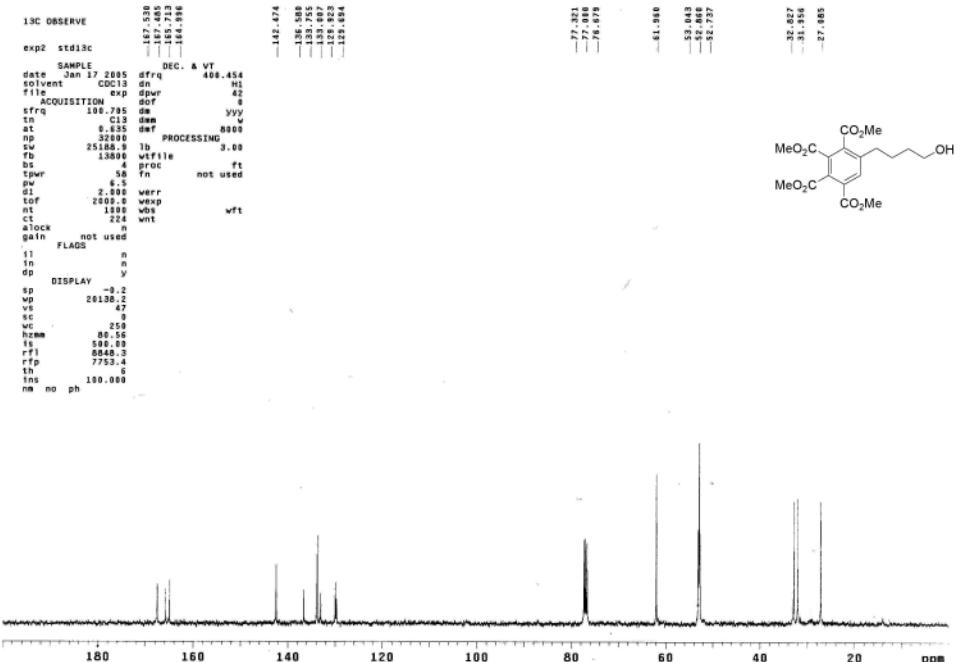
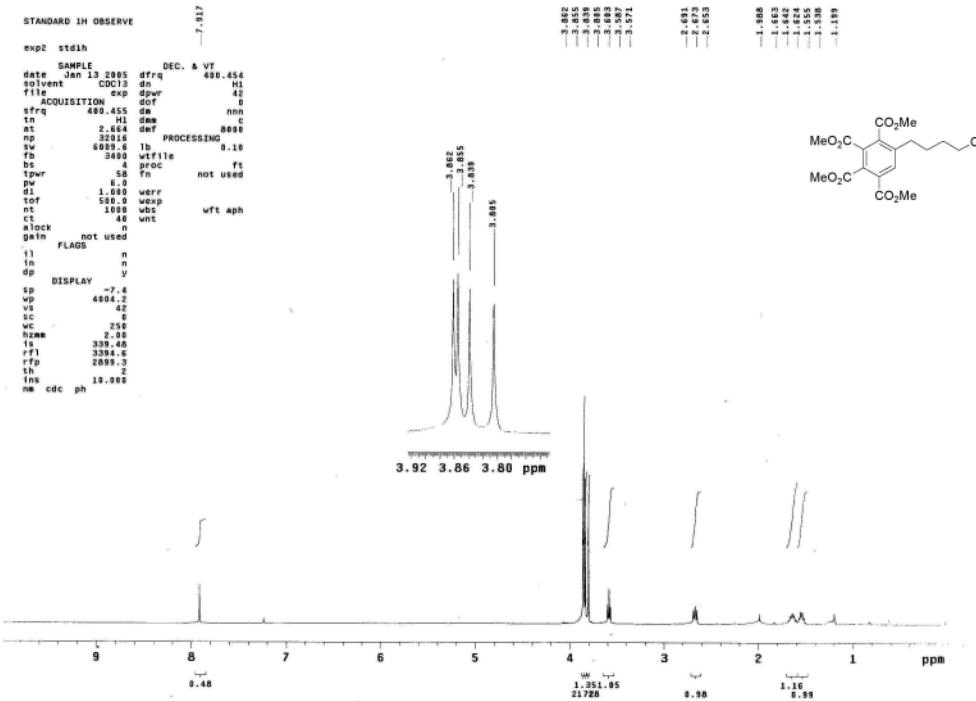
¹H and ¹³C NMR Spectra of compound 3e:



¹H and ¹³C NMR Spectra of compound 3f:



¹H and ¹³C NMR Spectra of compound 3g:



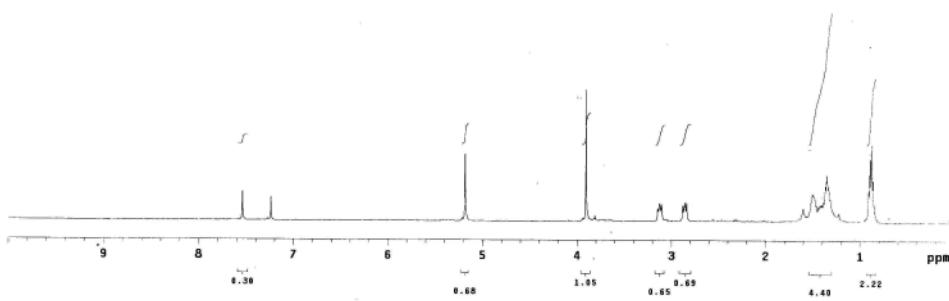
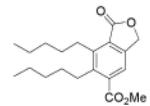
¹H and ¹³C NMR Spectra of compound 5a:

STANDARD 1M OVERVIEW

```

exp stdin      SAMPLE          DEC. & VT
date Jan 18 2005 dfrq 486.454
solvent      exp dpwr 462
sfrq          acqisition 6
sfrq          400.455 dm   mn
tn            2.65      c     8000
32816        PROCESSING
sw            6805.1  b    0.10
sw            6805.1  b    0.10
bs            4    profc  ft
tpwr          56      fn    not used
d1            1.0000  werr
d1            5000.00  wexp
nt            10000.00  wbs
nt            36      wnt  wft sph
clock         gain not used
FLAGS
n1            n
in            n
y
DISPLAY
sp            -e.1
wp            4864
sc            35
sc            0
sc            25
hzmw         16.02
ts            211.31
ts            334.00
rtfp          2865.3
ts             3
mn cdd ph 10.000

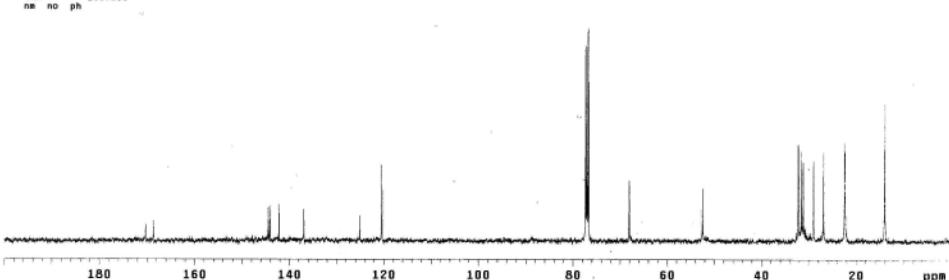
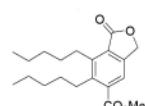
```



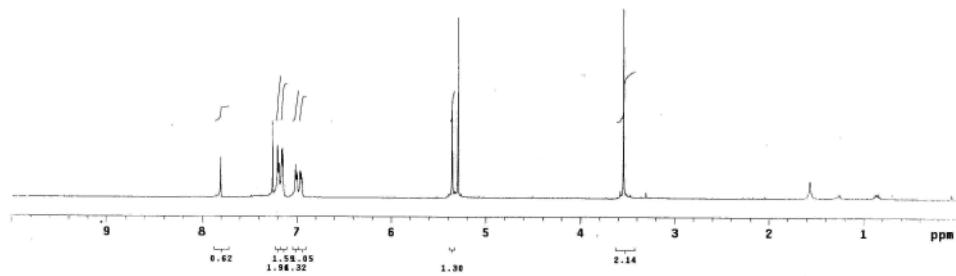
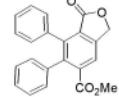
```

exp5 std13c      176   116
SAMPLE          DEC. & VT
date Jan 28 2005 dfrg 454.454
segment C013    dfrg 454.454
sector 00000000000000000000000000000000
ACQUISITION    dof 0
srq# 100.765     yyy
       C13  dms
       0.635     yyy
       32000     PROCESSING 8000
       25100.8    lb 3.66
       138000    wftfile
       58         ft
tpwr 58          not used
pwf 20000        werr
tof 20000        wexp
nt 180000        wbs
       12200        wft
alloc gain      not used
FLAGS
l1 n
n n
dp y
DISPLAY
sp -0.2
20130.8
wp
vs
sc
wc 250
wam 800
is 500.00
rtfl 8888.6
th 7755.3
inc 3
inc 100.800

```



¹H and ¹³C NMR Spectra of compound 5b:

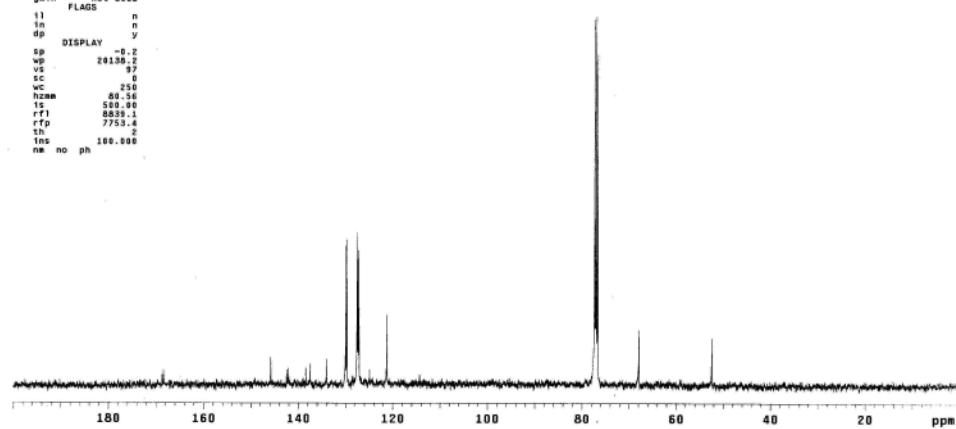
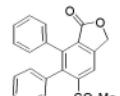


```

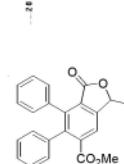
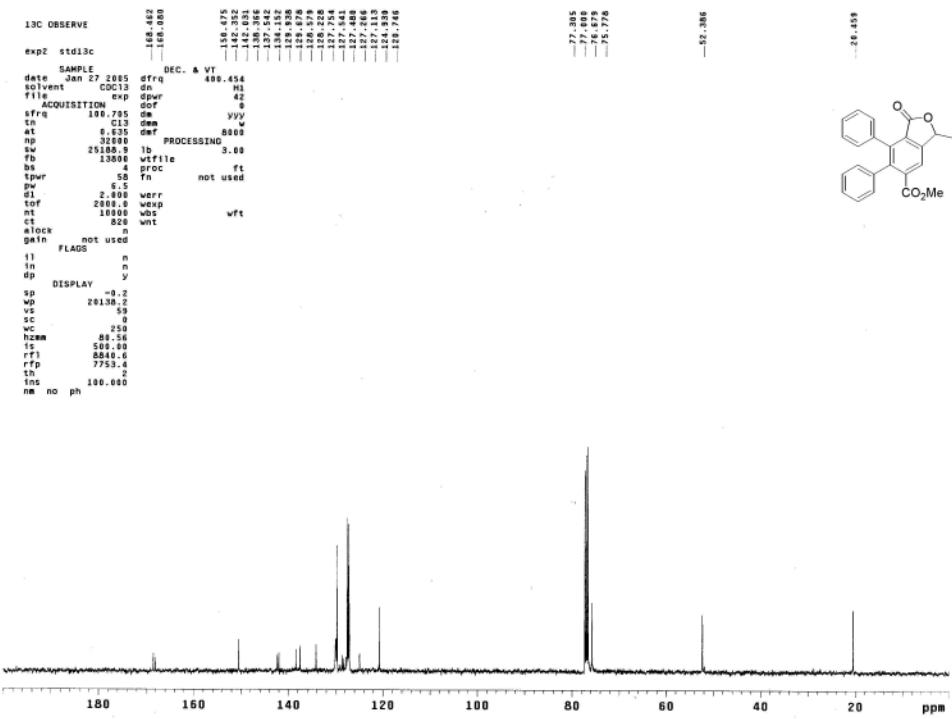
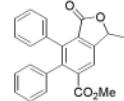
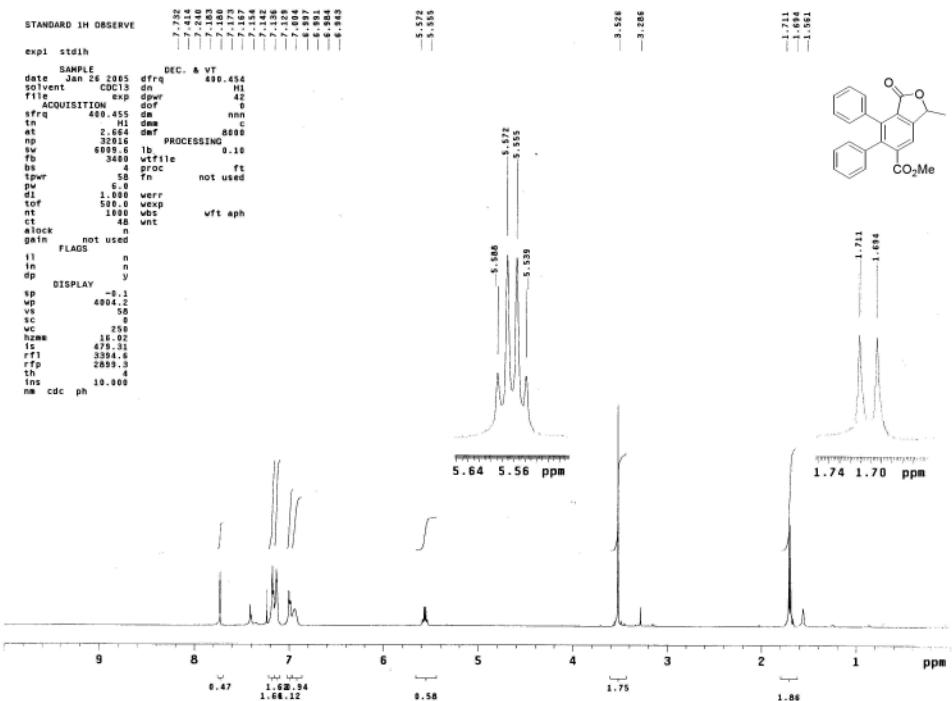
13C OBSERVE

exp5 std13c           -166.721   -166.416          -145.833   -145.459
                                DEC. & VT      480.454
date SAMPLE             date Jan 11 2005 dfrq    480.454
solvent C6DCl3           file exp      dfrq      H1
file ACQUISITION         file exp      dfrq      42
sfrmq 105.700             file dot      dfrq      0
atm      0.000             file vvv      vvv
atn      0.000             file C13      vvv
atp      32098             file dms      vvv
atp      251588.9            PROCESSED
sw       100.000             file 100.000
fb       13864             file 13864
bs       4                 file 4
tpwr     5                 proc   not used
tpwr     5                 fn      not used
d1       2.000             werr
tof      2000.0             wexp
ct       1950.0             wexp
ct       1954.0             wft
block   not used
block   not used
FLAGS
  i1      n
  in      n
  dp      y
DISPLAY
sp       -0.2
wp       28135.0
vt       47
sc       0
kc       0
hmax   40.46
ts       500.00
rtf      8889.1
rtg      7753.4
th       100.000
NB, no ph

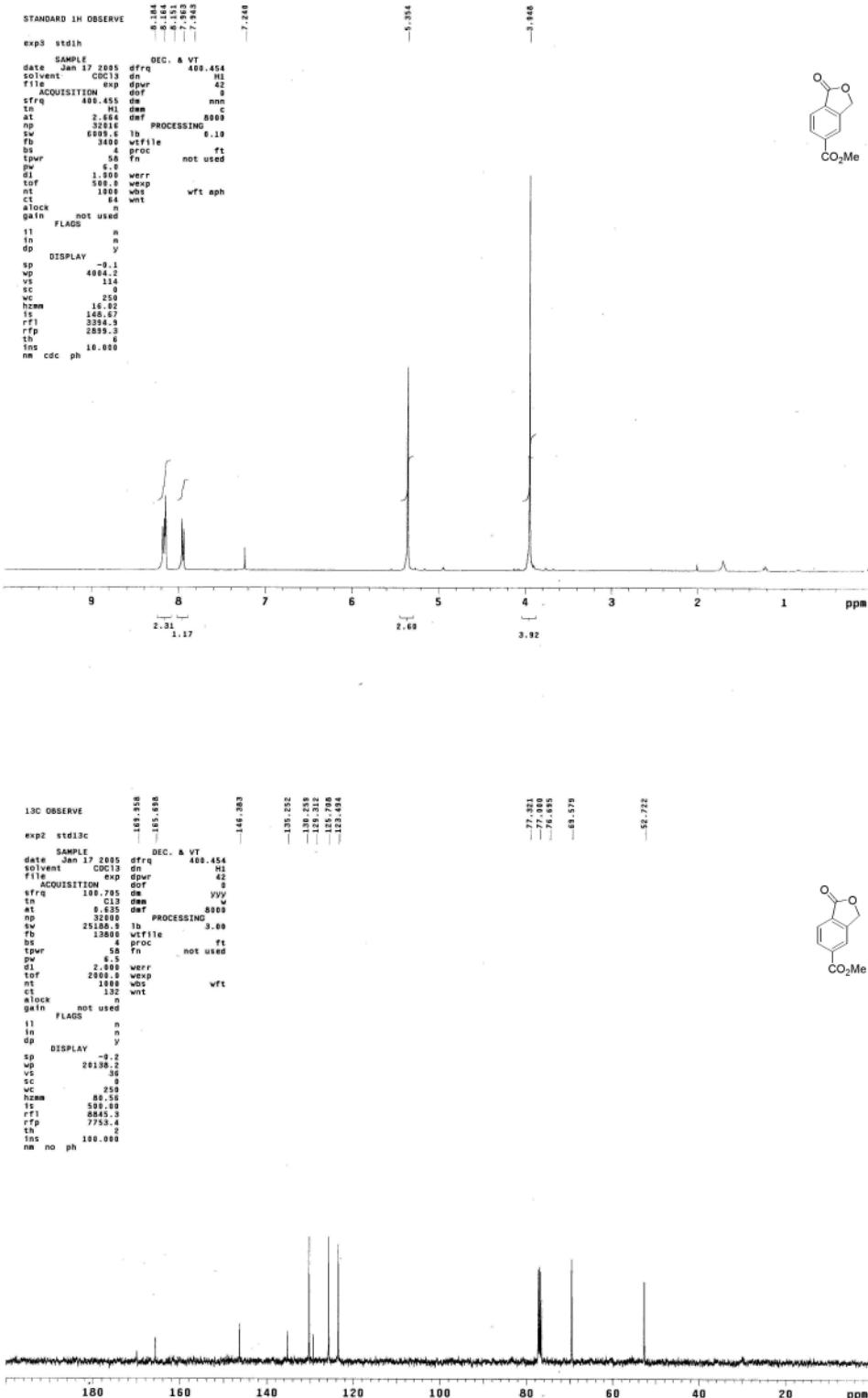
```



