Electronic Supplementary Material (ESI) for Chemical Communications

Supporting Information

Efficient synthesis of anthranilic esters via Pd-catalyzed dehydrogenative/decarbonylative coupling of anilides and glyoxylates

Sizhuo Wang, Zhiyong Yang, Jidan Liu, Kai Xie, Anwei Wang, Xiang chen, Ze Tan,*

State Key Laboratory of Chemo/Biosensing and Chemometrics, College of Chemistry and Chemical Engineering, Hunan University, Changsha 410082, P.R. China. Tel: +86-731 88822400;

E-mail: ztanze@gmail.com

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I. General Information

All the solvents and commercially available reagents were purchased from commercial sources and used directly. ^{1}H NMR and ^{13}C NMR were recorded in $CDCl_3$ at room temperature on the Varian INOVA-400 spectrometer (400 MHz ^{1}H) . The chemical-shifts scale is based on internal TMS. The coupling constants, *J*, are reported in Hertz (HZ). Products were purified by flash column chromatography on 200-300 mesh silica gel, SiO_2.

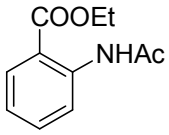
II. Representative Procedure and Selected Compounds Data

2-Acetylamino-benzoic acid ethyl ester

A 25 mL tube was charged with acetanilide (67.5mg, 0.5mmol), glyoxylate (204mg, 1.0mmol), TBHP (0.36mL, 2.0mmol), dppp (20.7mg, 0.05mmol), $Pd(TFA)_2$ (13.9mg, 0.05 mmol) and toluene (1mL). The reaction tube was then capped under N_2 and stirred at 120^{o}C for 16-24h. Upon completion of the reaction, the reaction tube was allowed to cool room temperature and quenched with brine and diluted with DMC. The organic layer was separated and the aqueous layer was washed with DMC. The filtrate was concentrated under reduced pressure. The crude products were purified with flash column chromatography on silica gel (petroleum ether/ethyl acetate, 8/1) to afford 56.9 mg of the desired 2-Acetylamino –benzoic acid ethyl ester in 55% yield. Better yield (66%) can be obtained if the reaction was stopped at 12h, and another batch of $Pd(TFA)_2$ (5mol%) was added.

Analytical data of Acetylamino-benzoic acid ethyl ester

2-Acetylamino-benzoic acid ethyl ester [1-4]



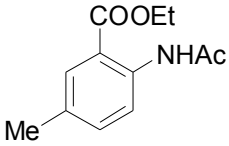
TLC: R_f= 0.60 (petroleum ether/ ethyl acetate, 4/1); Yield 66%;

^{1}H NMR (400 MHz, $CDCl_3$): δ 11.14 (s, 1H), 8.70 (d, *J* = 8.4Hz, 1H), 8.04-8.06 (m, 1H), 7.52-7.56 (m, 1H), 7.07-7.10 (m, 1H), 4.38 (q, *J* = 7.2Hz, 2H), 2.25 (s, 3H), 1.42 (t, *J* = 7.2Hz, 3H);

^{13}C NMR (100MHz, $CDCl_3$): δ 169.22, 168.29, 141.46, 134.54, 130.73, 122.38, 120.23, 114.98, 61.38, 25.48, 14.15;

HRMS: calcd for $C_{11}H_{13}NO_3$: 207.0895 , found: 207.0893;

2-Acetylamino-5-methyl-benzoic acid ethyl ester



TLC: R_f= 0.6 (petroleum ether/ethyl acetate, 4/1); Yield 60%;

^{1}H NMR (400 MHz, $CDCl_3$): δ 11.00 (s, 1H), 8.58 (d, *J* = 8.8Hz, 1H), 7.83 (s, 1H), 7.35 (dd, *J* = 8.8Hz, 2Hz, 1H), 4.37 (dd, *J* = 14.4Hz, 6.4Hz, 2H), 2.33 (s, 3H) , 2.23 (s, 3H), 1.43 (t, *J* = 7.2Hz, 3H);

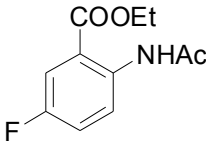
^{13}C NMR (100MHz, $CDCl_3$): δ 168.89, 168.32, 139.12, 135.22, 131.84, 130.74, 120.19, 114.85, 61.28, 25.44, 20.65, 14.17;

LRMS: m/z calcd for $C_{12}H_{15}NO_3$: 221, found: 221;

Anal. Calcd for $C_{12}H_{15}NO_3$: C, 65.14; H, 6.83; N, 6.33; Found: C, 65.37; H, 6.96; N, 6.58;

IR.(KBr)/cm^{-1}: 3268, 2925, 1694, 1239;

2-Acetylamino-5-fluoro-benzoic acid ethyl ester



TLC: R_f= 0.45 (petroleum ether/ ethyl acetate, 4/1); Yield 40%;

^{1}H NMR (400 MHz, $CDCl_3$): δ 10.97 (s, 1H), 8.71 (dd, *J* = 9.6 Hz, 5.2 Hz, 1H), 7.72 (dd, *J* = 9.2 Hz, 3.2 Hz, 1H), 7.29-7.24 (m, 1H), 4.39 (dd, *J* = 14.4 Hz, 7.6 Hz, 2H), 2.24 (s, 3H), 1.43 (t, *J* = 7.2Hz, 3H);

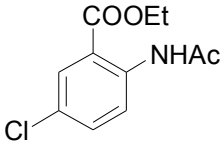
^{13}C NMR (100MHz, $CDCl_3$): δ 168.93, 167.23, 157.15 (d, $J_{C\text{-}F}$ = 241 Hz), 137.92, 122.07 (d, $J_{C\text{-}F}$ = 7.6 Hz), 121.53 (d, $J_{C\text{-}F}$ = 21.3 Hz), 116.72 (d, $J_{C\text{-}F}$ = 24.4

Hz), 116.24 (d, J_{C-F} = 6.8 Hz), 61.80, 25.38, 14.10;

HRMS: m/z calcd for $C_{11}H_{12}FNO_3$: 225.0801 , found: 225.0804;

IR.(KBr)/cm^{-1}: 3281, 2987, 1701, 1236;

2-Acetylamino-5-chloro-benzoic acid ethyl ester



TLC: R_f=0.45 (petroleum ether/ ethyl acetate, 4/1); Yield 41%;

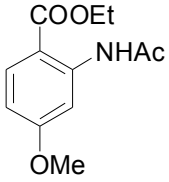
^{1}H NMR (400 MHz, CDCl$_3$): δ 11.05(s, 1H), 8.69 (d, *J* = 9.2Hz, 1H), 7.99 (s, 1H), 7.48 (dd, *J* = 9.2Hz, 2.8Hz, 1H), 4.18-4.36(m, 2H), 2.24 (s, 3H), 1.43 (t, *J* = 6.8Hz, 3H);

^{13}C NMR (100MHz, CDCl$_3$): δ 169.09, 167.22, 140.08, 134.32, 130.27, 127.33, 121.64, 116.18, 61.85, 25.46, 14.12.

HRMS: m/z calcd for $C_{11}H_{12}ClNO_3$: 241.0506 , found: 241.0510;

IR.(KBr)/cm^{-1}: 3265, 2961, 1703, 1235;

2-Acetylamino-4-methoxy-benzoic acid ethyl ester



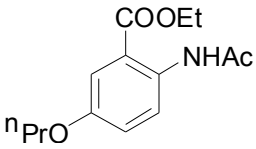
TLC: R_f= 0.56 (petroleum ether/ ethyl acetate, 4/1); Yield 51%;

^{1}H NMR (400 MHz, CDCl$_3$): δ 11.34(s, 1H), 8.38(d, *J* = 2.4Hz, 1H), 7.97 (d, *J* = 8.8Hz, 1H), 6.60 (dd, *J* = 12Hz, 2.8Hz, 1H), 4.37-4.32 (m, 2H), 3.87 (s, 3H), 2.24 (s, 3H), 1.40 (t, *J* = 7.2Hz, 3H);

^{13}C NMR (100MHz, CDCl$_3$): δ 169.33, 168.19, 164.35, 143.73, 132.31, 109.62, 107.41, 103.72, 60.94, 55.49, 25.61, 14.21;

HRMS: m/z calcd for $C_{12}H_{15}NO_4$: 237.1001 , found: 237.1008;

2-Acetylamino-5-propoxy-benzoic acid ethyl ester



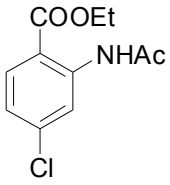
TLC: R_f= 0.58 (petroleum ether/ ethyl acetate, 4/1); Yield 58%;

^{1}H NMR (400 MHz, CDCl$_3$): δ 10.84(s, 1H), 8.60 (d, *J* = 9.2Hz, 1H), 7.53 (s, 1H), 7.11 (dd, *J* = 9.2Hz, 3.2Hz, 1H), 4.40-4.35 (m, 2H), 3.92 (t, *J* = 6.8Hz, 2H), 2.22 (s, 3H), 1.84-1.79 (m, 2H), 1.42(t, *J* = 7.2Hz, 3H), 1.05 (t, *J* = 7.6Hz, 3H);

^{13}C NMR (100MHz, CDCl$_3$): δ 168.64, 167.94, 153.81, 134.96, 121.72, 120.85, 116.08, 115.59, 69.82, 61.41, 25.32, 22.48, 14.15, 10.46;

HRMS: m/z calcd for $C_{14}H_{19}NO_4$: 265.1314 , found: 265.1318;

2-Acetylamino-4-chloro-benzoic acid ethyl ester



TLC: R_f= 0.45 (petroleum ether/ ethyl acetate, 4/1); Yield 49%;

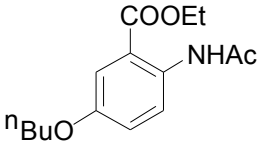
^{1}H NMR (400 MHz, $CDCl_3$): δ 11.19 (s, 1H), 8.80 (d, *J* = 2Hz, 1H), 7.97 (d, *J* = 8.8Hz, 1H), 7.05 (dd, *J* = 8.4Hz, 2.4Hz, 1H), 4.38 (dd, *J* = 14.4Hz, 7.6Hz, 2H), 2.25 (s, 3H), 1.42 (t, *J* = 6.8Hz, 3H);

^{13}C NMR (100MHz, $CDCl_3$): δ 169.29, 167.71, 142.30, 140.81, 131.80, 128.79, 122.64, 120.80, 113.13, 16.64, 25.47, 14.12;

LRMS: m/z calcd for $C_{11}H_{12}ClNO_3$: 241, found: 241;

Anal. Calcd for $C_{11}H_{12}ClNO_3$: C, 54.67; H, 5.00; N, 5.80; Found: C, 54.60; H, 4.95; N, 5.74;.

2-Acetylamino-5-butoxy-benzoic acid ethyl ester



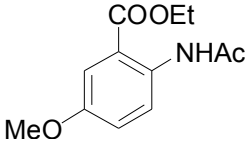
TLC: R_f= 0.60 (petroleum ether/ ethyl acetate, 4/1); Yield 54%;

^{1}H NMR (400 MHz, $CDCl_3$): δ 10.83 (s, 1H), 8.60(d, *J* = 9.2Hz, 1H), 7.52 (s, 1H), 7.11 (d, *J* = 9.2Hz, 1H), 4.04 (t , *J* = 7.6Hz, 2H), 3.96 (t, *J* = 6.8Hz, 2H), 2.22 (s, 3H), 1.79-1.75 (m, 2H), 1.53-1.47 (m, 2H), 1.44-1.40 (m, 3H), 0.98(t, *J* = 14.8Hz, 3H);

^{13}C NMR (100MHz, $CDCl_3$): δ 168.64, 167.96, 153.84, 134.98, 121.74, 120.85, 116.10, 115.60, 68.02, 61.43, 31.20, 25.34, 19.17, 14.17, 13.83;

HRMS: m/z calcd for $C_{15}H_{21}NO_4$: 279.1471 , found: 279.1474;

2-Acetylamino-5-methoxy-benzoic acid ethyl ester



TLC: R_f= 0.55 (petroleum ether/ ethyl acetate, 4/1); Yield 63%;

^{1}H NMR (400 MHz, $CDCl_3$): δ 10.82 (s, 1H), 8.61(d, *J* = 9.2Hz, 1H), 7.52 (s, 1H), 7.11 (dd, *J* = 9.6Hz, 3.6Hz, 1H), 4.38 (dd, *J* = 14.8Hz, 7.6Hz, 2H), 3.82 (s, 3H), 2.21 (s, 3H), 1.41 (t, *J* = 6.8Hz, 3H);

^{13}C NMR (100MHz, $CDCl_3$): δ 168.66, 167.90, 154.23, 135.19, 121.81, 120.47, 116.09, 114.75, 61.47, 55.56, 25.35, 14.16;

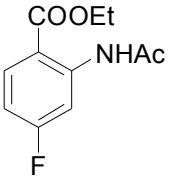
LRMS: m/z calcd for $C_{12}H_{15}NO_4$: 237.1001 , found: 237.0944;

Anal. Calcd for $C_{12}H_{15}NO_4$: C, 60.75; H,6.37; N, 5.90; Found: C, 60.97; H,6.10;

N, 5.63;

IR.(KBr)/cm^{-1}: 3267, 2982, 1680, 1240;

2-Acetylamino-4-fluoro-benzoic acid ethyl ester



TLC: R_f= 0.45 (petroleum ether/ ethyl acetate, 4/1); Yield 41%;

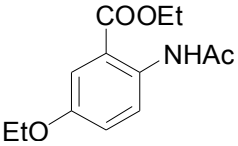
^{1}H NMR (400 MHz, CDCl$_3$): δ 11.29 (s, 1H), 8.53 (dd, *J* = 12.4Hz, 2.8Hz, 1H), 8.06 (dd, *J* = 7.2Hz, 1H), 6.79-6.74 (m, 1H), 4.38 (dd, *J* = 14.8Hz, 7.2Hz, 2H), 2.25 (s, 3H), 1.42 (t, *J* = 7.2Hz, 3H);

^{13}C NMR (100MHz, CDCl$_3$): δ 169.36, 167.65, 166.19 (d, $J_{C\text{-}F}$ = 251 Hz), 143.65 (d, $J_{C\text{-}F}$ = 13 Hz), 132.54 (d, $J_{C\text{-}F}$ = 10.7 Hz), 111.10, 109.69 (d, $J_{C\text{-}F}$ = 22.9 Hz), 107.32 (d, $J_{C\text{-}F}$ = 27.5 Hz), 61.48, 25.48, 14.17;

LRMS: m/z calcd for $C_{11}H_{12}FNO_3$: 225 , found: 225;

Anal. Calcd for $C_{11}H_{12}FNO_3$: C, 58.66; H, 5.37; N, 6.22; Found: C, 58.89; H, 5.18; N, 6.02.