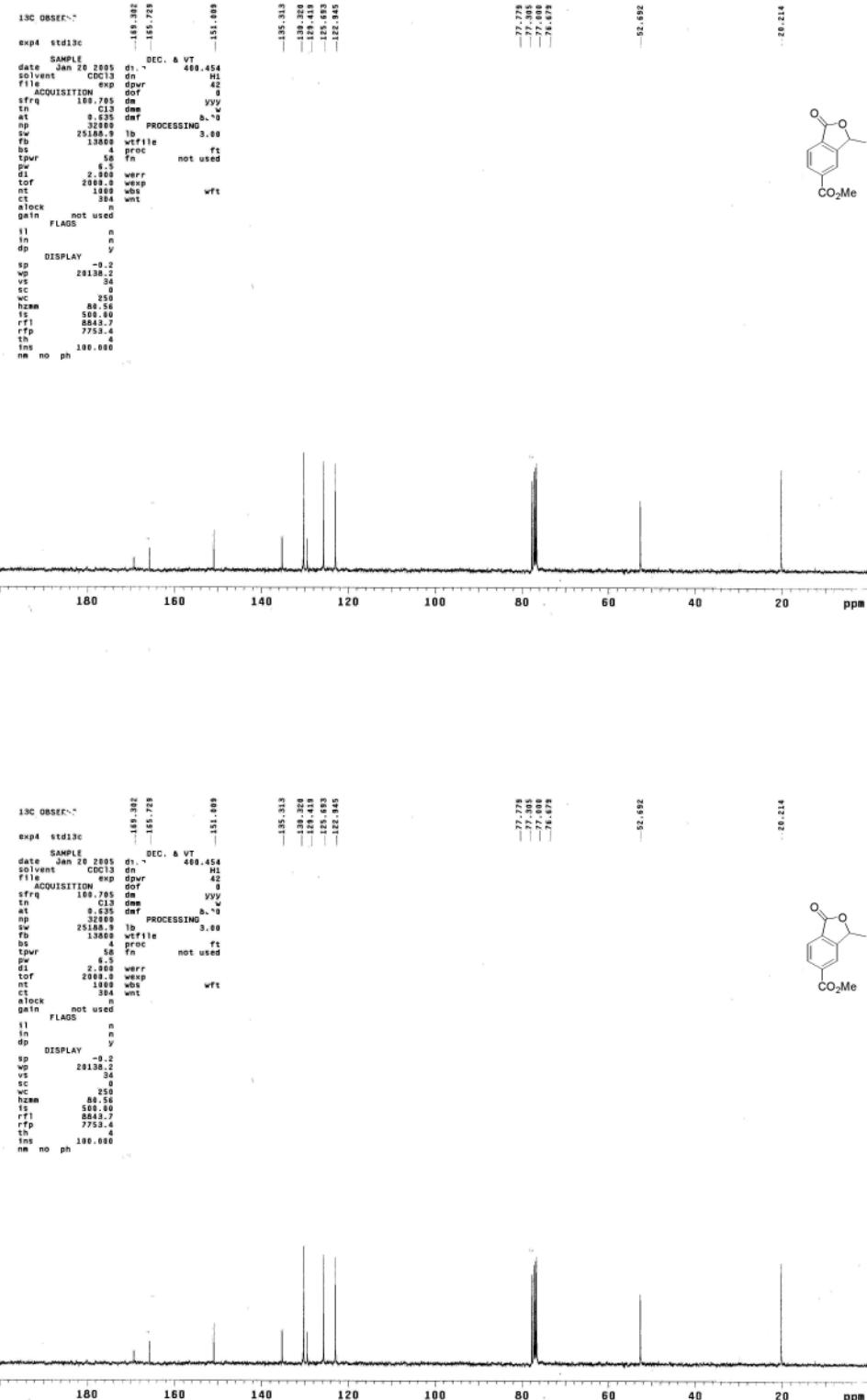
¹H and ¹³C NMR Spectra of compound 5c:



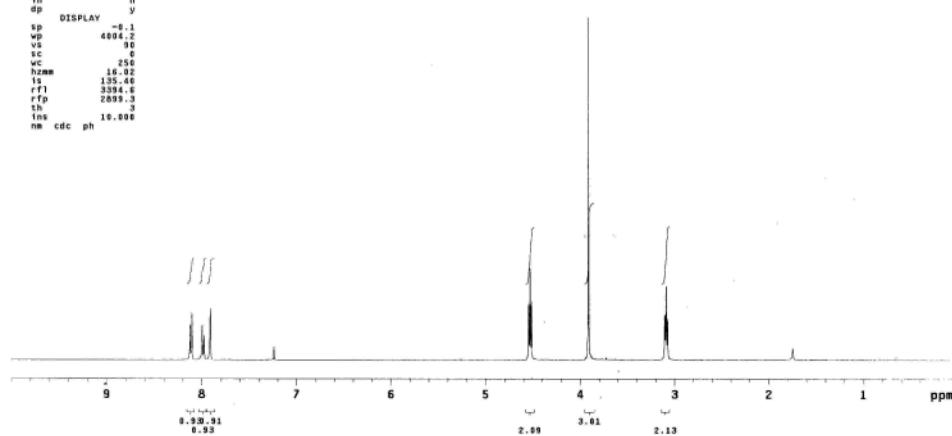
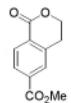
¹H and ¹³C NMR Spectra of compound **5d**:



¹H and ¹³C NMR Spectra of compound 5e:



¹H and ¹³C NMR Spectra of compound **5f**:



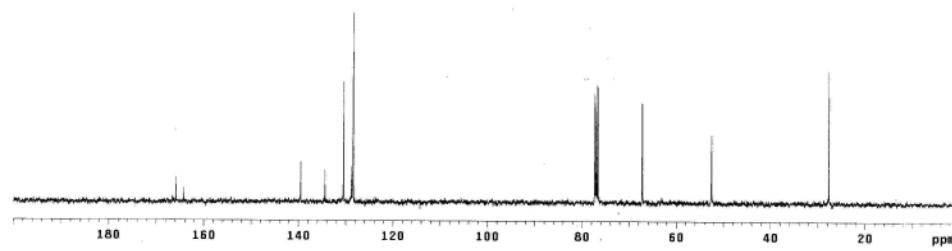
```

13C OBSERVE                                -165.866
                                         -144.141

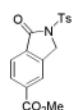
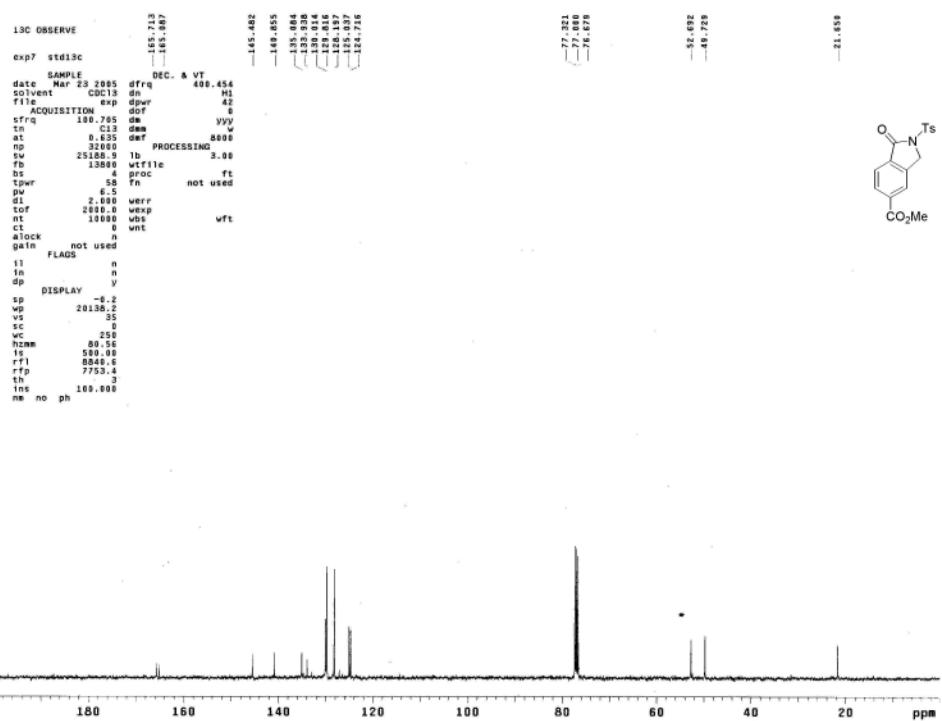
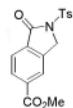
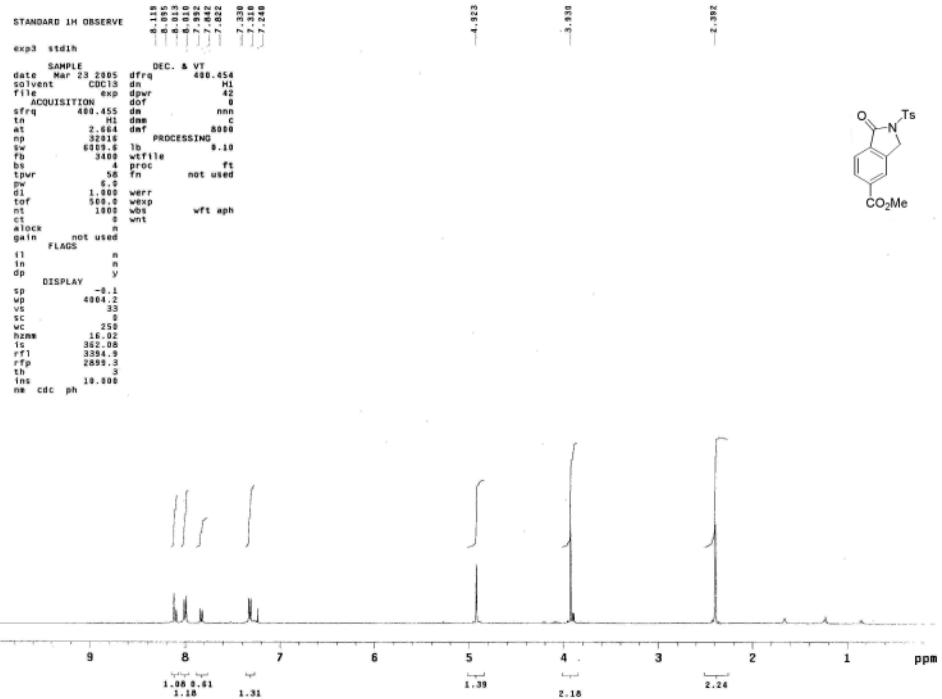
exp4 ttd13c

      SAMPLE          DEC. & VT
      solvent        dfrq    480.453
      file           exp     111
                           dfrw    42
      ACQUISITION    dof     0
      sfrq        100.1es   999
                           C13    dme
      at            4.855   8800
                           PROCESSING
      sw            1b      3.00
      25188.0       1b      3.00
      to            13880   1file
      bs            1       ft
      tpwr        58      fn    not used
      ls            1
      di            2.000   werr
      tof          2800.0   wexp
      1111.0       1obs
      ct            204     wft
      alock        n
      galm        not used
      FLAGS
      in            n
      dp            y
      DISPLAY
      sp            -9.2
      wp          2813.8
      sc            0
      scs           255
      hzms        .4500
      ts            500.00
      rfp          897.00
      th            773.2
      100.000      2
      100.000      2
      nr no ph

```



¹H and ¹³C NMR Spectra of compound **7a**:



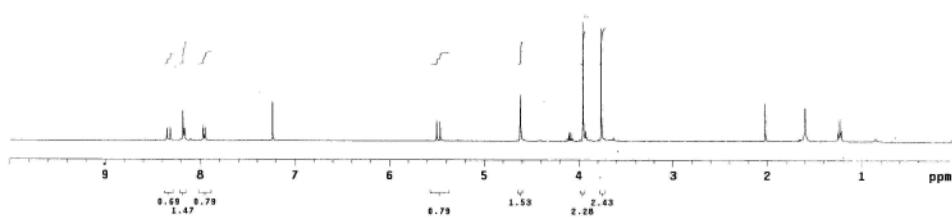
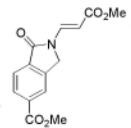
¹H and ¹³C NMR Spectra of compound 7b:

```

prsd      .8-351.0
exp? sthd  .8-181.0
          7.975-7.195

          SAMPLE           DEC. & VT
          Jan 28 2005      dfrq. 454
solvent    C6C13      dn   HI
          exp      dprw   42
          acq      nnn
          tfrq. 405.455  nnn
          at      nnn
          t       2.664  def     8000
          np      32216  PROCESSING
          n       8951  ib      8.10
          f       3406  profc   42
          s       1000  profg   42
          tpvr   5.0      not used
          pw      6.0
          l       1200  werr
          tof      508.0  wexp
          t       169   wbs
          ct      100   wft
          aclock  gain   not used
          gain
          FLAGS
          t1      n
          t2      n
          dp      y
          DISPLAY
          sp      -0.1
          ap      4004.2
          sc      0
          sc      250
          tscm   15.177
          tscm   33.774
          rfp      2099.3
          th      3
          nm cde ph  10.000