2-Acetylamino-5-ethoxy-benzoic acid ethyl ester



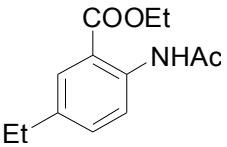
TLC: R_f= 0.57 (petroleum ether/ ethyl acetate, 4/1); Yield 62%;

^{1}H NMR (400 MHz, CDCl$_3$): δ 10.84(s, 1H), 8.61 (d, *J* = 9.2Hz, 1H), 7.53(s, 1H), 7.10(dd, *J* = 9.2Hz, 2.8Hz, 1H), 4.37 (dd, *J* = 14Hz, 6.8Hz, 2H), 4.04 (dd, *J* = 14Hz,7.2Hz, 2H), 2.22 (s, 3H), 1.44-1.40 (m, 6H);

^{13}C NMR (100MHz, CDCl$_3$): δ 167.71, 166.95, 152.64, 134.04, 120.77, 119.86, 115.13, 114.62, 62.84, 60.45, 24.34, 13.75, 13.17;

HRMS: m/z calcd for $C_{13}H_{17}NO_4$: 251.1158 , found: 251.1156;

2-Acetylamino-5-ethyl-benzoic acid ethyl ester



TLC: R_f=0.58 (petroleum ether/ ethyl acetate, 4/1); Yield 61%;

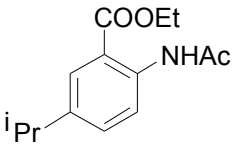
^{1}H NMR (400 MHz, CDCl$_3$): δ 11.00 (s, 1H), 8.59 (d, *J* = 8.8Hz, 1H), 7.85 (s, 1H), 7.38 (dd, *J* = 8.4Hz, 2Hz, 1H), 4.40 (dd, *J* = 14,4Hz, 7.2Hz, 2H), 2.63 (dd, *J* = 15.2Hz, 7.6Hz, 2H), 2.23 (s, 3H), 1.43 (t, *J* = 7.2Hz, 3H), 1.23 (t, *J* = 7.6Hz, 3H);

^{13}C NMR (100MHz, CDCl$_3$): δ 168.91, 168.37, 139.31, 138.28, 134.13, 129.66, 120.35, 114.95, 61.29, 28.10, 25.46, 15.59, 14.20;

LRMS: m/z calcd for $C_{13}H_{17}NO_3$: 235 , found: 235;

Anal. Calcd for $C_{13}H_{17}NO_3$: C, 66.36; H, 7.28; N, 5.95; Found: C, 66.17; H, 7.42; N, 5.55;

2-Acetylamino-5-isopropyl-benzoic acid ethyl ester



TLC: R_f= 0.56 (petroleum ether/ ethyl acetate, 4/1); Yield 62%;

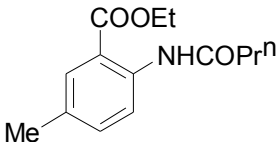
^{1}H NMR (400 MHz, $CDCl_3$): δ 11.00(s, 1H), 8.60(d, *J* = 8.8Hz, 1H), 7.86 (s, 1H), 7.42 (dd, *J* = 8.8Hz, 2.4Hz, 1H), 4.39 (dd, *J* = 14.8Hz, 5.6Hz, 2H), 2.94-2.87 (m, 1H), 2.23 (s, 3H), 1.43 (t, *J* = 6.8Hz, 3H), 1.25-1.24 (m, 6H);

^{13}C NMR (100MHz, $CDCl_3$): δ 168.91, 168.39, 142.88, 139.37, 132.65, 128.29, 120.41, 114.95, 61.30, 33.44, 25.44, 23.85, 14.21;

LRMS: m/z calcd for $C_{14}H_{19}NO_3$: 249 , found: 249;

Anal. Calcd for $C_{14}H_{19}NO_3$: C, 67.45; H, 7.68; N, 5.62; Found: C, 67.11; H, 7.74; N, 5.49;

2-Propionylamino-5-methyl-benzoic acid ethyl ester



TLC: R_f= 0.49 (petroleum ether/ ethyl acetate, 4/1); Yield 47%;

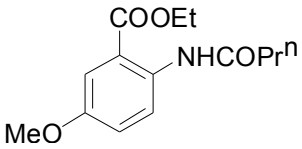
^{1}H NMR (400 MHz, $CDCl_3$): δ 11.02(s, 1H), 8.62 (d, *J* = 8.8Hz, 1H), 7.83(s, 1H), 7.35 (dd, *J* = 8.8Hz, 2Hz, 1H), 4.37 (dd, *J* = 14Hz, 6.8Hz, 2H), 2.47 (dd, *J* = 14.8Hz, 7.2Hz, 2H), 2.33 (s, 3H), 1.42 (t, *J* = 7.6Hz, 3H), 1.27 (t, *J* = 7.6Hz, 3H);

^{13}C NMR (100MHz, $CDCl_3$): δ 172.69, 168.35, 139.26, 135.25, 131.69, 130.76, 120.23, 114.83, 61.25, 31.61, 20.65, 14.19, 9.63;

HRMS: m/z calcd for $C_{13}H_{17}NO_3$: 235.1208 , found: 235.1210;

IR.(KBr)/cm^{-1}: 3262, 2938, 1689, 1235;

2-Propionylamino-5- methoxy-benzoic acid ethyl ester



TLC: R_f= 0.46 (petroleum ether/ ethyl acetate, 4/1); Yield 45%;

^{1}H NMR (400 MHz, $CDCl_3$): δ 10.87 (s, 1H), 8.66 (d, *J* = 9.6Hz, 1H), 7.53 (d, *J* = 2.8Hz, 1H), 7.12 (dd, *J* = 9.2Hz, 2.8Hz, 1H), 4.38 (dd, *J* = 14.4Hz, 7.2Hz, 2H), 3.82 (s, 3H), 2.46 (dd, *J* = 15.2Hz, 7.6Hz, 2H), 1.42 (t, *J* = 7.2Hz, 3H), 1.27 (t, *J* = 7.6Hz, 3H);

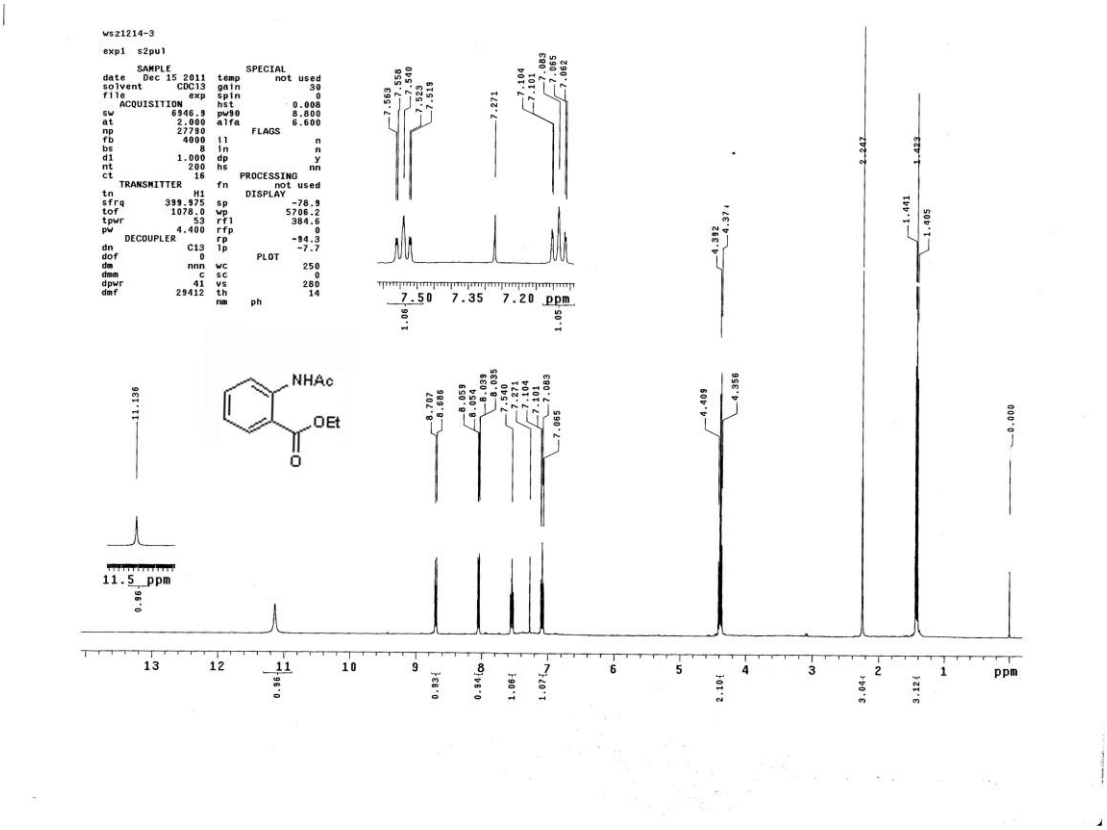
^{13}C NMR (100MHz, $CDCl_3$): δ 172.45, 167.91, 154.15, 135.32, 121.82, 120.48, 116.06, 114.78, 61.43, 55.56, 31.52, 14.16, 9.66;

HRMS: m/z calcd for $C_{13}H_{17}NO_4$: 251.1158 , found: 251.1160;

III.References and notes

1 H. Hiroshi, I. Naoto, *Tetrahed. Lett*. 1979, **26**, 2403.
2 K. Clausen, S. O.Lawesson, *Bull. Soc. Chim. Belg.* 1979, **88**, 305.
3 L. K. Sharma, , S. Kumar, P. Yadav, R. K. P. Singh, *Indian J. Chem. B* 2008, **47B**, 1277.
4 R. Shang, Y. Fu, J. B. Li, S. L. Zhang, Q.X. Guo, L. Liu. *J. Am. Chem. Soc*. 2009, **131**, 5738.

IV. ^{1}H and ^{13}C NMR Spectra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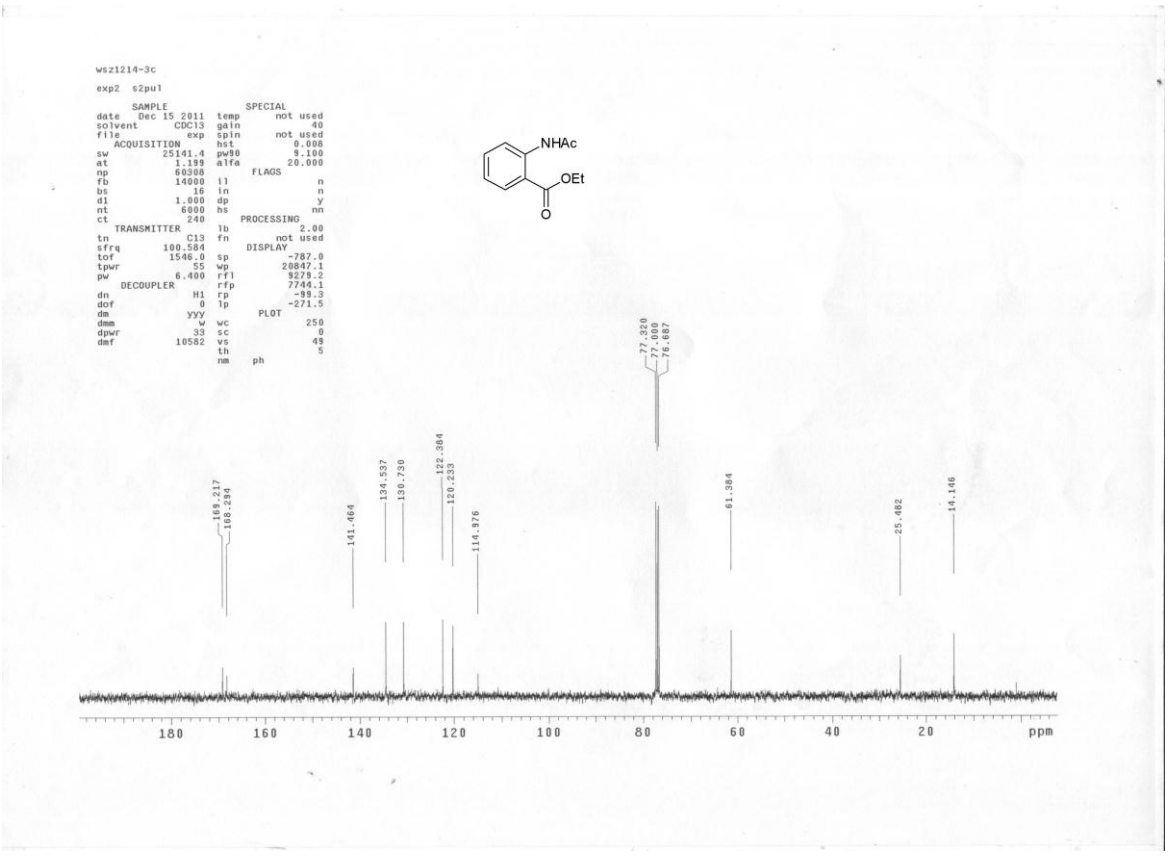
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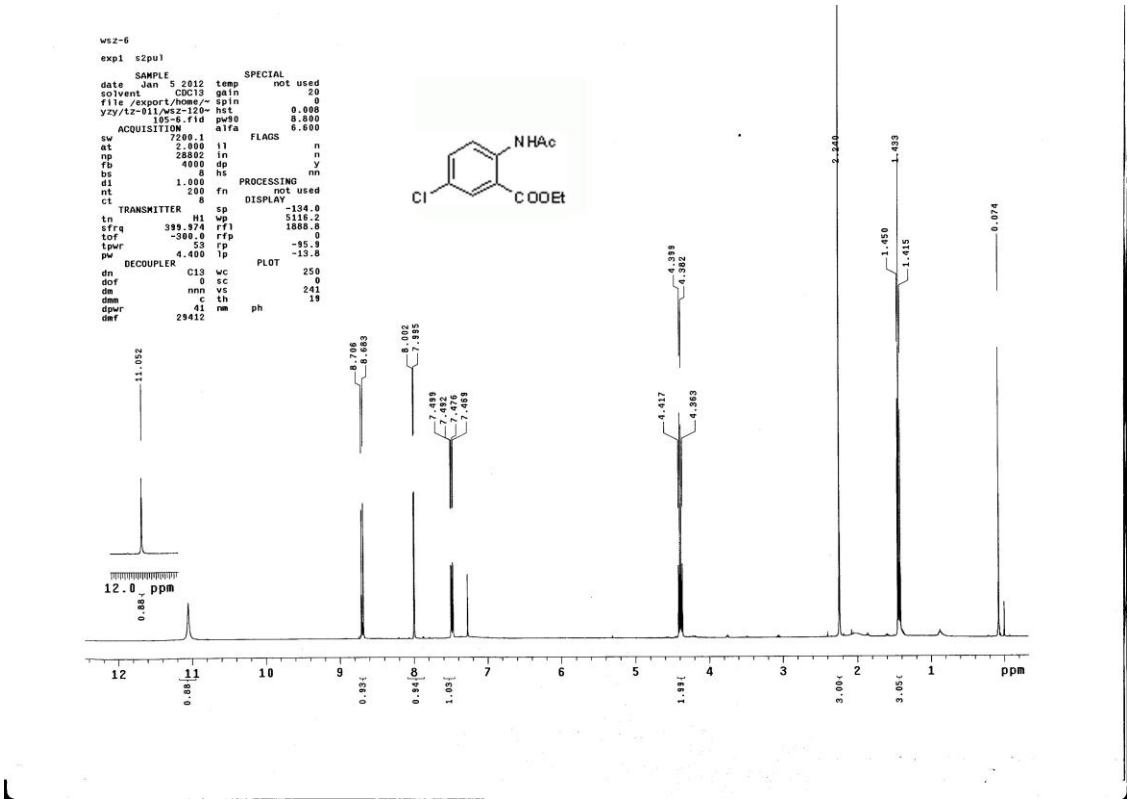
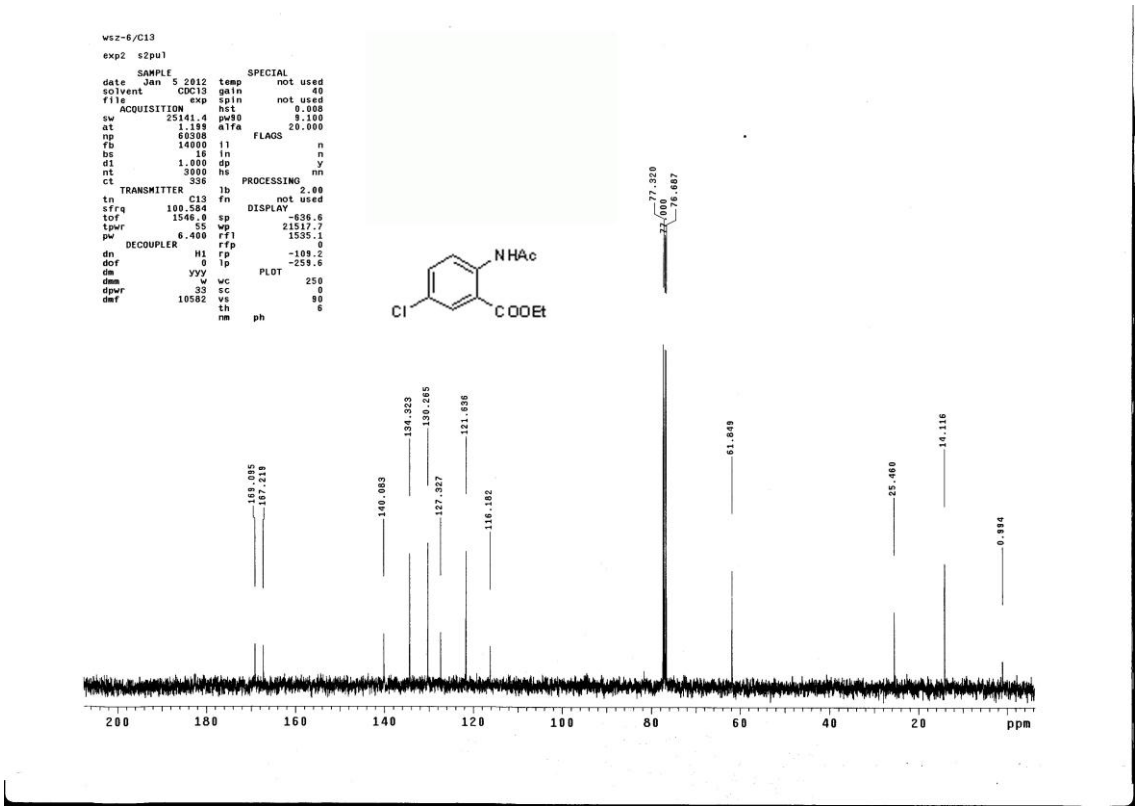
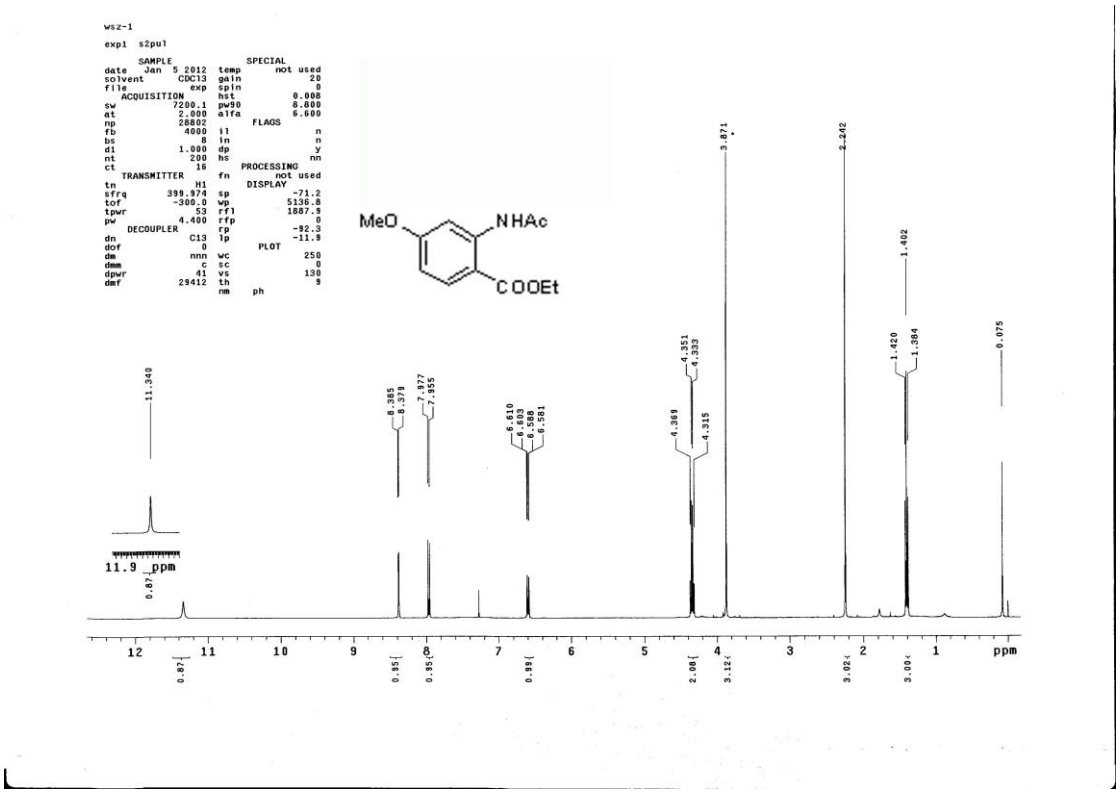
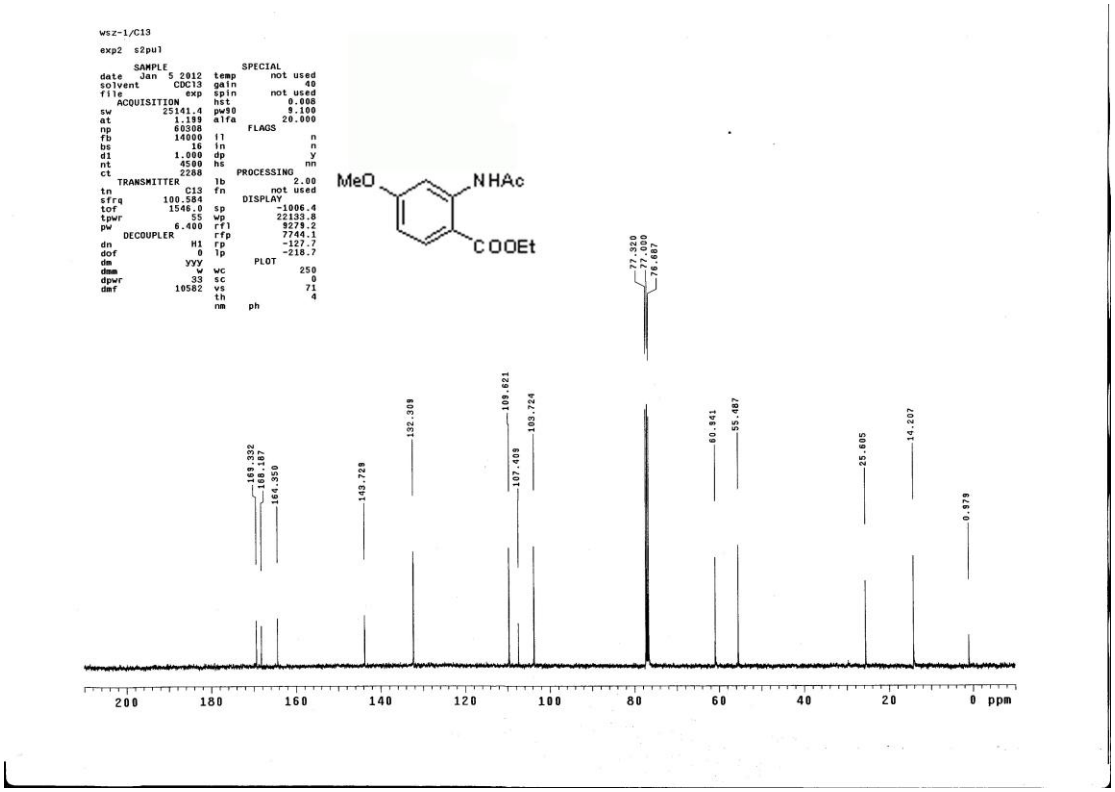
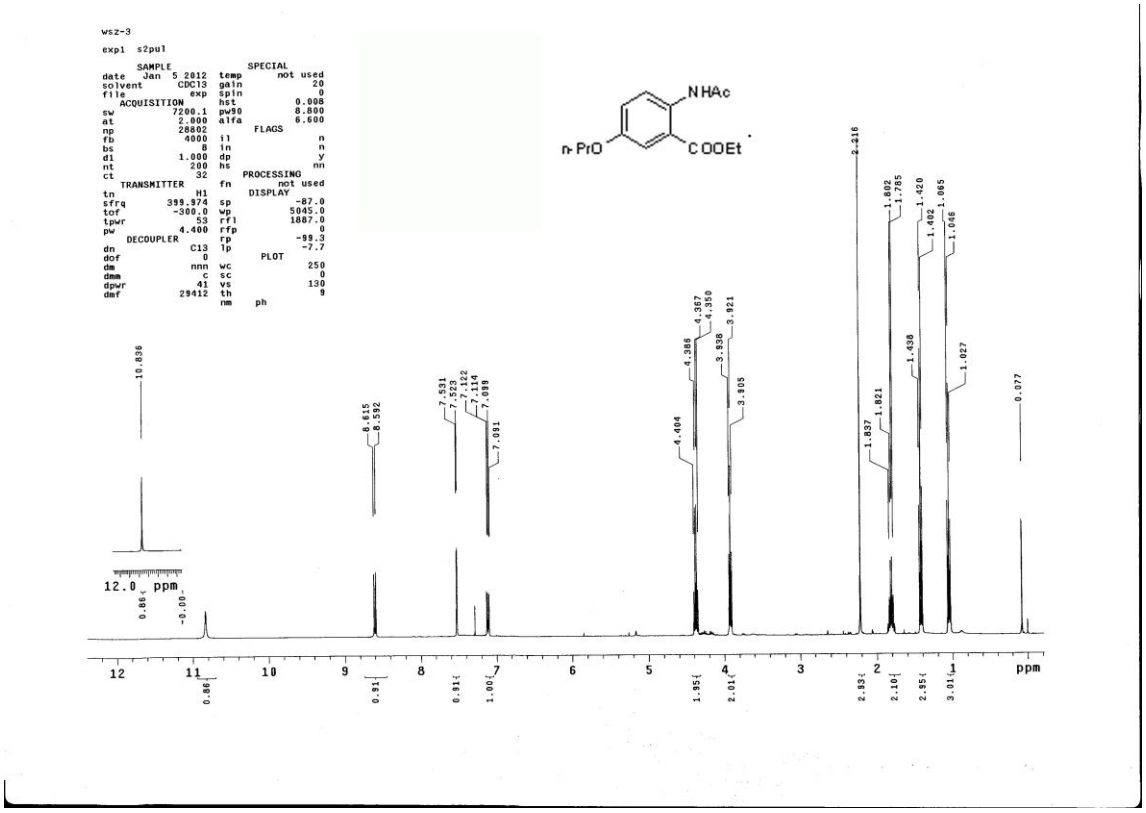
OEt
ppm

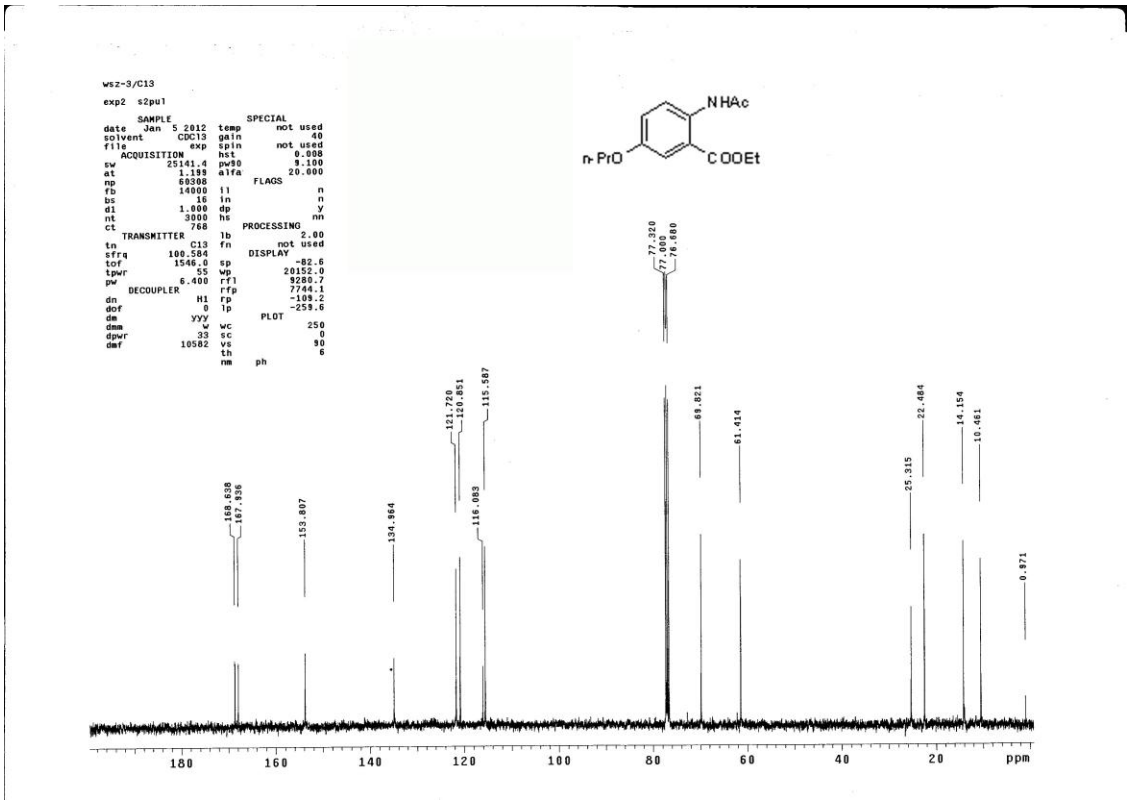

wsz-6
exp1 s2pul
NHAc
Cl
COOEt
11.052
8.706
8.683
8.002
7.995
7.499
7.492
7.476
7.469
4.399
4.382
4.417
4.363
2.240
1.459
1.433
1.415
0.074
12.0 ppm
ppm

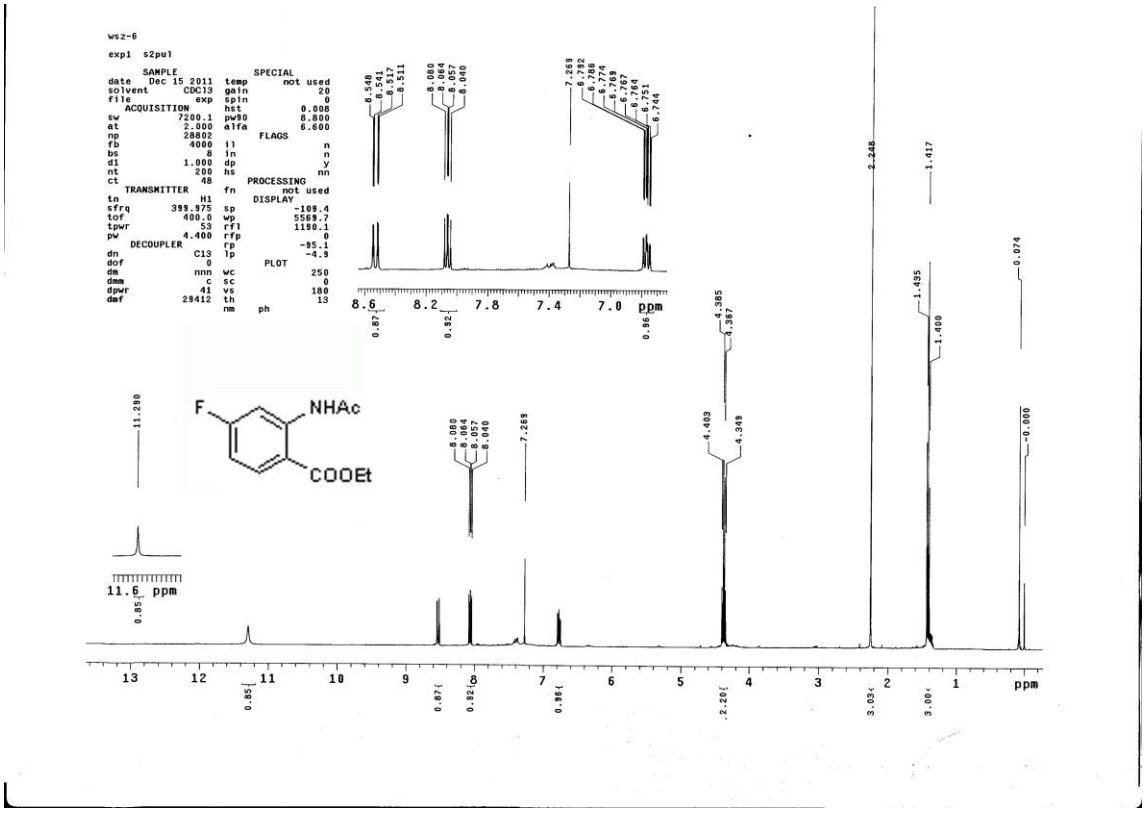

wsz-6/C13
exp2 s2pul
NHAc
Cl
COOEt
77.320
77.000
76.687
169.095
167.218
140.083
134.323
130.265
127.327
121.936
116.182
61.849
25.460
14.116
0.994
ppm

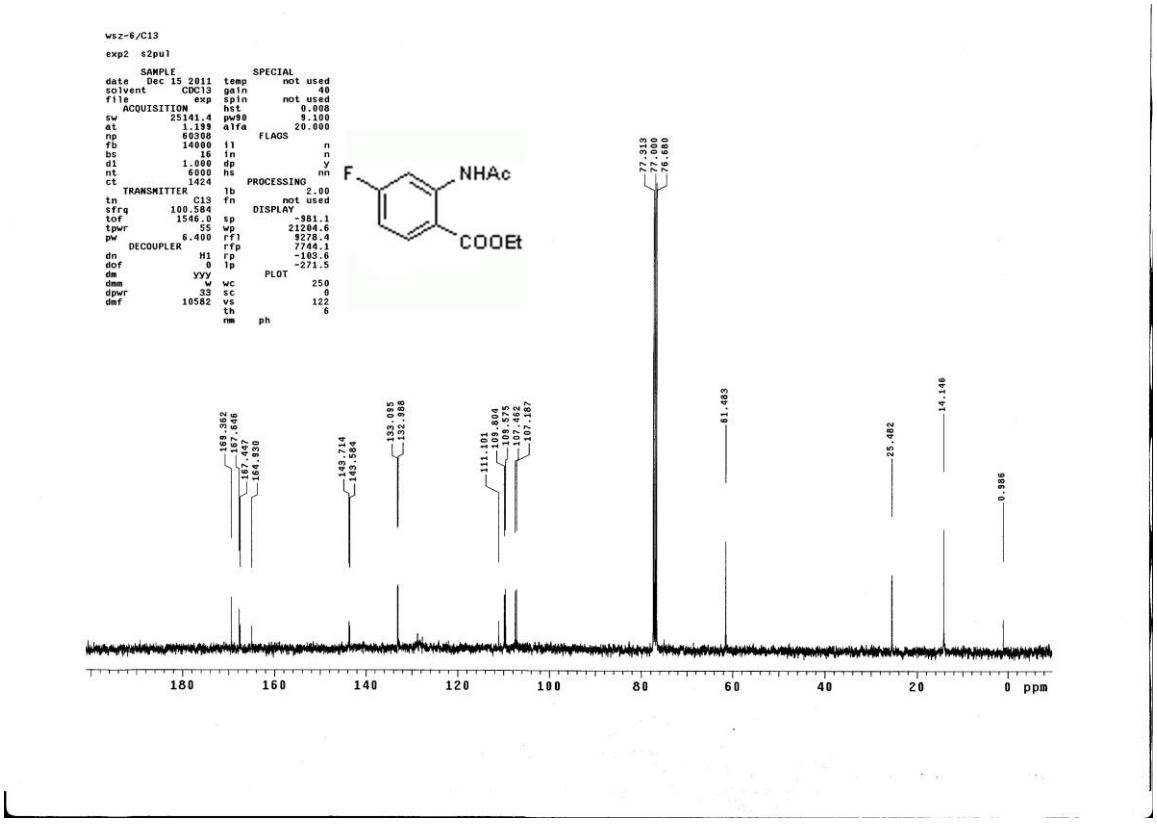

wsz-1
exp1 s2pul
MeO
NHAc
COOEt
11.340
8.385
8.379
7.977
7.955
6.610
6.603
6.588
6.581
4.369
4.351
4.333
4.315
3.871
2.242
1.402
1.420
1.384
0.075
11.9 ppm
ppm

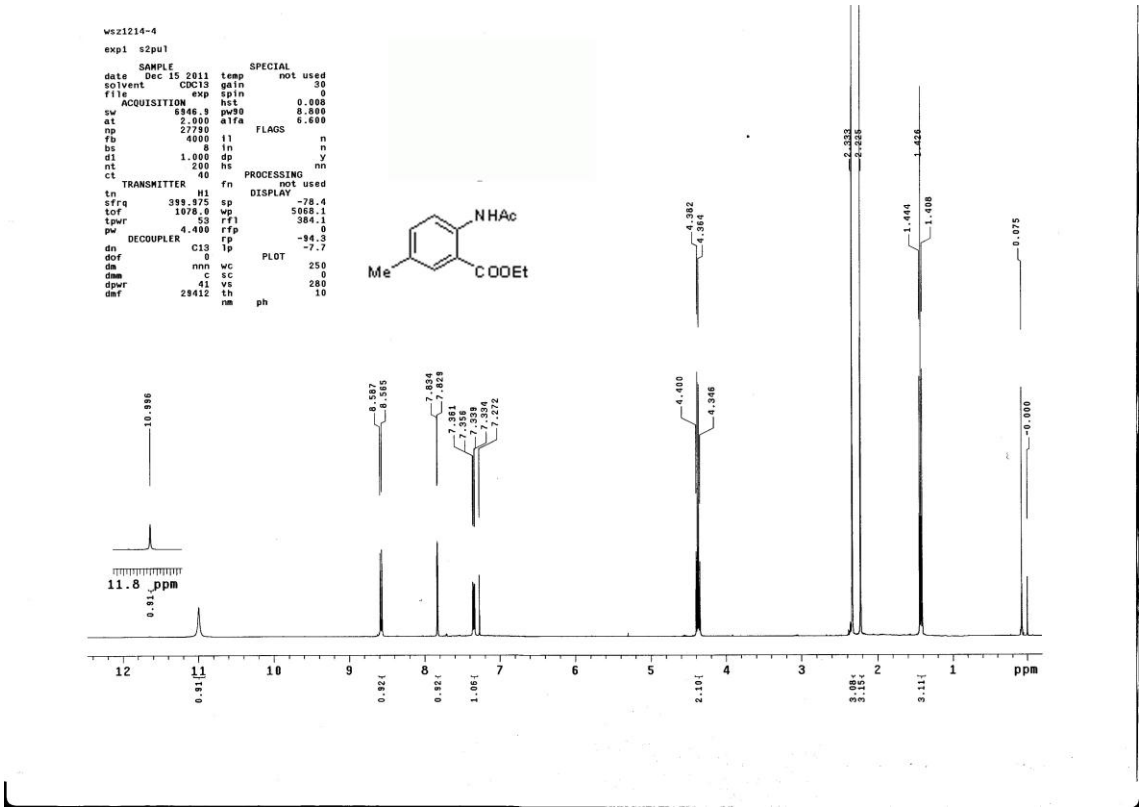

wsz-1/C13
exp2 s2pul
MeO
NHAc
COOEt
169.332
168.107
164.350
143.728
132.308
109.621
107.408
103.724
77.320
77.000
76.687
60.841
55.487
25.605
14.207
0.979
ppm

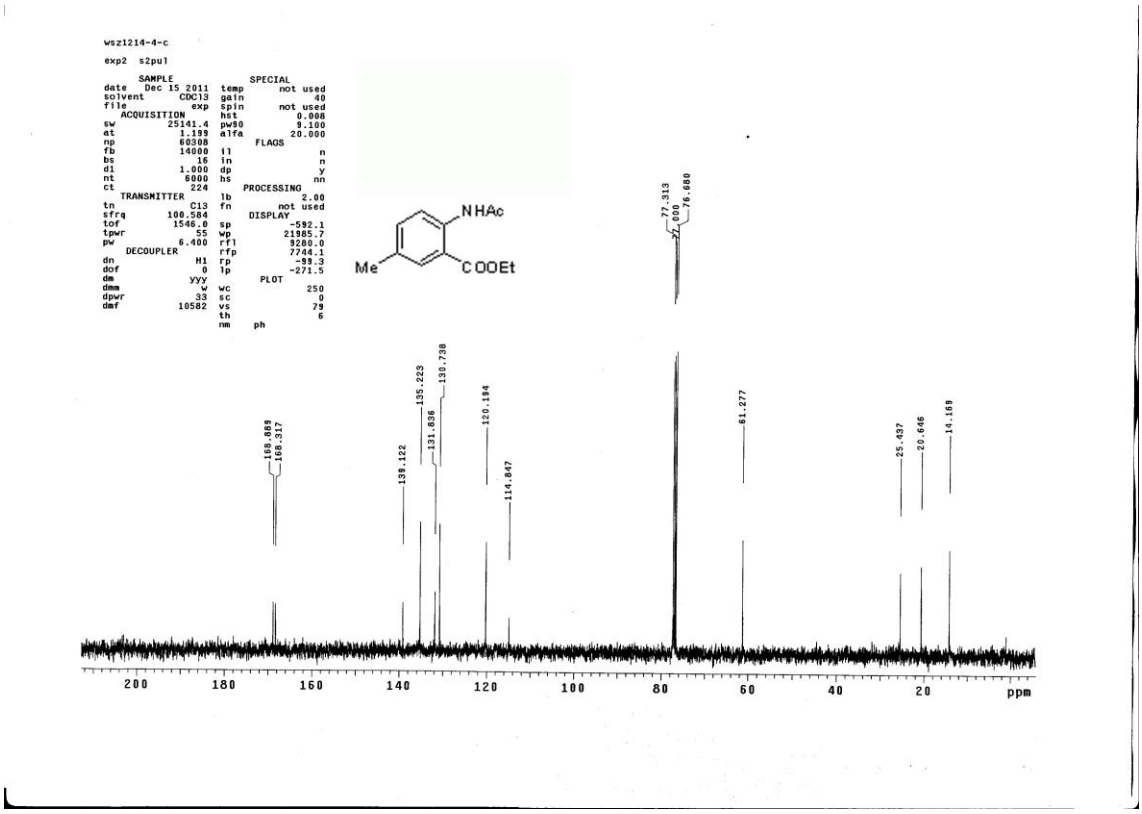

wsz-3
exp1 s2pul
SAMPLE
date Jan 5 2012
solvent CDCl3
file exp
ACQUISITION
sw 7200.1
at 2.800
np 28802
fb 4000
bs 8
dl 1.000
nt 200
ct 32
TRANSMITTER
tn H1
sfrq 399.974
tof -300.0
tpwr 53
pw 4.400
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 41
dmf 29412
SPECIAL
temp not used
gain 20
spin 0
hst 0.008
pw90 8.800
alfa 6.600
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -87.0
wp 5045.0
rfl 1887.0
rfp 0
rp -99.3
lp -7.7
PLOT
wc 250
sc 0
vs 130
th 9
nm ph
NHAc
n-PrO
COOEt
12.0 ppm
ppm

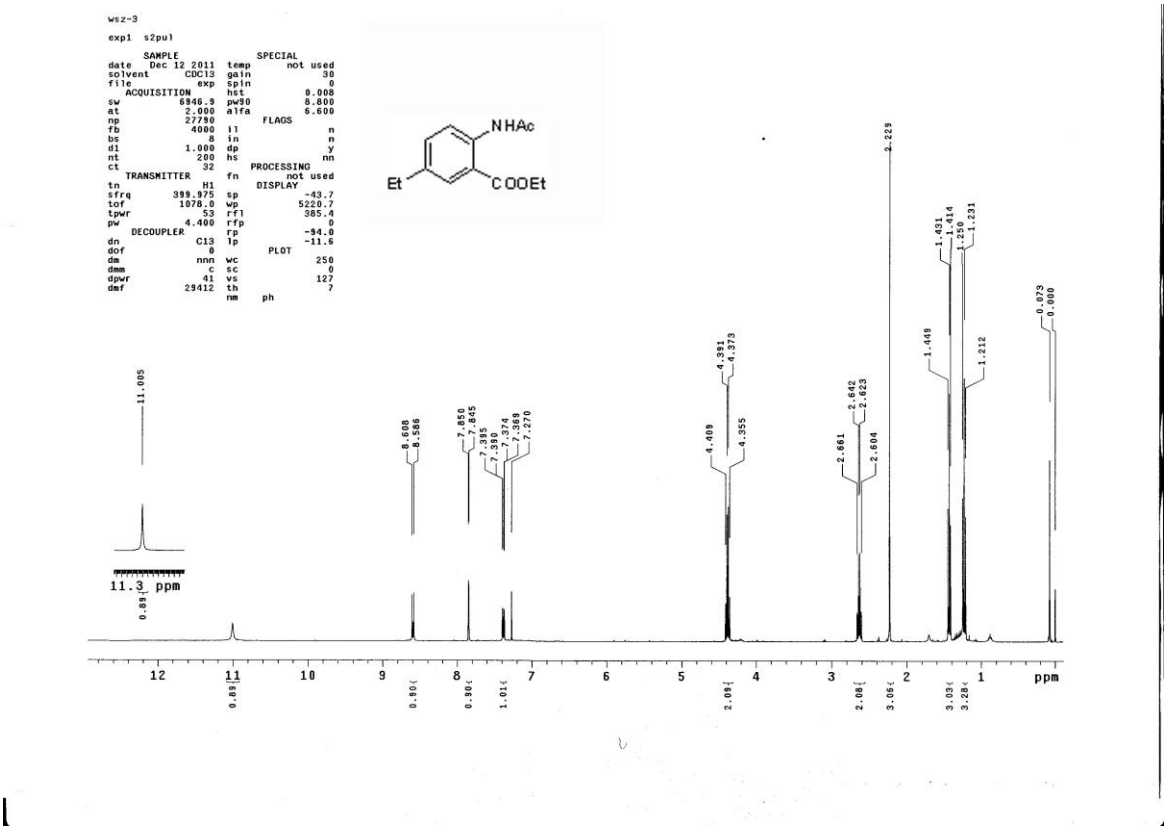

wsz-3/C13
exp2 s2pul
SAMPLE
date Jan 5 2012
solvent CDCl3
file exp
ACQUISITION
sw 25141.4
at 1.199
np 60308
fb 14000
bs 16
dl 1.000
nt 3000
ct 768
TRANSMITTER
tn C13
sfrq 100.584
tof 1546.0
tpwr 55
pw 6.400
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 33
dmf 10582
SPECIAL
temp not used
gain 40
spin not used
hst 0.008
pw90 9.100
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 2.00
fn not used
DISPLAY
sp -82.6
wp 20152.0
rfl 9280.7
rfp 7744.1
rp -109.2
lp -259.6
PLOT
wc 250
sc 0
vs 90
th 6
nm ph
NHAc
n-PrO
COOEt
ppm

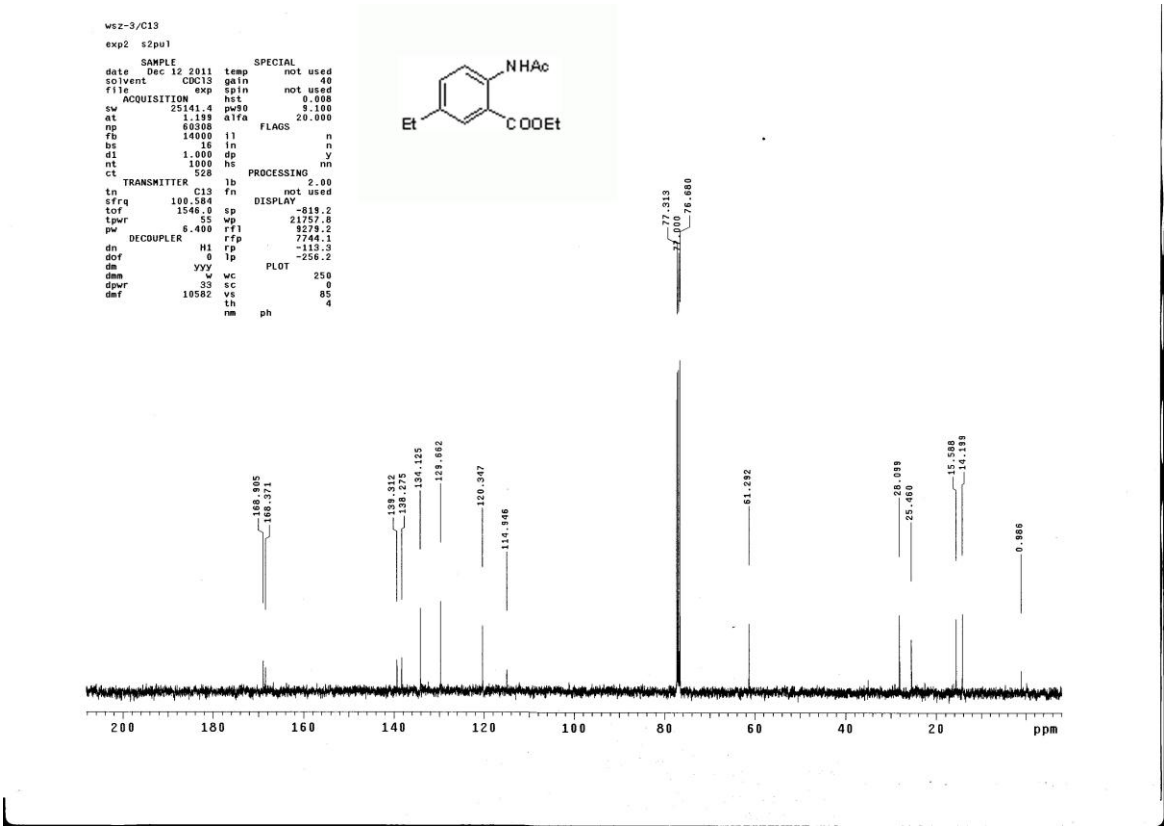

F
NHAc
COOEt
11.6 ppm
ppm

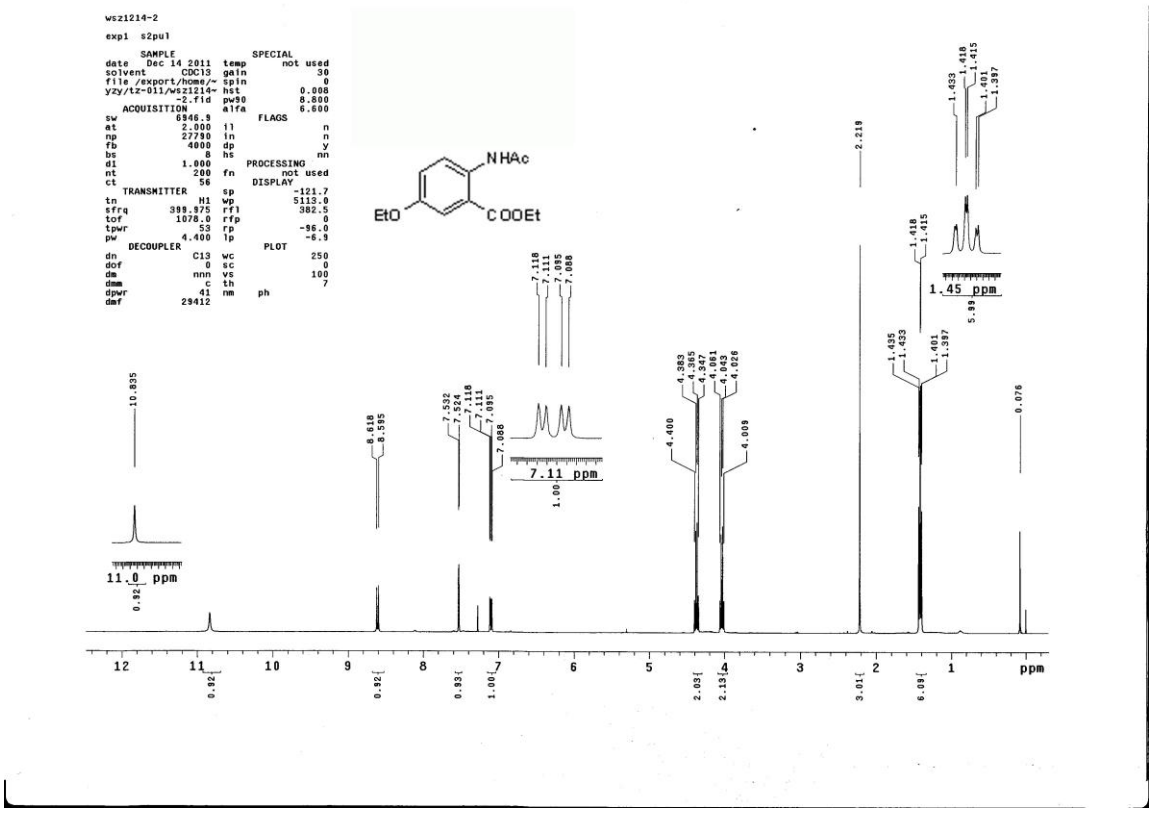

F
NHAc
COOEt
ppm

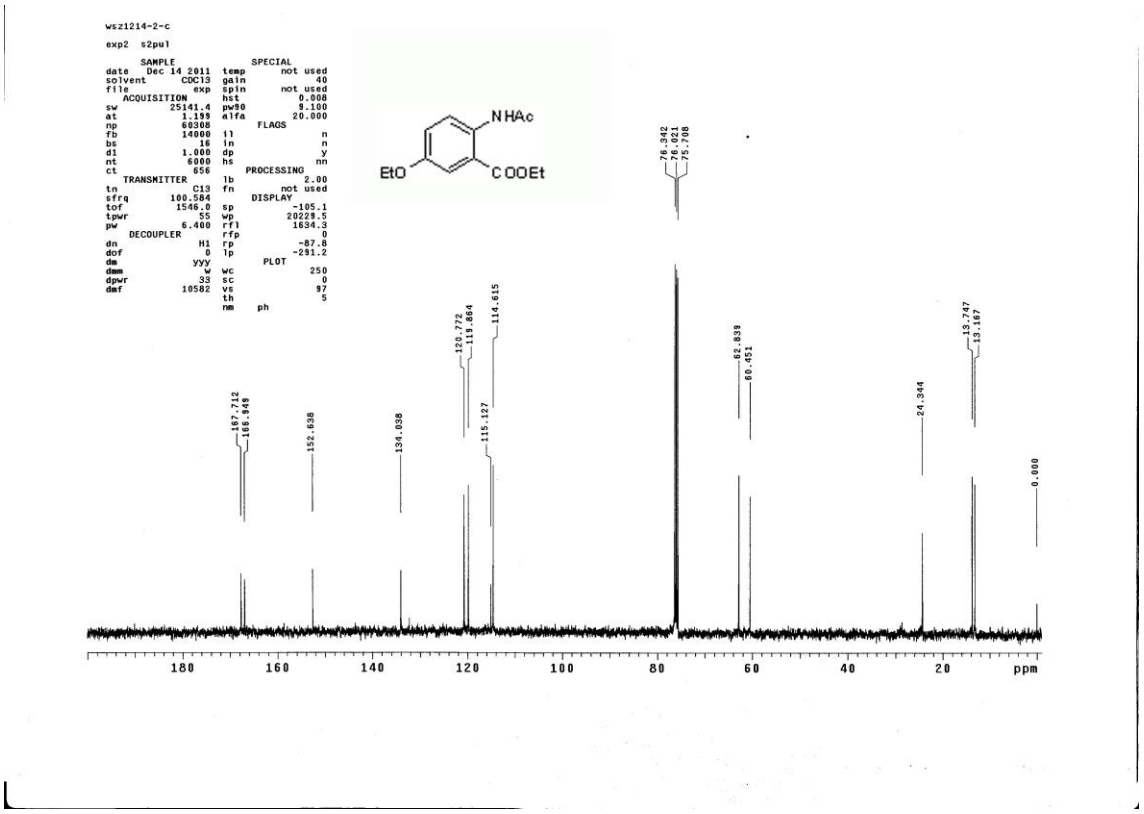

wsz1214-4
exp1 s2pul
NHAc
Me
COOEt
11.8 ppm
ppm

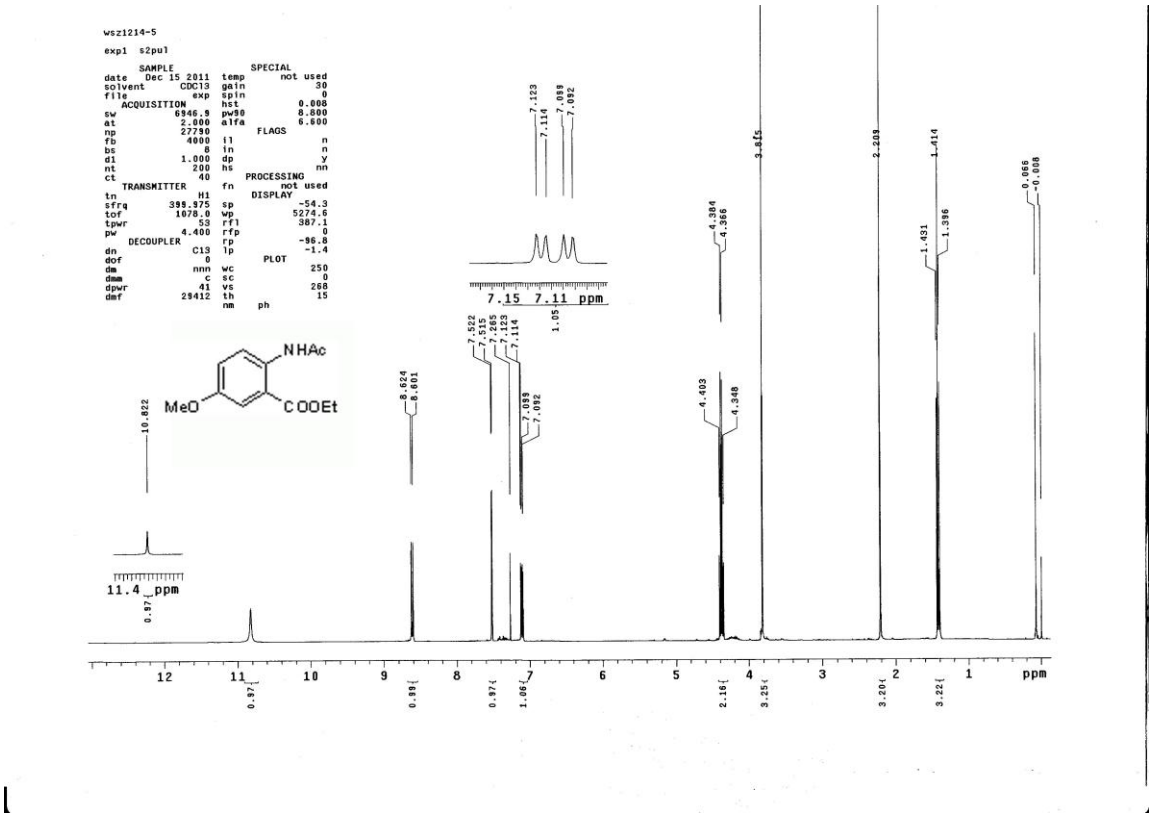

wsz1214-4-c
exp2 s2pul
NHAc
Me
COOEt
ppm

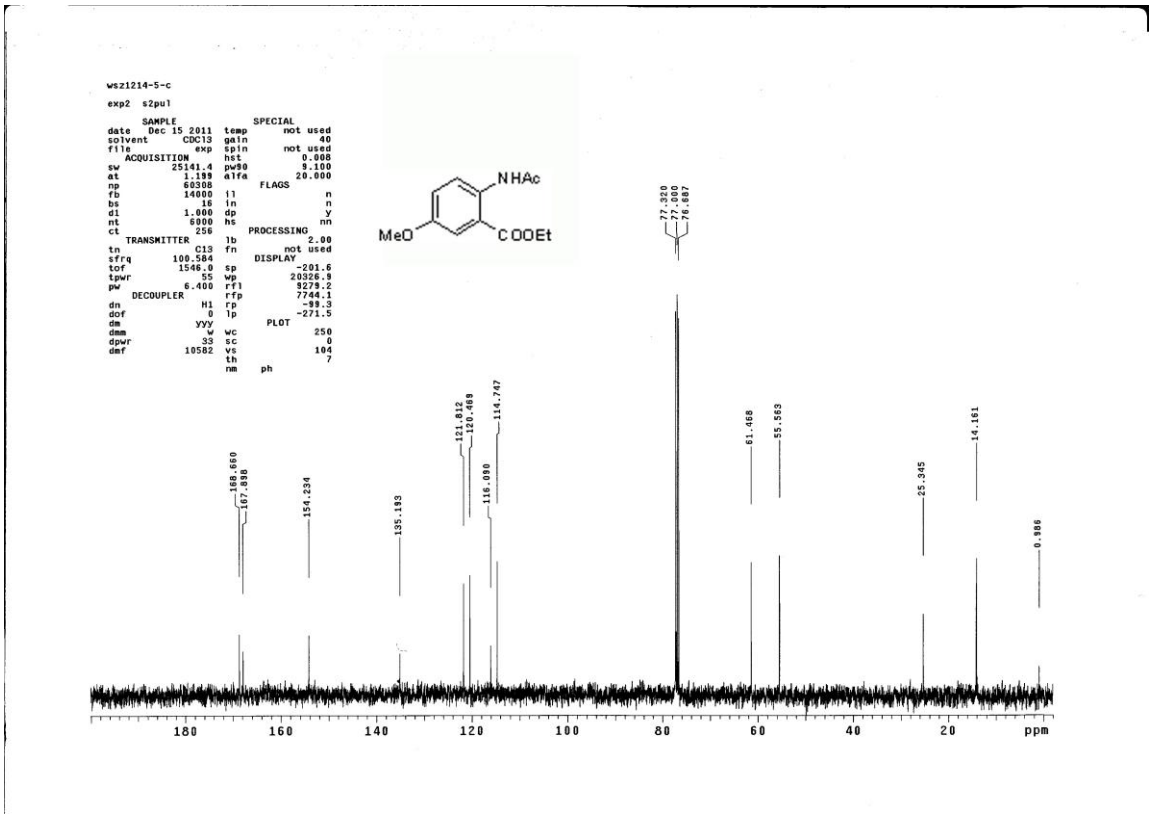

wsz-3
exp1 s2pul
SAMPLE
date Dec 12 2011
solvent CDCl3
file exp
ACQUISITION
sw 6946.8
at 2.000
np 27790
fb 4000
bs 8
d1 1.000
nt 200
ct 32
TRANSMITTER
tn H1
sfrq 399.975
tof 1078.0
tpwr 53
pw 4.400
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 41
dmf 29412
SPECIAL
temp not used
gain 30
spin 0
hst 8.008
pw90 8.800
alfa 6.600
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -43.7
wp 5220.7
rfl 385.4
rfp 0
rp -94.0
lp -11.6
PLOT
wc 250
sc 0
vs 127
th 7
nm ph
NHAc
Et
COOEt
11.005
8.608
8.586
7.856
7.845
7.395
7.390
7.374
7.369
7.270
4.391
4.373
4.409
4.355
2.661
2.642
2.623
2.604
2.229
1.449
1.431
1.414
1.250
1.231
1.212
0.873
0.860
11.3 ppm
0.89
ppm

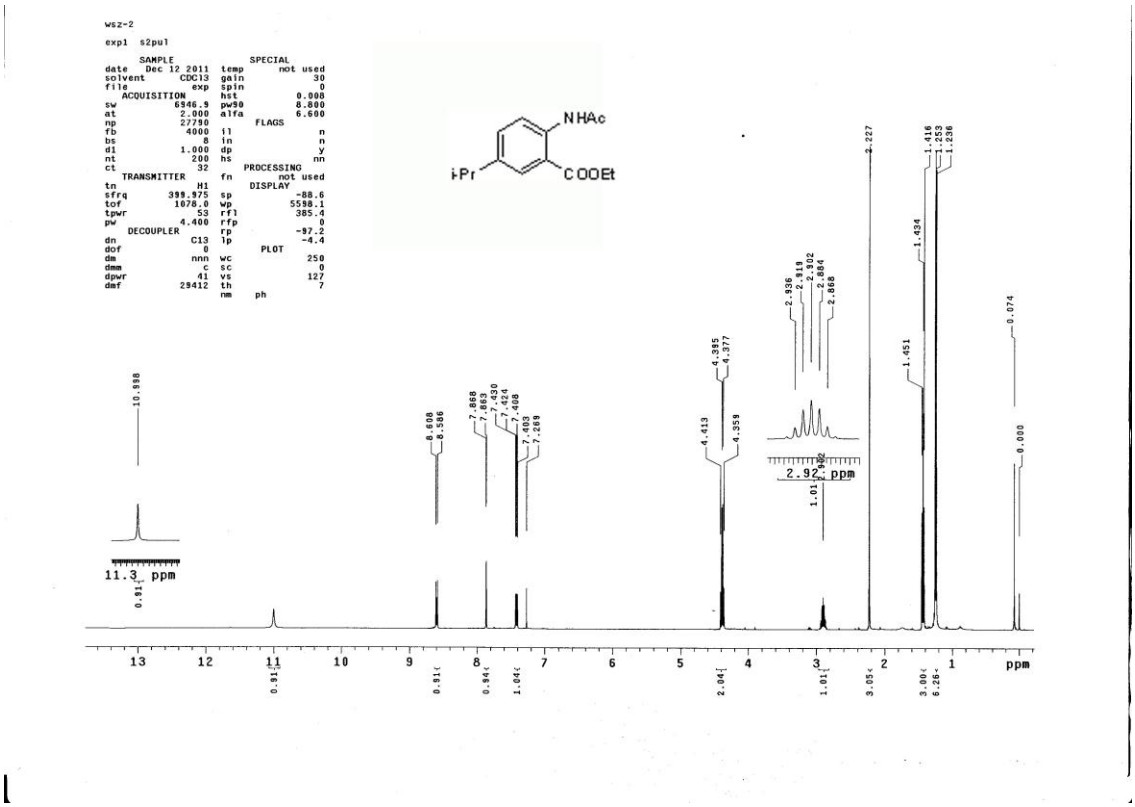

wsz-3/C13
exp2 s2pul
SAMPLE
date Dec 12 2011
solvent CDCl3
file exp
ACQUISITION
sw 25141.4
at 1.199
np 60308
fb 14000
bs 16
d1 1.000
nt 1000
ct 528
TRANSMITTER
tn C13
sfrq 100.584
tof 1546.0
tpwr 55
pw 6.400
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 33
dmf 10582
SPECIAL
temp not used
gain 40
spin not used
hst 0.008
pw90 9.100
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 2.00
fn not used
DISPLAY
sp -819.2
wp 21757.8
rfl 9279.2
rfp 7744.1
rp -113.9
lp -256.2
PLOT
wc 250
sc 0
vs 85
th 4
nm ph
NHAc
Et
COOEt
77.313
77.000
76.680
166.905
166.371
138.312
138.275
134.125
129.662
120.347
114.946
61.292
28.099
25.460
15.588
14.199
0.986
ppm

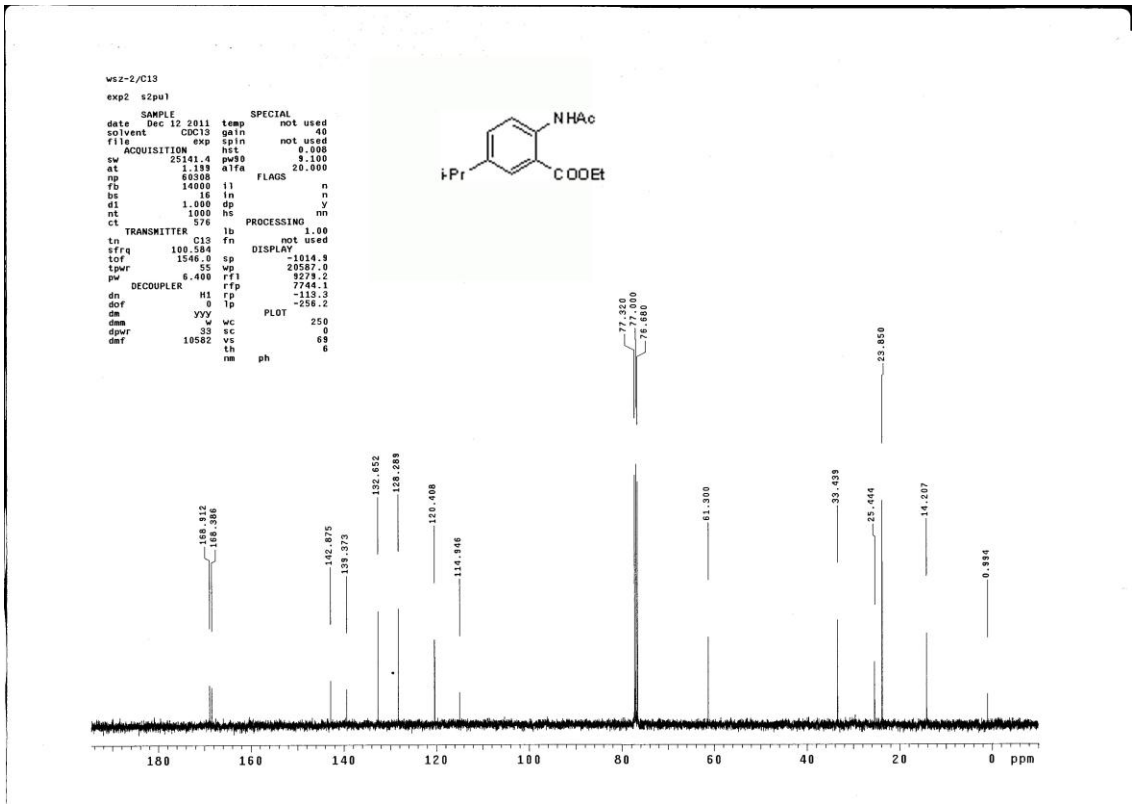

wsz1214-2
exp1 s2pul
NHAc
EtO
COOEt
1.45 ppm
7.11 ppm
11.0 ppm
ppm

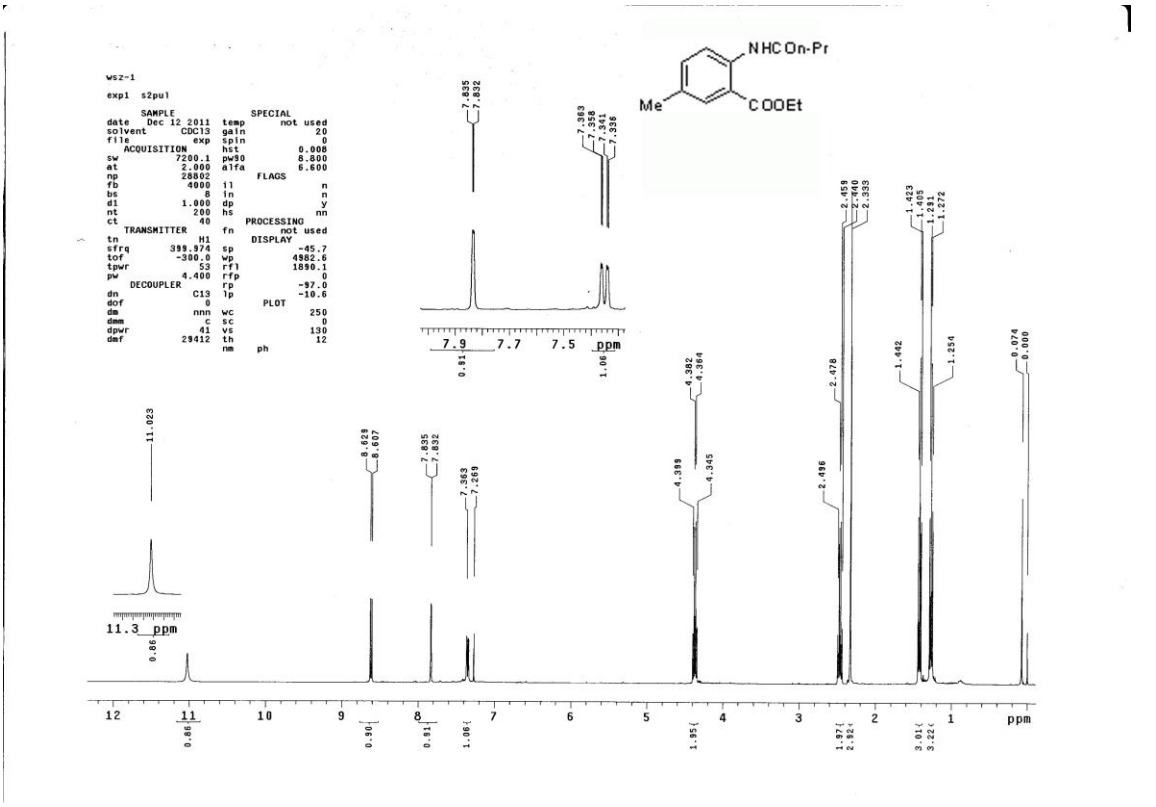

wsz1214-2-c
exp2 s2pul
NHAc
EtO
COOEt
ppm

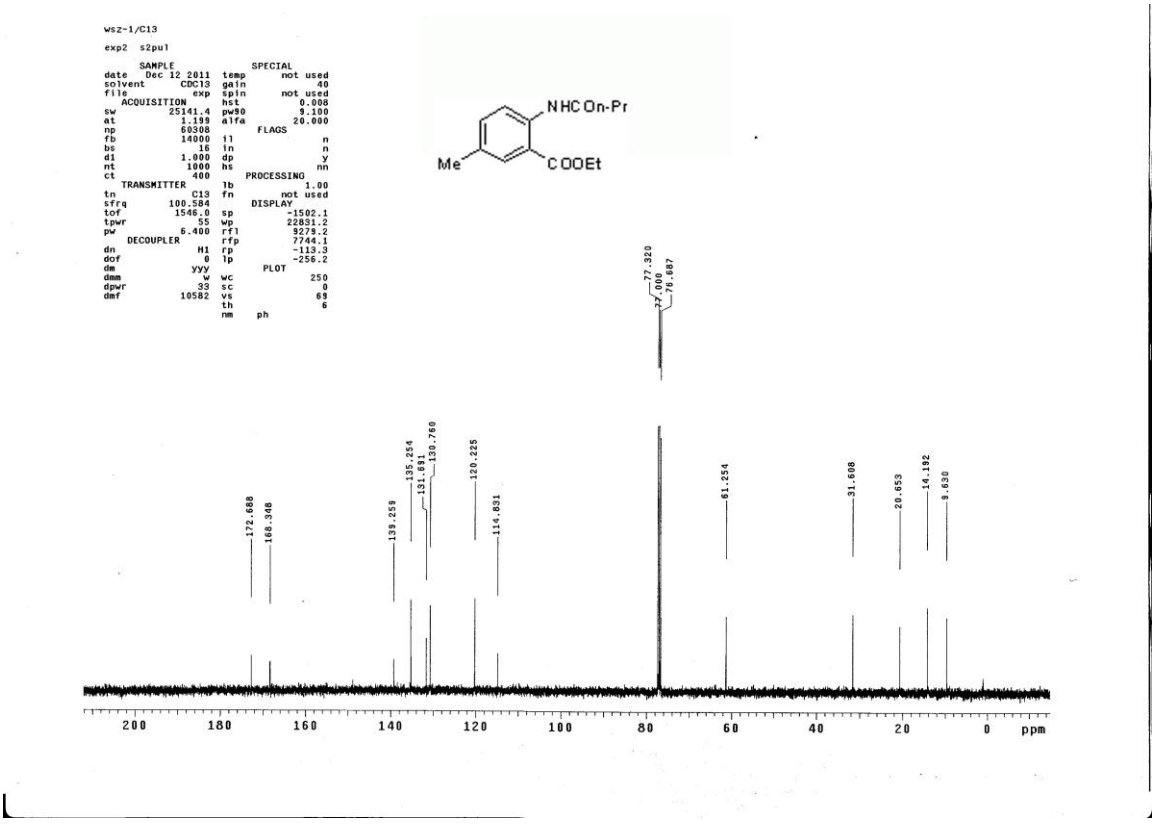

wsz1214-5
exp1 s2pul
NHAc
MeO
COOEt
7.15 7.11 ppm
11.4 ppm
ppm

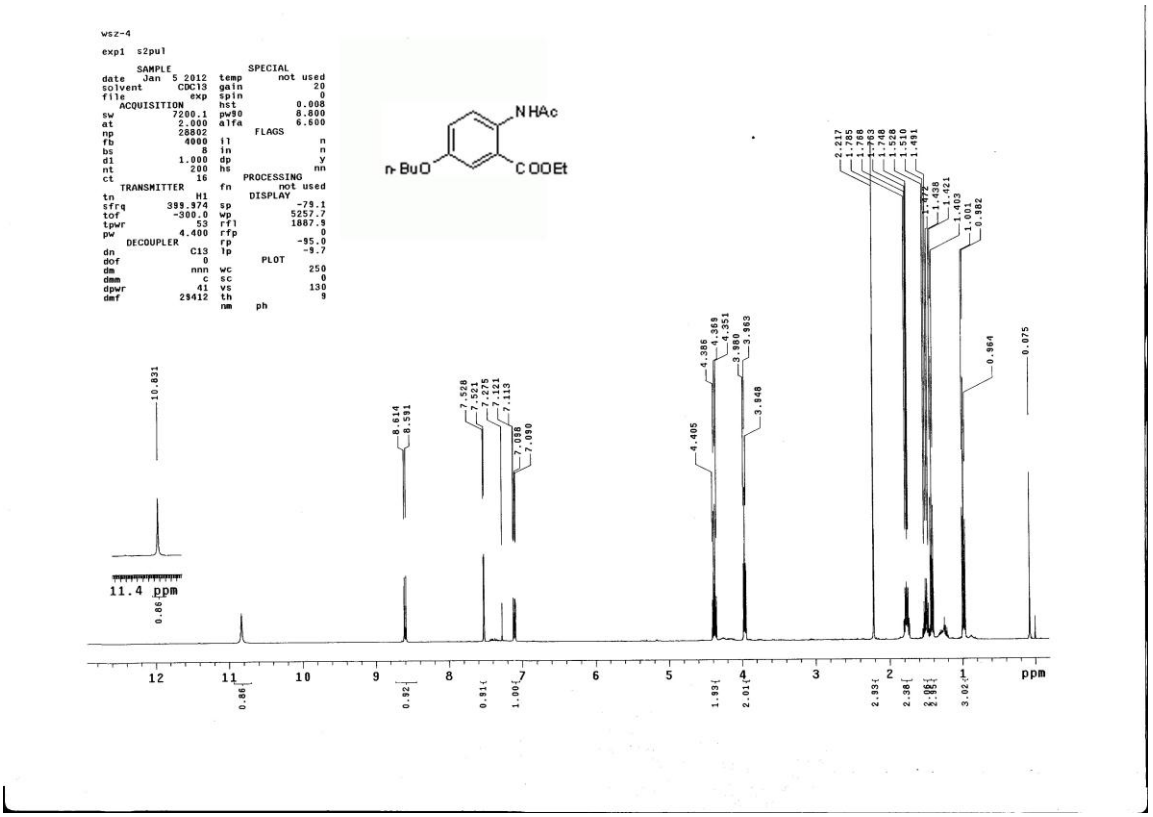

wsz1214-5-c
exp2 s2pul
NHAc
MeO
COOEt
ppm

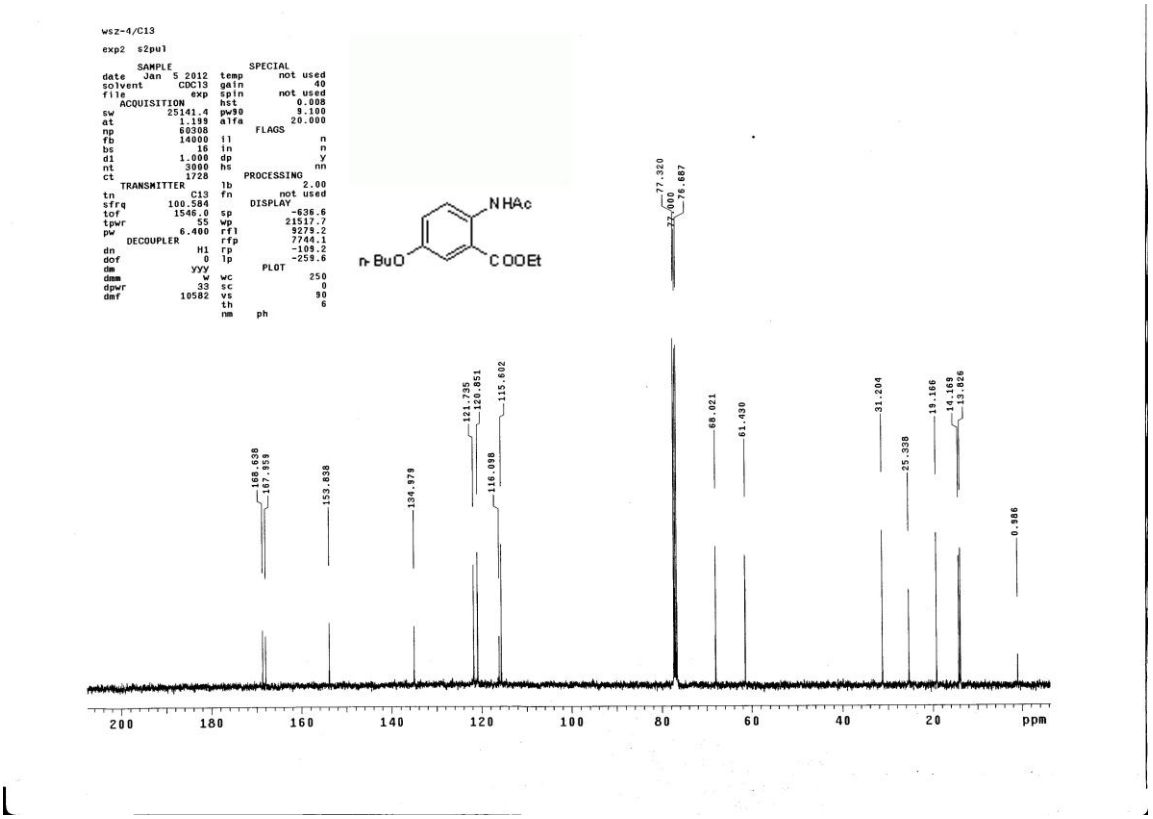

wsz-2
exp1 s2pul
SAMPLE
date Dec 12 2011
solvent CDCl3
file exp
ACQUISITION
sw 6946.9
at 2.000
np 27790
fb 4000
bs 8
d1 1.000
nt 200
ct 32
TRANSMITTER
tn H1
sfrq 399.975
tof 1078.0
tpwr 53
pw 4.400
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 41
dmf 29412
SPECIAL
temp not used
gain 30
spin 0
hst 0.000
pw90 8.800
alfa 6.600
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -88.6
wp 5538.1
rfl 385.4
rfp 0
rp -97.2
lp -4.4
PLOT
wc 250
sc 0
vs 127
th 7
nm ph
NHAc
i-Pr
COOEt
10.988
8.608
8.586
7.868
7.863
7.439
7.434
7.408
7.403
7.289
4.413
4.385
4.377
4.359
2.938
2.918
2.902
2.884
2.868
2.227
1.451
1.434
1.418
1.415
1.236
0.074
0.000
11.3 ppm
2.92 ppm
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm

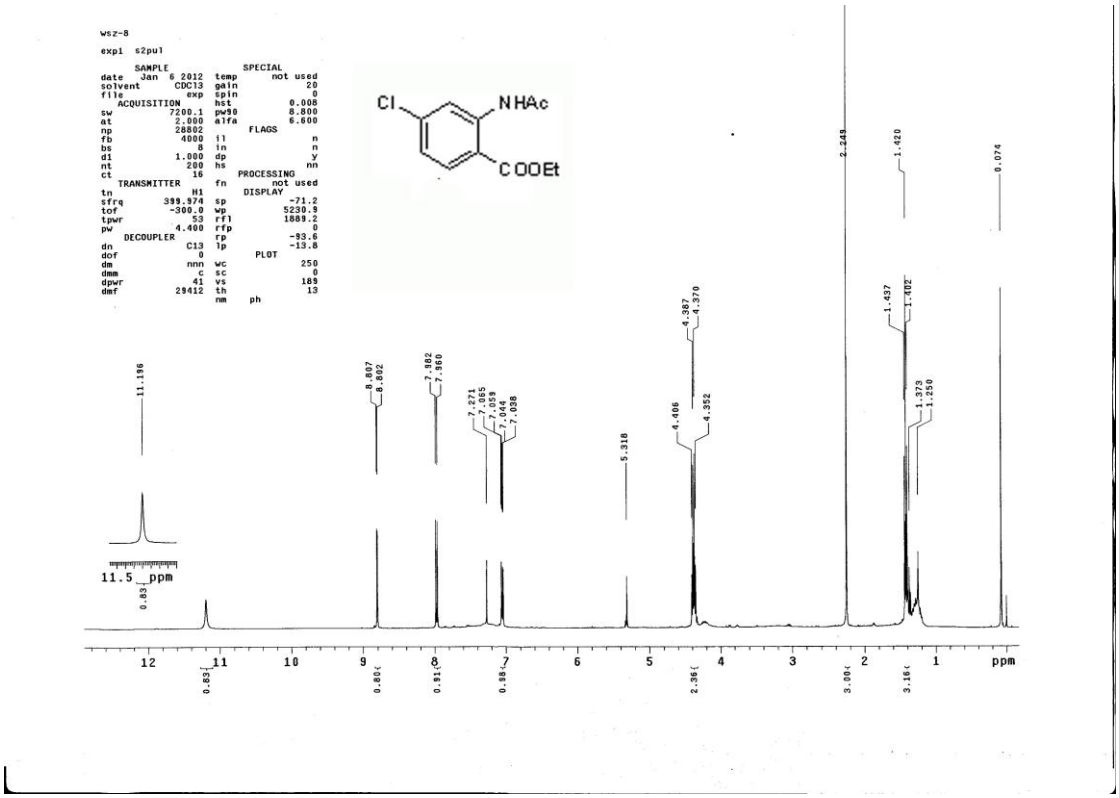

wsz-2/C13
exp2 s2pul
SAMPLE
date Dec 12 2011
solvent CDCl3
file exp
ACQUISITION
sw 25141.4
at 1.199
np 60308
fb 14000
bs 16
d1 1.000
nt 1000
ct 576
TRANSMITTER
tn C13
sfrq 100.584
tof 1546.0
tpwr 55
pw 6.400
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 33
dmf 10582
SPECIAL
temp not used
gain 40
spin not used
hst 0.000
pw90 9.100
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn not used
DISPLAY
sp -1014.9
wp 20587.0
rfl 9279.2
rfp 7744.1
rp -113.3
lp -256.2
PLOT
wc 250
sc 0
vs 69
th 6
nm ph
NHAc
i-Pr
COOEt
168.812
166.386
142.875
139.373
132.652
128.289
120.408
114.946
77.320
77.000
76.680
61.300
33.439
25.444
23.850
14.207
0.934
180
160
140
120
100
80
60
40
20
0
ppm

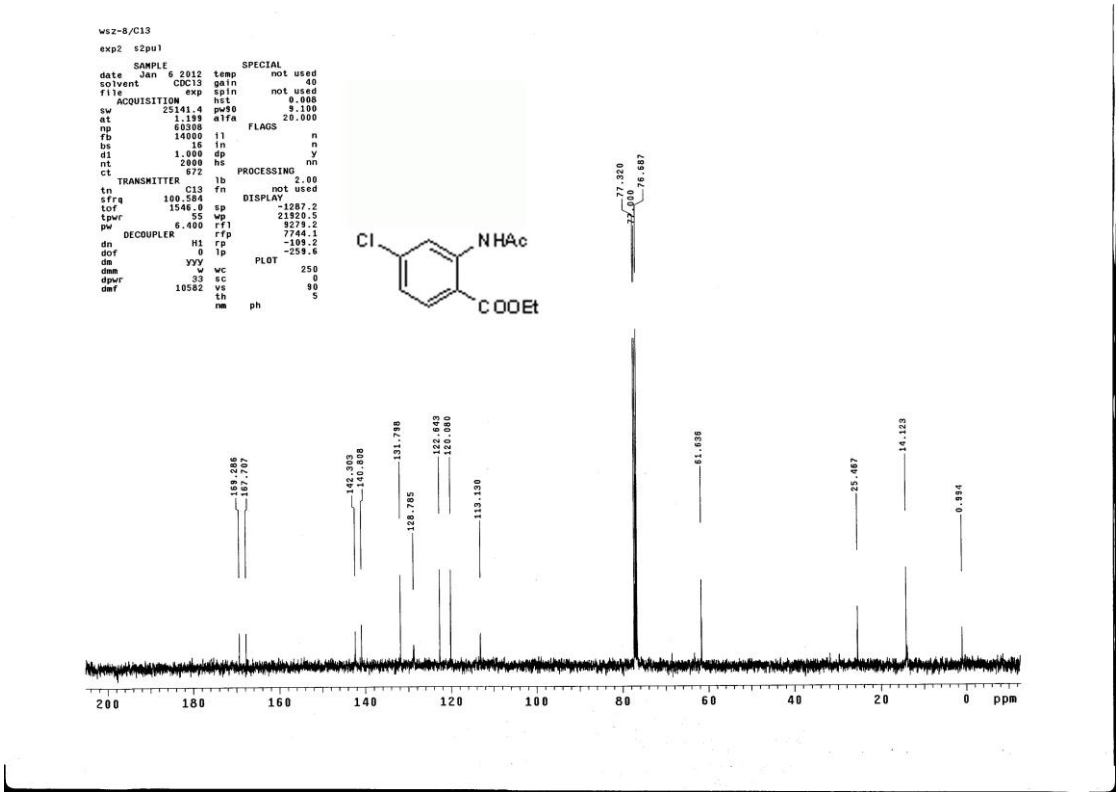

NHC On-Pr
Me
COOEt
11.3 ppm
ppm


NHC On-Pr
Me
COOEt
ppm


wsz-4
exp1 s2pul
SAMPLE
date Jan 5 2012
solvent CDCl3
file exp
ACQUISITION
sw 7200.1
at 2.000
np 28802
fb 4000
bs 8
dl 1.000
nt 200
ct 16
TRANSMITTER
tn H1
sfrq 399.974
tof -300.0
tpwr 53
pw 4.400
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 41
dmf 25412
SPECIAL
temp not used
gain 20
spin 0
hst 0.008
pw90 8.800
alfa 6.600
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -79.1
wp 5257.7
rfl 1887.9
rfp 0
rp -95.0
lp -9.7
PLOT
wc 250
sc 0
vs 130
th 9
nm ph
NHAc
n-BuO
COOEt
11.4 ppm
ppm


wsz-4/C13
exp2 s2pul
SAMPLE
date Jan 5 2012
solvent CDCl3
file exp
ACQUISITION
sw 25141.4
at 1.198
np 60308
fb 14000
bs 16
dl 1.000
nt 3000
ct 1728
TRANSMITTER
tn C13
sfrq 100.584
tof 1546.0
tpwr 55
pw 6.400
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 33
dmf 10582
SPECIAL
temp not used
gain 40
spin not used
hst 0.008
pw90 9.100
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 2.00
fn not used
DISPLAY
sp -636.6
wp 21517.7
rfl 9279.2
rfp 7744.1
rp -109.2
lp -259.6
PLOT
wc 250
sc 0
vs 90
th 6
nm ph
NHAc
n-BuO
COOEt
ppm


wsz-8
exp1 s2pul
SAMPLE
date Jan 6 2012
solvent CDCl3
file exp
ACQUISITION
sw 7200.1
at 2.000
np 28802
fb 4000
bs 8
d1 1.000
nt 200
ct 16
TRANSMITTER
tn H1
sfrq 399.974
tof -300.0
tpwr 53
pw 4.400
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 41
dmf 29412
SPECIAL
temp not used
gain 20
spin 0
hst 0.008
pw90 8.800
alfa 6.600
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -71.2
wp 5230.9
rfl 1889.2
rfp 0
rp -93.6
lp -13.8
PLOT
wc 250
sc 0
vs 189
th 13
nm ph
Cl
NHAc
COOEt
11.196
8.807
8.602
7.382
7.360
7.271
7.065
7.059
7.044
7.038
5.310
4.406
4.387
4.370
4.352
2.249
1.437
1.420
1.402
1.373
1.250
0.074
11.5 ppm
12
11
10
9
8
7
6
5
4
3
2
1
ppm


wsz-8/C13
exp2 s2pul
SAMPLE
date Jan 6 2012
solvent CDCl3
file exp
ACQUISITION
sw 25141.4
at 1.199
np 60308
fb 14000
bs 16
d1 1.000
nt 2000
ct 672
TRANSMITTER
tn C13
sfrq 100.584
tof 1546.0
tpwr 55
pw 6.400
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 33
dmf 10582
SPECIAL
temp not used
gain 40
spin not used
hst 0.008
pw90 5.100
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 2.00
fn not used
DISPLAY
sp -1287.2
wp 21920.5
rfl 9279.2
rfp 7744.1
rp -109.2
lp -259.6
PLOT
wc 250
sc 0
vs 90
th 5
nm ph
Cl
NHAc
COOEt
169.286
167.707
142.303
140.808
131.798
128.785
122.643
120.080
113.130
77.320
77.007
76.687
61.638
25.467
14.123
0.994
200
180
160
140
120
100
80
60
40
20
0
ppm