```



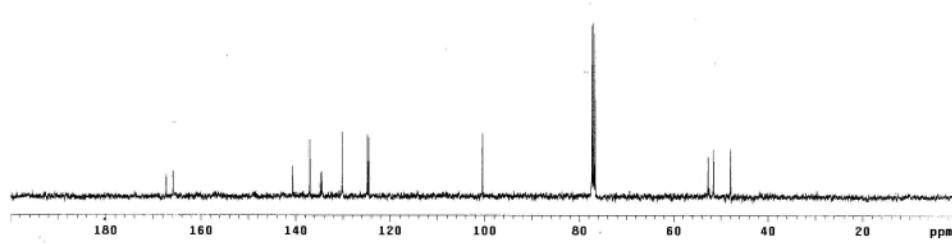
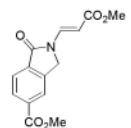
```

13C OBSERVE          -167.266
                     -165.866

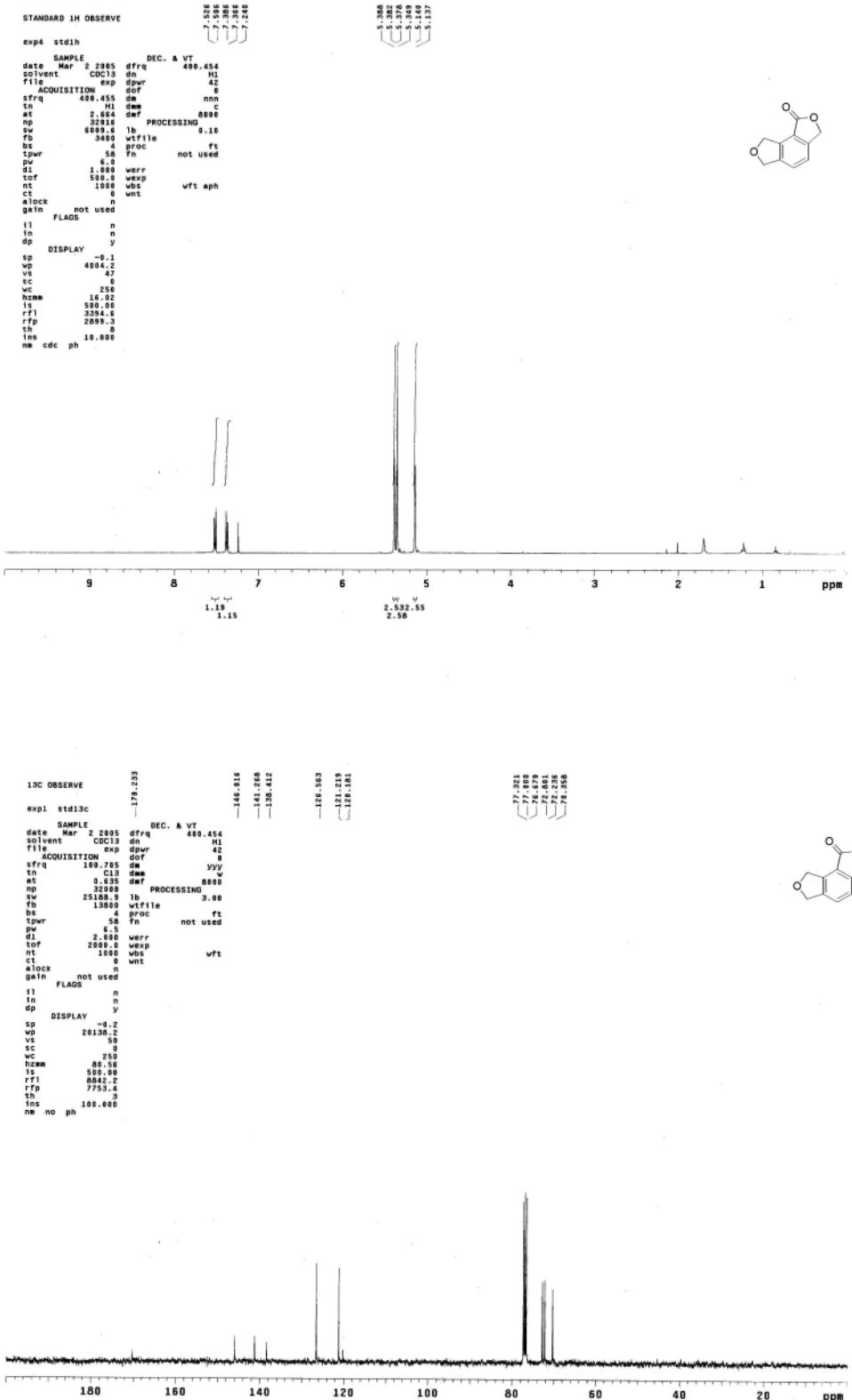
exp6 std13c

SAMPLE           DEC. & VT
date Jan 28 2005 dfrq 408.454
time 10:00:00      dpwr 42
Title exp        dof 0
ACQUISITION      yea 97
    nra 120.765
    tn 0.635       C13 dms 8600
    sw 32768.0      PROCESSING
    25168.0         3.00
    18000.0
    16000.0
    14000.0
    12000.0
    10000.0
    8000.0
    6000.0
    4000.0
    2000.0
    0.000
    tof 2000.0      werr
    1800.0         wexp
    1600.0         wft
    1400.0
    1200.0
    1000.0
    800.0
    600.0
    400.0
    200.0
    0.000
    gain  not used
    Flags
    l1  n
    l2  n
    dp  y
    DISPLAY
    sp -0.2
    wp 28138.2
    sc 0
    sc 0
    sc 0
    sc 250
    sc 500
    sc 500.00
    rft 8846.4
    rp 7753.4
    th 4
    th 100.000
    th 0.000
    th 0.000

```



¹H and ¹³C NMR Spectra of compound 9:



¹H NMR Spectrum of compound **13**:

