Supporting Information

One-step preparation of xanthones via Pd-catalyzed annulation of 1,2-dibromoarenes and salicylaldehydes†

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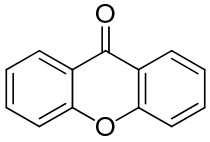
General Consideration:

All solvents and reagents were purchased from the suppliers and used without further purification. ^{1}H NMR and ^{13}C NMR were recorded in $CDCl_3$ at room temperature on a Varian INOVA-400 spectrometer (400 MHz ^{1}H). The chemical-shifts scale is based on internal TMS. All reactions were carried out under dry nitrogen atmosphere.

Representative Procedure and Selected Compounds Data

9H- xanthone[1,2,3]



In a 25ml round-bottom flask equipped with a reflux condenser under N_2 was placed salicylaldehyde (122 mg, 1 mmol) and 1,2-dibromobenzene (470 mg, 2 mmol), K_2CO_3 (278 mg, 2 mmol), $(PPh_3)_2PdCl_2$ (35 mg, 0.05 mmol) and 5ml DMF. The mixture was heated to 130 ℃ for 12 hours before it was cooled to room temperature . The reaction mixture was diluted with diethyl ether (25 mL) and washed with water and brine. The aqueous layer was extracted with diethyl ether (3x25 mL). The organic layers were combined and dried with MgSO4, filtered, and the solvent was removed under reduced pressure. The residue was purified by flash chromatography on silica gel (petroleum ether/ethyl acetate, 50/1) to afford the 118 mg of the desired product in 61% yield.

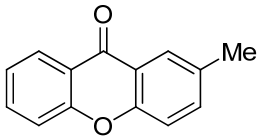
Yield: 61%，colorless crystals, mp: 172-173 ℃;

^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.26-7.30 (m, 2 H), 7.39 (d, *J* = 8.4 Hz, 2 H), 7.6 (t, *J* = 4.2 Hz, 2 H), 8.25 (t, *J* = 3.8 Hz, 2 H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 116.93 (2 C), 120.78 (2 C), 122.85 (2 C), 125.66 (2 C), 133.76 (2 C), 155.10 (2 C), 176.15;

HRMS m/z calcd. for $C_{13}H_8O_2$, 196.0524, found 196.0529.

2-Methyl-9H-xanthone[2]



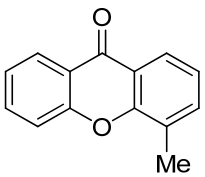
Yield: 54%, colorless oil;

^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.39 (s, 3 H), 7.27-7.32 (m, 2 H), 7.40 (d, *J* = 8.4 Hz, 1 H), 7.45 (d, *J* = 8.6 Hz, 1 H), 7.63 (t, *J* = 7.6 Hz, 1 H), 8.04 (s, 1 H), 8.26 (d, *J* = 8.0 Hz, 1 H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 19.81, 116.72, 116.93, 120.47, 120.81, 122.68, 125.01, 125.72, 132.68, 133.60, 135.05, 153.40, 155.19, 176.27;

HRMS m/z calcd for C14H10O2, 210.0681, found 210.0686..

4-Methyl-9H-xanthone[1]



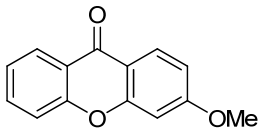
Yield: 56%, colorless crystals, mp: 125-126 ℃;

^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.47 (s, 3 H), 7.19-7.15 (m, 1 H), 7.28 (t, *J* = 7.6 Hz, 1 H), 7.30-7.47 (m, 2 H), 7.61-7.65 (m, 1 H), 8.09 (d, *J* = 8.4 Hz, 1 H), 8.25 (d, *J* = 8.0 Hz, 1 H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 14.75, 117, 120.63（2 C）, 122.37, 122.81, 123.28, 125.65, 126.22, 133.60, 134.65, 153.52, 155.03, 176.50;

HRMS m/z calcd for C14H10O2, 210.0681, found 210.0684.

3-Methoxy-9H-xanthone[2]



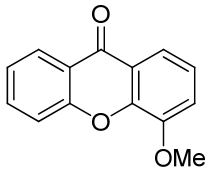
Yield: 53%, colorless oil..

^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.86 (s, 3 H), 6.81 (s, 1 H), 6.87 (d, *J* = 8.8 Hz, 1 H), 7.30 (t, *J* = 7.2 Hz, 1 H), 7.38 (d, *J* = 8.4 Hz, 1 H), 7.60-7.64 (m, 1 H), 8.18 (d, *J* = 8.8 Hz, 1 H), 8.25 (d, *J* = 7.6 Hz , 1 H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 54.80, 99.21, 112.23, 114.84, 116.66, 120.99, 122.86, 125.66, 127.29, 133.25, 155.22, 157.08, 164.1, 175.27;

HRMS m/z calcd for C14H10O3, 226.0630, Found 226.0632.

4-Methoxy-9H-xanthone[1,2]



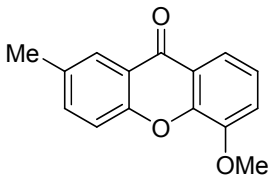
Yield: 52%, colorless crystals, mp: 174-175 ℃;

^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.94 (s, 3 H), 7.14 (d, *J* = 8.0 Hz, 1 H), 7.18 (d, *J* = 8.0 Hz, 1 H), 7.28-7.53 (m, 1 H), 7.51 (d, *J* = 8.0 Hz, 1 H), 7.62-7.66 (m, 1 H), 7.81 (d, *J* = 7.6 Hz, 1 H), 8.25 (d, *J* = 8.0 Hz, 1 H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 56.311, 115.26, 117.48, 118.18, 121.58, 122.60, 123.33, 123.97, 126.52, 134.66, 146.43, 148.53, 155.84, 177.04;

HRMS m/z calcd for C14H10O3, 226.0630, Found 226.0634.

5-Methoxy-2-methyl-9H-xanthone / 5-Methoxy-3-methyl-9H-xanthone



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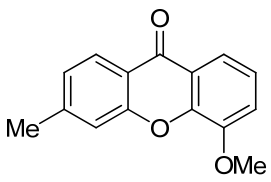


b

Yield: 63%, colorless oil.

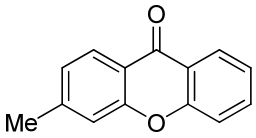
^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.40 (s, 3 H), 2.43 (s, 3 H), 3.96 (s, 6 H), 7.14-7.23 (m, 5 H), 7.34 (s, 1 H), 7.46 (t, *J* = 4.0 Hz, 2 H) 7.82-7.85 (m, 2 H), 8.043 (s, 1 H), 8.14 (d, *J* = 8.0 Hz, 1 H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 19.82, 20.94, 55.41 (2 C), 114.20, 116.63 (2 C), 116.67 (2 C), 117.03 (2 C), 122.20 (2 C), 122.26 (2 C), 124.61, 124.91, 125.41 (2 C), 132.88, 135.04, 145.25, 145.61 (2 C), 147.64 (2 C), 153.21, 155.05, 175.94, 176.20;

LRMS (CI) for $C_{15}H_{12}O_3$ $[M+H]^+$ calcd. 241, found 241.

Anal. Calcd for $C_{15}H_{12}O_3$: C, 74.99; H, 5.03; Found: C, 75.05; H, 5.12.

3-Methyl-9H-xanthone[2] / 2-Methyl-9H-xanthone[2]



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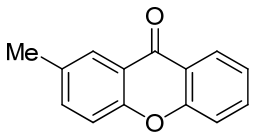


b

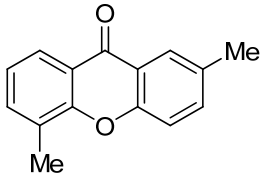
Yield: 56% (**a**: **b** = 1 : 1), colorless oil.

^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.35 (s, 3 H), 2.38 (s, 3 H), 7.06 (d, *J* = 8.4 Hz, 1 H), 7.14 (s, 1 H), 7.25 (t, *J* = 7.8 Hz, 1 H), 7.34-7.36 (m, 2 H), 7.39-7.42 (m, 1 H), 7.57-7.61 (m, 2 H), 8.00 (s, 1 H), 8.10 (d, *J* = 8.0 Hz, 1 H), 8.21-8.24 (m, 2 H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 19.77, 20.92, 116.68, 116.86 (2 C), 116.89 (2 C), 122.63 (2 C), 124.70, 124.38, 124.96, 125.45, 125.62, 125.66 (2 C), 132.63, 133.48, 133.55, 133.73, 134.99, 145.25, 153.34, 155.09, 155.13, 155.22, 175.91, 176.18;

LRMS (CI) for $C_{14}H_{10}O_2$ $[M+H]^+$ calcd. 211, found 211.

2,5-Dimethyl-9H-xanthone **3,5-Dimethyl-9H-xanthone**



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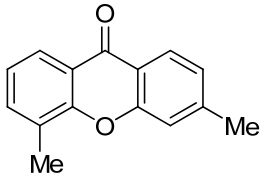


b

Yield: 60% (a: b = 1 : 1), colorless oil.

a: colorless oil. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.42 (s, 3 H), 2.46 (s, 3 H), 7.10 (d, *J* = 8.0 Hz, 1 H), 7.17 (t, *J* = 7.2 Hz, 1 H), 7.24 (s, 1 H), 7.46 (d, *J* = 7.2 Hz, 1 H), 8.09 (d, *J* = 8.0 Hz, 1 H), 8.24 (t, *J* = 7.6 Hz, 1 H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 14.75, 20.93, 116.77, 118.41, 120.71, 122.229, 123.25, 124.37, 125.44, 126.12, 134.43, 145.10, 153.48, 155.14, 176.32;

LRMS (CI) for $C_{15}H_{12}O_2$ $[M+H]^+$ calcd. 225, found 225.

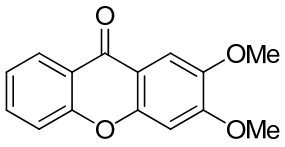
b: colorless oil. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.39 (s, 3H), 2.48 (s, 3 H), 7.19 (m, 1 H), 7.35 (d, *J* = 8.0 Hz, 1 H), 7.45 (d, *J* = 8.0 Hz, 1 H), 7.47 (d, *J* = 7.2 Hz, 1 H), 8.036 (s, 1 H), 8.10 (t, *J* = 8.0 Hz, 1 H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 14.78, 19.82, 116.78, 120.25, 120.61, 122.17, 123.30, 124.95, 126.18, 132.60, 134.47, 134.90, 153.29, 153.50, 176.61;

LRMS (CI) for $C_{15}H_{12}O_2$ $[M+H]^+$ calcd. 225, found 225.

Anal. Calcd for $C_{15}H_{12}O_2$: C, 80.34; H, 5.39; Found: C, 80.42; H, 5.44.

2,3-Dimethoxy-9H-xanthone[2,3]



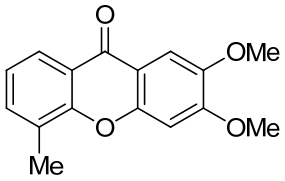
Yield: 36%, colorless oil.

^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.86 (s, 3 H), 3.94 (s, 3 H), 6.85 (s, 1 H), 7.30 (t, *J* = 8.0 Hz, 1 H), 7.38 (d, *J* = 8.0 Hz, 1 H), 7.60 (s, 1 H), 7.63 (m, 1 H), 8.27 (d, *J* = 8.0 Hz, 1 H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 55.33, 55.45, 98.63, 104.42, 113.91, 116.64, 120.5.1, 122.73, 125.53, 132.93, 145.73, 151.43, 154.45, 155.06, 175.04;

HRMS m/z calcd forC15H12O4, 256.0736, found 256.0740.

2,3-Dimethoxy-5-methyl-9H-xanthone



Yield: 33%, colorless oil.

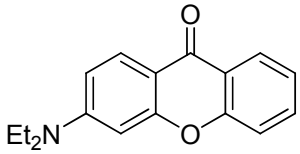
^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.47 (s, 3 H), 3.92 (s, 3 H), 3.95 (s, 3 H), 6.86 (s, 1 H), 7.16-7.20 (m, 1 H), 7.44 (d, *J* = 7.2 Hz, 1 H), 7.58 (s, 1 H), 8.10 (d, *J* = 8.0 Hz, 1 H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 14.77, 55.32, 55.47, 98.70, 104.36, 113.71, 120.31, 122.23, 123.11, 125.89, 133.84, 145.70, 151.30, 153.47, 154.36, 175.38;

LRMS (CI) for $C_{16}H_{14}O_4$ $[M+H]^+$ calcd. 271, found 271.

Anal. Calcd for $C_{16}H_{14}O_4$: C, 71.10; H, 5.22; Found: C, 71.20; H, 5.30.

3-(Diethyl amino) - 9H-xanthone



Yield: 38%, black oil.

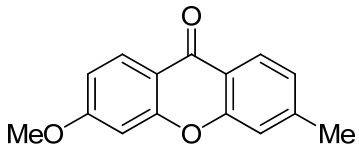
^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.17 (t, *J* = 7.2 Hz, 6 H), 3.35-3.41 (m, 4 H), 6.4 (s, 1 H), 6.60-6.63 (m, 1 H), 7.19-7.25 (m, 1 H), 7.30 (d, *J* = 8.0 Hz, 1 H), 7.52-7.56 (m, 1 H), 8.05 (d, *J* = 9.2 Hz, 1 H) 8.21-8.23 (m, 1 H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 11.5 (2 C), 43.78 (2 C), 95.11, 108.39, 110.24, 116.29, 121.28, 122.27, 125.45, 127.14, 132.48, 151.82, 155.10, 157.66, 174.58;

LRMS (CI) for $C_{17}H_{17}NO_2$ $[M+H]^+$ calcd. 268, found 268.

Anal. Calcd for $C_{17}H_{17}NO_2$: C, 76.38; H, 6.41; N, 5.24; Found: C, 76.48; H, 6.52; N, 5.28.

3-Methoxy-6-methyl-9H-xanthone **6-Methoxy-2-methyl-9H-xanthone**



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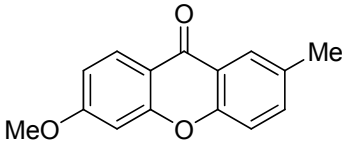


b

Yield: 59% (**a** : **b** = 1 : 1), colorless oil.

a: ^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.41 (s, 3 H), 3.84 (s, 3 H), 6.74 (s, 1 H), 6.82-6.85 (m, 1 H), 7.08 (d, *J* = 8.0 Hz, 1 H), 7.16 (d, *J* = 8.0 Hz, 1 H), 8.10 (d, *J* = 8.0 Hz, 1 H), 8.14 (d, *J* = 8.0 Hz, 1 H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 21.88, 55.75, 100.17, 112.99, 115.82 (2 C), 117.45, 119.66, 125.31, 126.39, 128.16, 145.66, 156.27, 157.97, 164.87, 176.11;

LRMS (CI) for $C_{15}H_{12}O_3$ $[M+H]^+$ calcd. 241, found 241;

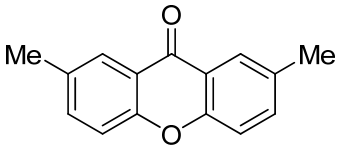
b: ^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.37 (s, 3 H), 3.83 (s, 3 H), 6.75 (s, 1 H), 6.81-6.84 (m, 1 H), 7.24 (d, *J* = 8.0 Hz, 1 H), 7.39 (d, *J* = 8.0 Hz, 1 H), 8.0 (d, *J* = 8.0 Hz, 1 H), 8.14 (d, *J* = 8.0 Hz, 1 H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 20.78, 55.74, 100.05, 113.05, 115.72 (2 C), 117.38, 121.50, 125.94, 128.18, 133.56, 135.42, 154.36, 158.01, 164.90, 176.31;

LRMS (CI) for $C_{15}H_{12}O_3$ $[M+H]^+$ calcd. 241, found 241.

Anal. Calcd for $C_{15}H_{12}O_3$: C, 74.99; H, 5.03; Found: C, 75.12; H, 5.16.

2,7-Dimethyl-9H-xanthone **2,6-Dimethyl-9H-xanthone**



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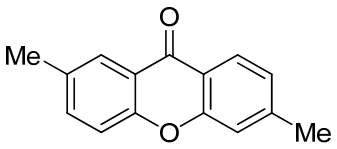


b

Yield: 61% (**a**: **b** = 1 : 1), colorless oil..

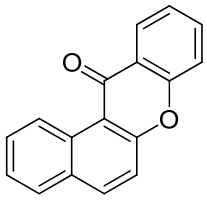
^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.34 (s, 9 H), 2.37 (s, 3 H), 7.04 (d, *J* = 8.0 Hz, 1 H), 7.11 (s, 1 H), 7.17-7.24 (m, 3 H), 7.37-7.39 (m, 3 H), 7.99 (s, 3 H), 8.09 (d, *J* = 8.0 Hz, 1 H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 19.78 (3 C), 20.93, 116.65, 116.68, 118.54 (3 C), 120.37 (2 C), 120.47, 124.23, 124.95 (2 C), 125.46, 132.43 (2 C), 132.50, 134.79 (3 C), 134.89, 145.09, 153.32 (2 C), 153.39, 155.26, 176.08, 176.35;

LRMS (CI) for $C_{15}H_{12}O_2$ $[M+H]^+$ calcd. 245, found 245.

Anal. Calcd for $C_{15}H_{12}O_2$: C, 80.34; H, 5.39; Found: C, 80.52; H, 5.48.

12H-benzo[a]xanthone



Yield: 34%, colorless oil..

^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.39 (t, *J* = 7.6 Hz, 1 H), 7.48-7.55 (m, 3 H), 7.65-7.74 (m, 2 H),

7.84 (d, *J* = 8.0 Hz, 1 H) , 8.07 (d, *J* = 9.2 Hz, 1 H), 8.38 (d, *J* = 8.0 Hz, 1 H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 113.62, 116.52, 117.04, 122.63, 123.32, 125.14, 125.71, 125.98, 127.35, 128.56, 129.17, 130.19, 132.93, 135.66, 153.713, 156.66, 177.52;

HRMS m/z calcd for $C_{17}H_{10}O_2$, 246.0681, found 246.0684.

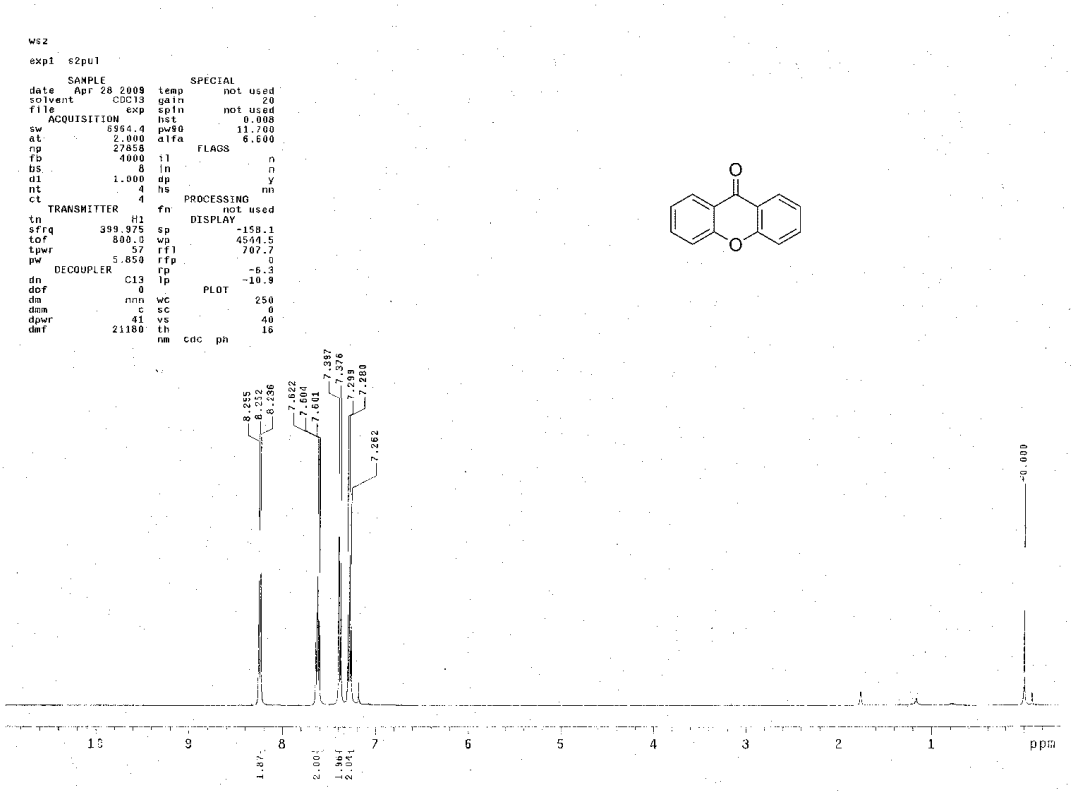
Anal. Calcd for $C_{17}H_{10}O_2$: C, 82.91; H, 4.09; Found: C, 83.03; H, 4.18.

Reference :

1. K. Okuma, A. Nojima, N. Matsunaga and K. Shioji, *Org. Lett*., 2009, **11**, 169.

2. Jian Zhao and Richard C. Larock*, *J. Org. Chem*., 2007, **72**, 583.

3. Jian Zhao and Richard C. Larock*, *Org. Lett*., 2005, **7**, 4273.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date Apr 28 2009
solvent CDCl3
file exp
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d1 1.000
nt 4
ct 4
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tpwr 57
pw 5.850
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dof 0
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dmm c
dpwr 41
dmf 21180
SPECIAL
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gain 20
spin not used
hst 0.008
pw90 11.700
alfa 6.600
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in n
dp y
hs nn
PROCESSING
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DISPLAY
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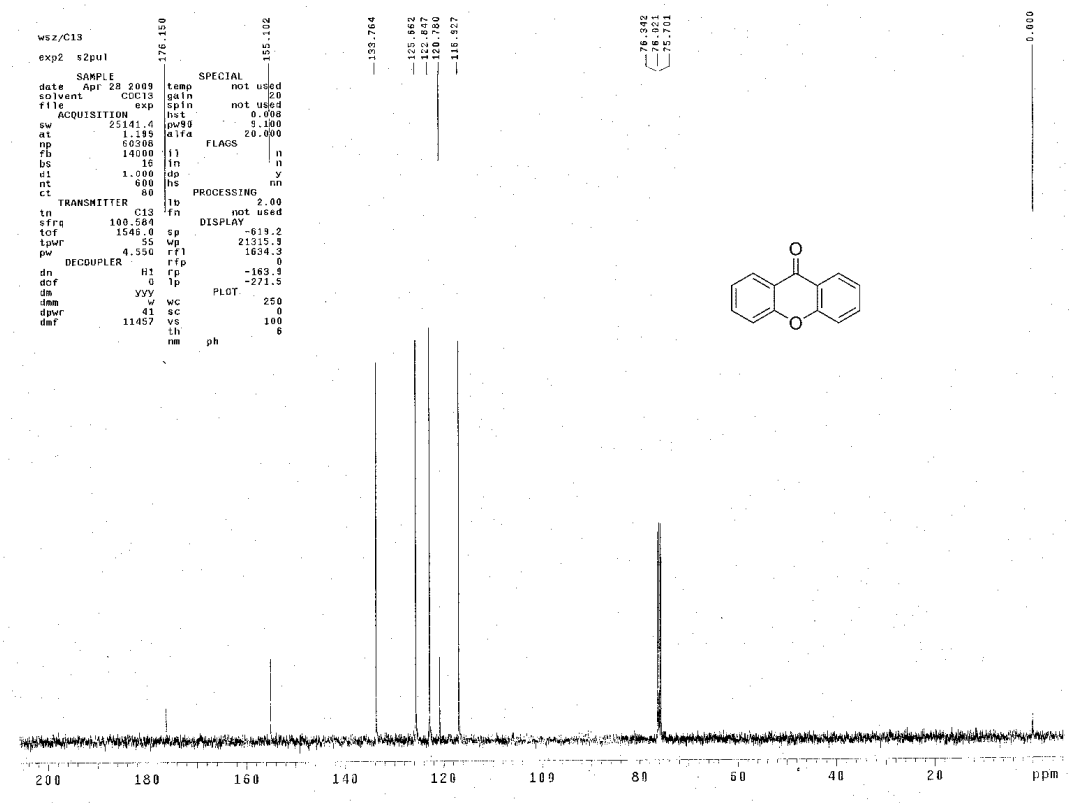
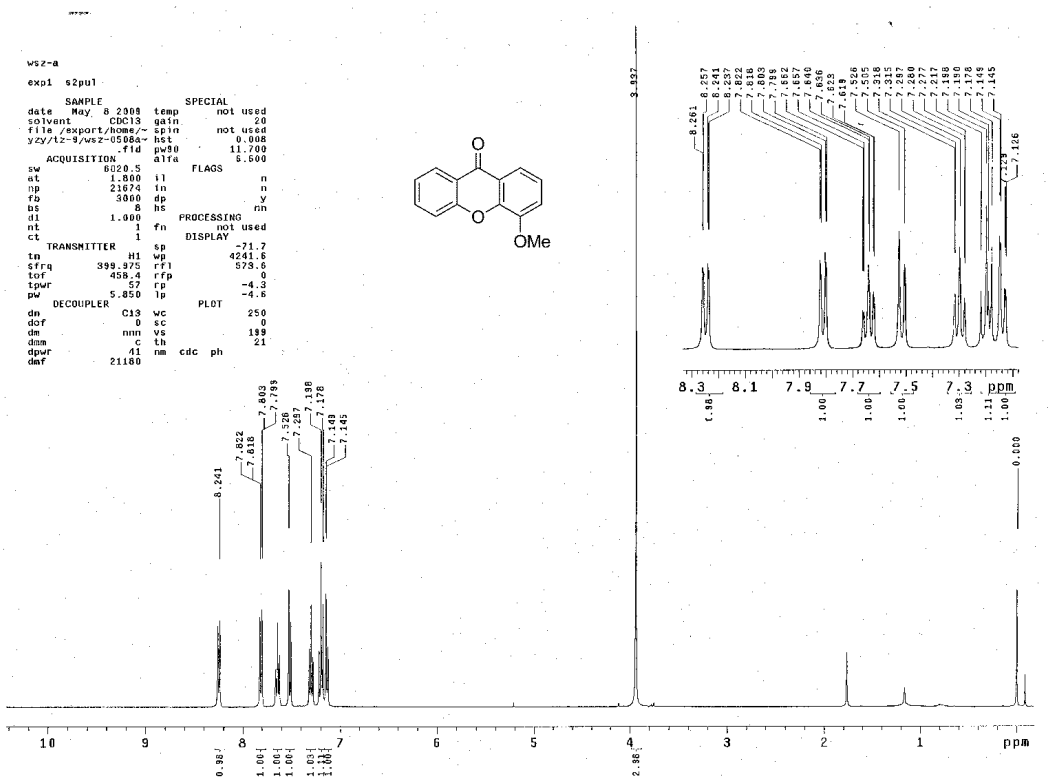
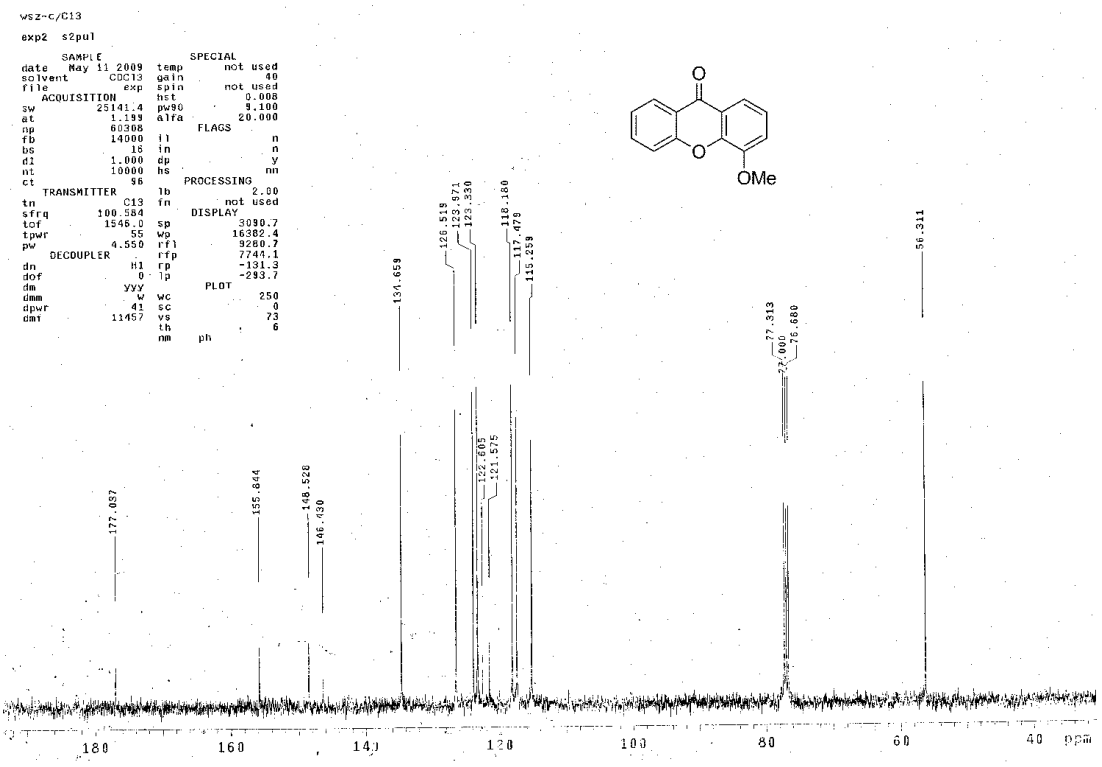
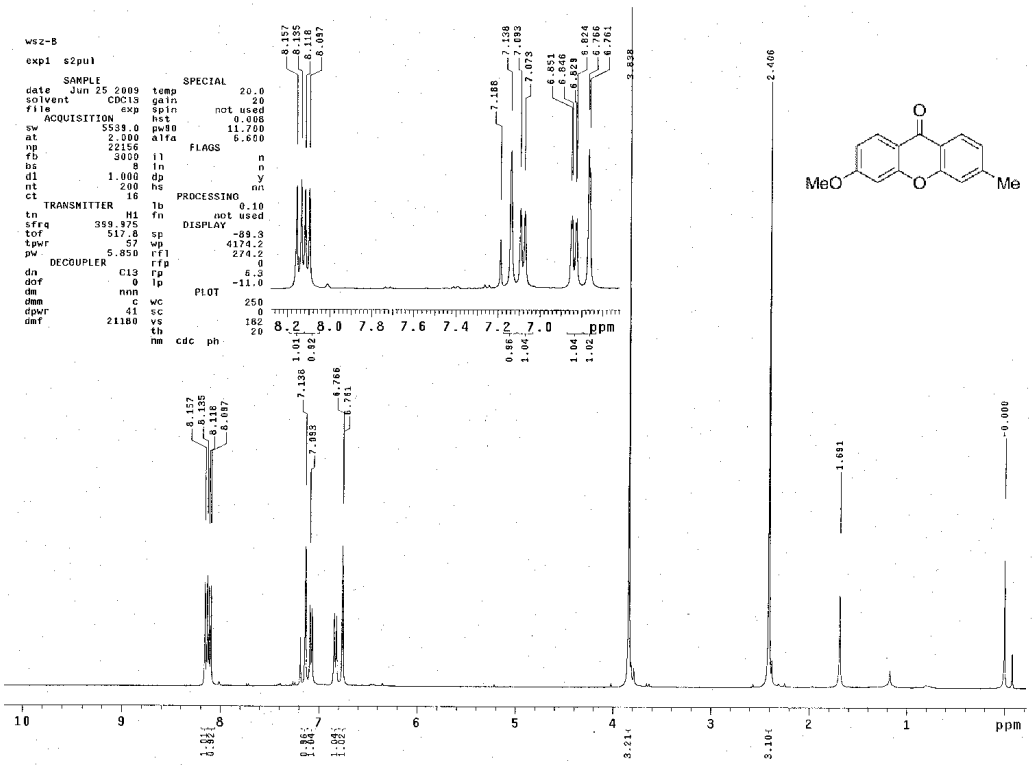
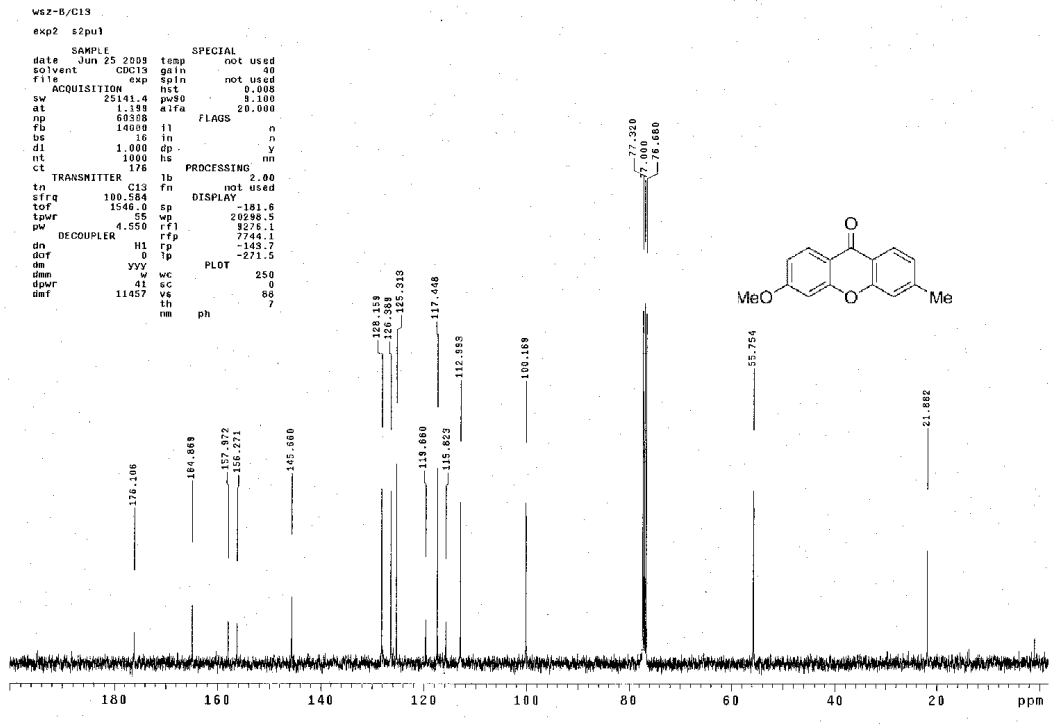
rfl 707.7
rfp 0
rp -6.3
lp -10.9
PLOT
wc 250
sc 0
vs 40
th 16
nm cdc ph
8.255
8.252
8.236
7.622
7.604
7.601
7.397
7.376
7.299
7.280
7.262
0.009
10
9
8
7
6
5
4
3
2
1
ppm
1.87
2.00
1.96
2.04

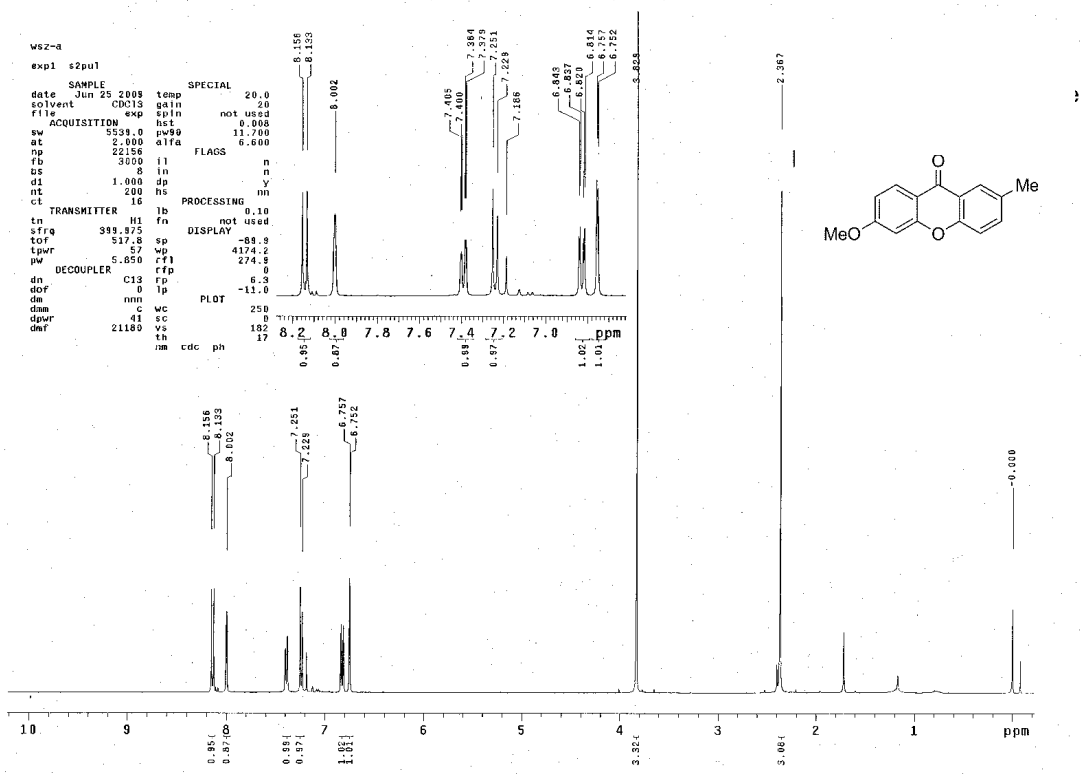

wsz/C13
exp2 s2pul
SAMPLE
date Apr 28 2009
solvent CDCl3
file exp
ACQUISITION
sw 25141.4
at 1.195
np 60308
fb 14000
bs 16
d1 1.000
nt 600
ct 80
TRANSMITTER
tn C13
sfrq 100.584
tof 1546.0
tpwr 55
pw 4.550
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 41
dmf 11457
SPECIAL
temp not used
gain 20
spin not used
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 -619.2
wp 21315.9
rfl 1634.3
rfp 0
rp -163.9
lp -271.5
PLOT
wc 250
sc 0
vs 100
th 6
nm ph
176.150
155.102
133.764
125.562
122.647
120.780
116.927
76.342
76.021
75.701
0.000
200
180
160
140
120
100
80
60
40
20
ppm

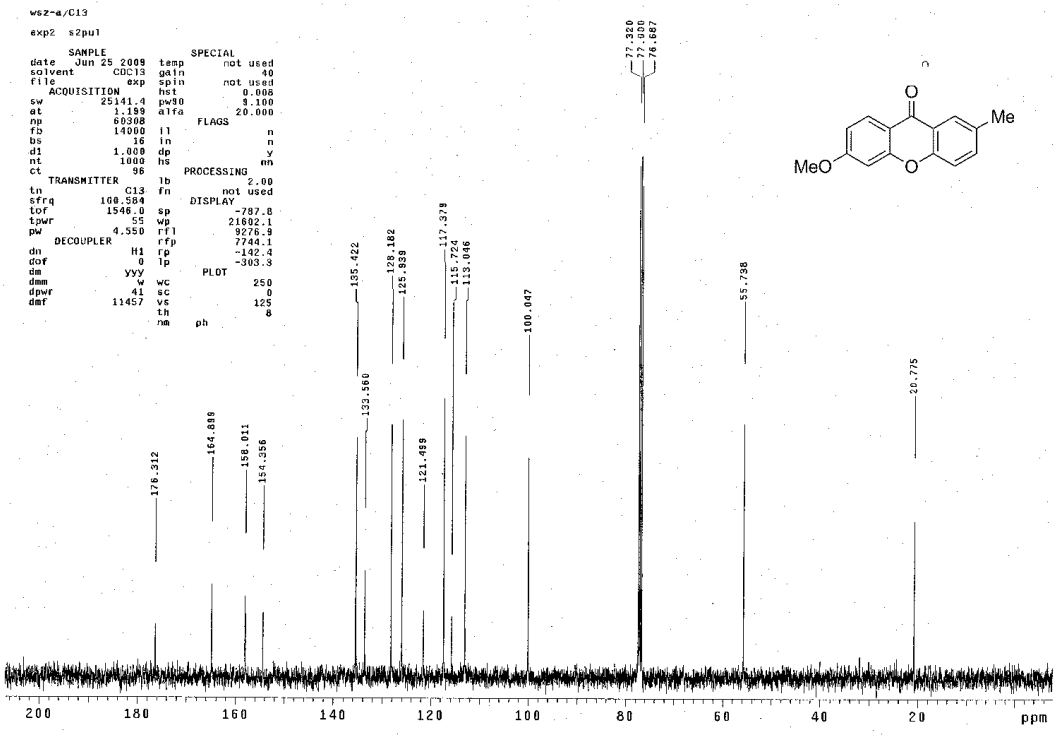

wsz-a
exp1 s2pul
SAMPLE
date May 8 2008
solvent CDCl3
file /export/home/~
yzy/tz-9/wsz-0508a~
.fid
ACQUISITION
sw 6020.5
at 1.800
np 21674
fb 3000
bs 8
d1 1.000
nt 1
ct 1
TRANSMITTER
tn H1
sfrq 399.975
tof 458.4
tpwr 57
pw 5.850
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 41
dmf 21180
SPECIAL
temp not used
gain 20
spin not used
hst 0.008
pw90 11.700
alfa 5.600
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -71.7
wp 4241.6
rfl 573.6
rfp 0
rp -4.3
lp -4.6
PLOT
wc 250
sc 0
vs 189
th 21
nm cdc ph
O
O
OMe
8.261
8.257
8.241
8.237
7.822
7.818
7.803
7.799
7.657
7.652
7.640
7.636
7.623
7.619
7.556
7.555
7.318
7.315
7.287
7.260
7.227
7.217
7.198
7.190
7.178
7.176
7.146
7.145
7.126
7.123
8.3
8.1
7.9
7.7
7.5
7.3
ppm
3.932
0.000
10
9
8
7
6
5
4
3
2
1
ppm

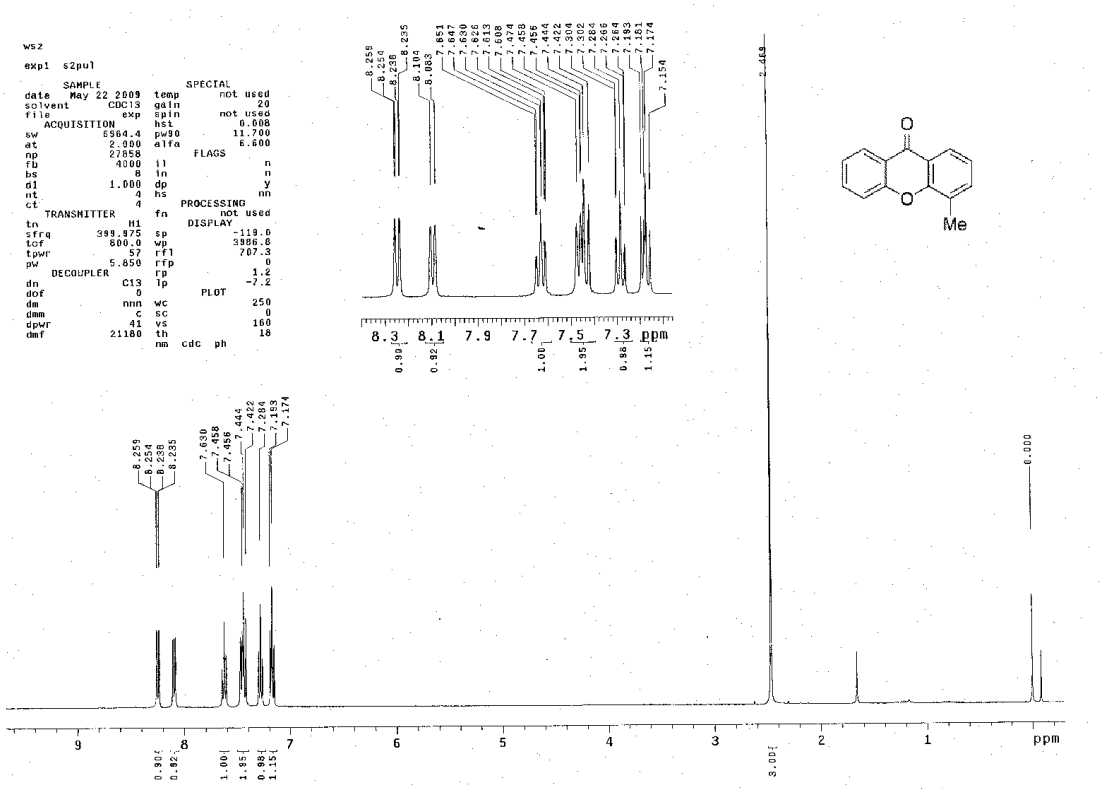

wsz-c/C13
exp2 s2pul
SAMPLE
date May 11 2009
solvent CDCl3
file exp
ACQUISITION
sw 25141.4
at 1.193
np 60308
fb 14000
bs 16
d1 1.000
nt 10000
ct 96
TRANSMITTER
tn C13
sfrq 100.584
tof 1546.0
tpwr 55
pw 4.550
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 41
dmf 11457
SPECIAL
temp not used
gain 40
spin not used
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 3090.7
wp 16382.4
rfl 9280.7
rfp 7744.1
rp -131.3
lp -293.7
PLOT
wc 250
sc 0
vs 73
th 6
nm ph
O
O
OMe
177.057
155.844
148.528
146.430
134.659
126.519
123.971
123.330
122.605
121.575
118.160
117.478
115.259
77.313
77.000
76.680
56.311
180
160
140
120
100
80
60
40
ppm

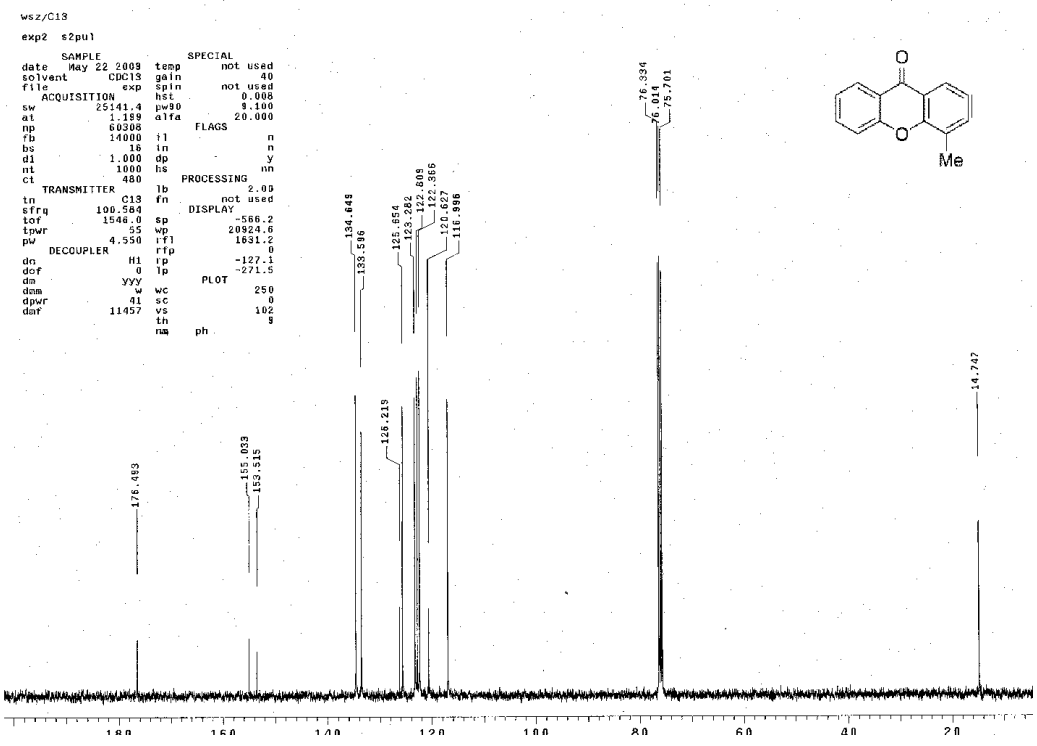

wsz-B
exp1 s2pul
SAMPLE
date Jun 25 2009
solvent CDCl3
file exp
ACQUISITION
sw 5539.0
at 2.000
np 22156
fb 3000
bs 8
d1 1.000
nt 200
ct 16
TRANSMITTER
tn H1
sfrq 399.975
tof 517.8
tpwr 57
pw 5.850
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 41
dmf 21180
SPECIAL
temp 20.0
gain 20
spin not used
hst 0.008
pw90 11.700
alfa 5.600
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.10
fn not used
DISPLAY
sp -89.3
wp 4174.2
rfl 274.2
rfp 0
rp 6.3
lp -11.0
PLOT
wc 250
sc 0
vs 182
th 20
nm cdc ph
8.157
8.135
8.118
8.087
7.138
7.093
7.073
7.188
6.851
6.840
6.824
6.766
6.761
3.838
2.466
1.681
0.000
MeO
Me
ppm

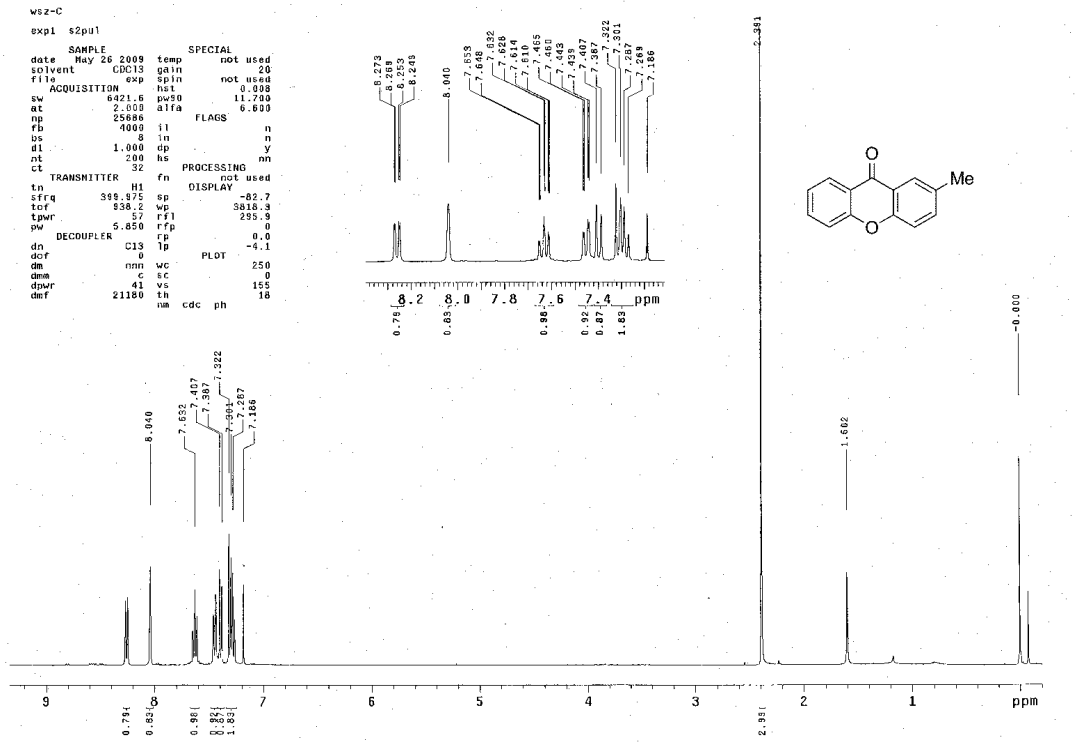

wsz-B/C13
exp2 s2pul
SAMPLE
date Jun 25 2009
solvent CDCl3
file exp
ACQUISITION
sw 25141.4
at 1.198
np 60308
fb 14000
bs 16
d1 1.000
nt 1000
ct 176
TRANSMITTER
tn C13
sfrq 100.584
tof 1548.0
tpwr 55
pw 4.550
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 41
dmf 11457
SPECIAL
temp not used
gain 40
spin not used
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 -181.6
wp 20298.5
rfl 9276.1
rfp 7744.1
rp -143.7
lp -271.5
PLOT
wc 250
sc 0
vs 88
th 7
nm ph
77.320
77.000
76.680
128.159
126.389
125.313
117.448
112.988
100.169
55.754
21.882
176.106
164.869
157.972
156.271
145.660
119.660
115.829
MeO
Me
ppm

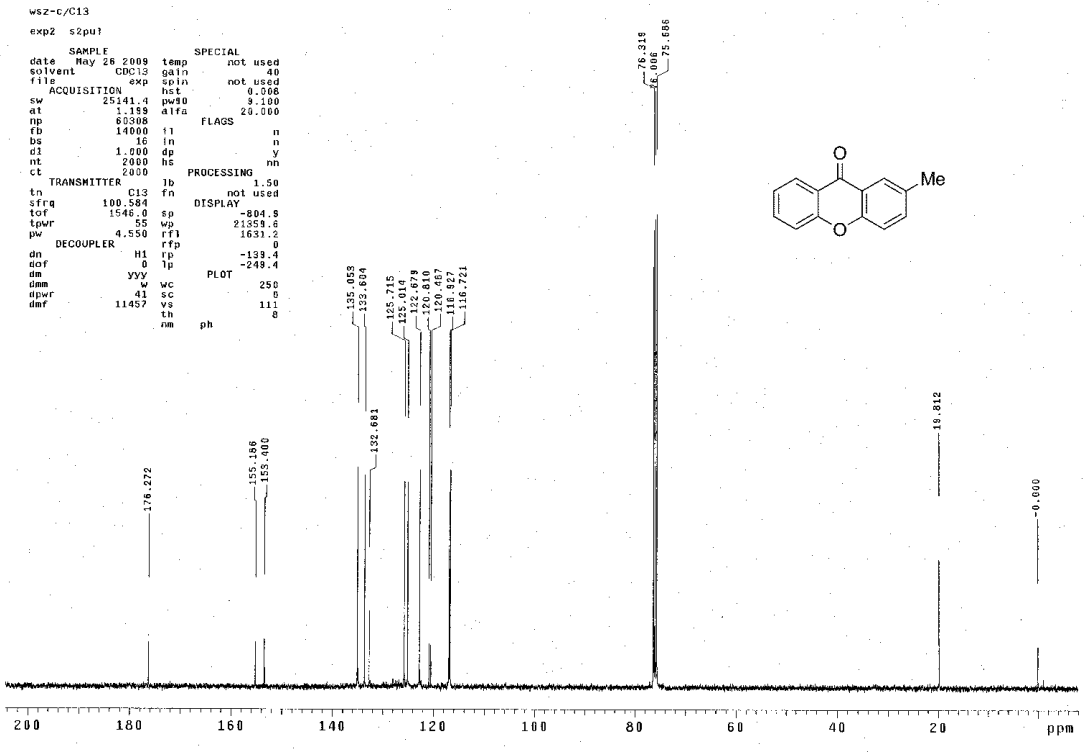

wsz-a
exp1 s2pul
SAMPLE
date Jun 25 2009
solvent CDCl3
file exp
ACQUISITION
sw 5539.0
at 2.000
np 22156
fb 3000
bs 8
d1 1.000
nt 200
ct 16
TRANSMITTER
tn H1
sfrq 399.975
tof 517.8
tpwr 57
pw 5.850
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 41
dmf 21180
SPECIAL
temp 20.0
gain 20
spin not used
hst 0.008
pw90 11.700
alfa 6.600
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.10
fn not used
DISPLAY
sp -88.9
wp 4174.2
rfl 274.8
rfp 0
rp 6.3
lp -11.0
PLOT
wc 250
sc 0
vs 182
th 17
nm cdc ph
8.156
8.139
8.002
7.405
7.400
7.384
7.379
7.251
7.229
7.186
6.843
6.837
6.820
6.814
6.757
6.752
3.824
2.367
-0.000
MeO
O
Me
ppm

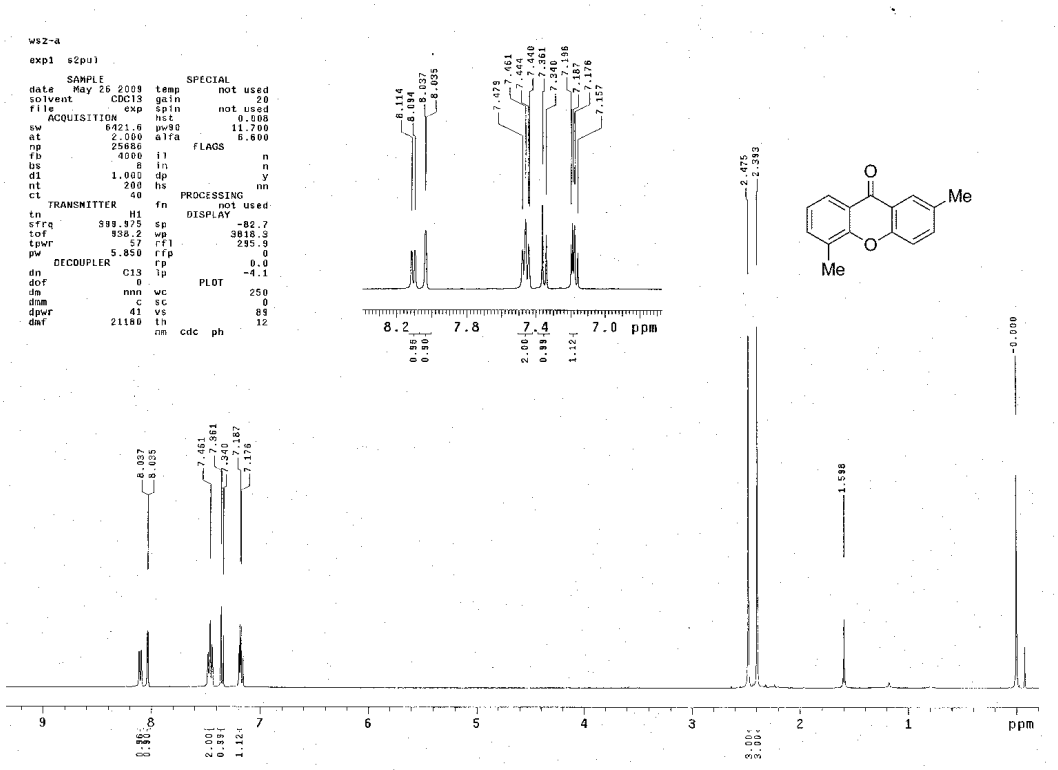

wsz-a/C13
exp2 s2pul
SAMPLE
date Jun 25 2009
solvent CDCl3
file exp
ACQUISITION
sw 25141.4
at 1.199
np 60308
fb 14000
bs 16
d1 1.000
nt 1000
ct 96
TRANSMITTER
tn C13
sfrq 100.584
tof 1546.0
tpwr 55
pw 4.550
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 41
dmf 11457
SPECIAL
temp not used
gain 40
spin not used
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 -787.8
wp 21602.1
rfl 9276.9
rfp 7744.1
rp -142.4
lp -303.3
PLOT
wc 250
sc 0
vs 125
th 8
nm ph
77.320
77.000
76.687
176.312
164.089
158.011
154.356
135.422
133.560
128.182
125.839
121.489
117.379
115.724
113.046
100.047
55.798
20.775
MeO
O
Me
ppm

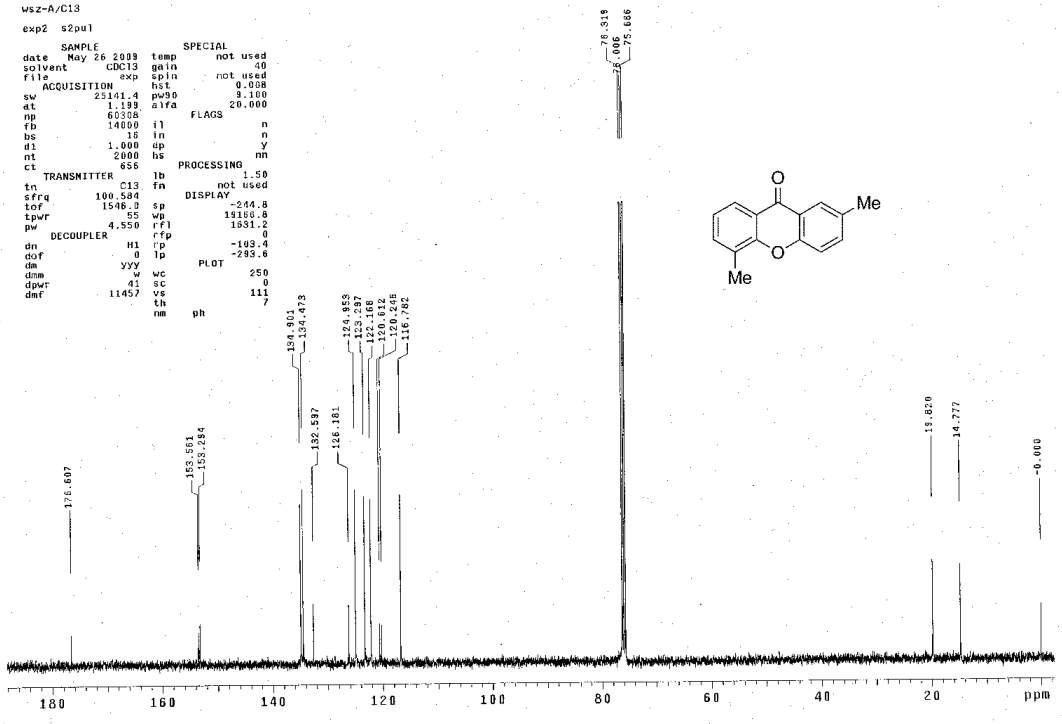

wsz
exp1 s2pul
SAMPLE
date May 22 2009
solvent CDCl3
file exp
ACQUISITION
sw 6564.4
at 2.000
np 27858
fb 4000
bs 8
d1 1.000
nt 4
ct 4
TRANSMITTER
tn H1
sfrq 399.875
tof 800.0
tpwr 57
pw 5.850
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 41
dmf 21180
SPECIAL
temp not used
gain 20
spin not used
hst 0.008
pw90 11.700
alfa 6.600
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -118.0
wp 5986.8
rfl 707.3
rfp 0
rp 1.2
lp -7.2
PLOT
wc 250
sc 0
vs 160
th 18
nm cdc ph
8.259
8.254
8.236
8.235
8.104
8.083
7.651
7.647
7.630
7.626
7.613
7.608
7.474
7.458
7.456
7.444
7.422
7.304
7.302
7.286
7.266
7.264
7.183
7.161
7.174
7.154
2.469
6.000
Me
8.3 8.1 7.9 7.7 7.5 7.3 ppm
0.90 0.82 1.00 1.95 0.98 1.15
ppm
3.00

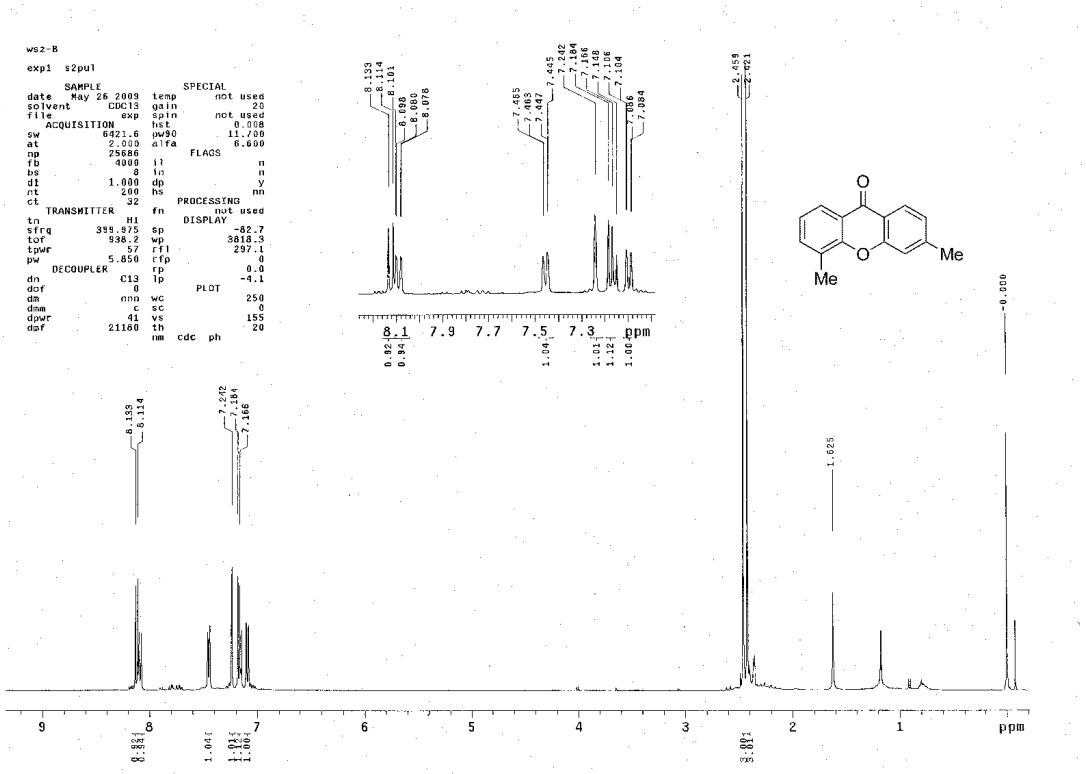

wsz/C13
exp2 s2pul
SAMPLE
date May 22 2009
solvent CDCl3
file exp
ACQUISITION
sw 25141.4
at 1.189
np 60308
fb 14000
bs 16
d1 1.000
nt 1000
ct 480
TRANSMITTER
tn C13
sfrq 100.584
tof 1548.0
tpwr 55
pw 4.550
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 41
dmf 11457
SPECIAL
temp not used
gain 40
spin not used
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 -566.2
wp 20924.6
rfl 1631.2
rfp 0
rp -127.1
lp -271.5
PLOT
wc 250
sc 0
vs 102
th 9
nm ph
176.493
155.053
153.515
134.648
133.596
126.219
125.654
123.282
122.809
122.366
120.627
116.996
76.334
76.014
75.701
14.747
Me

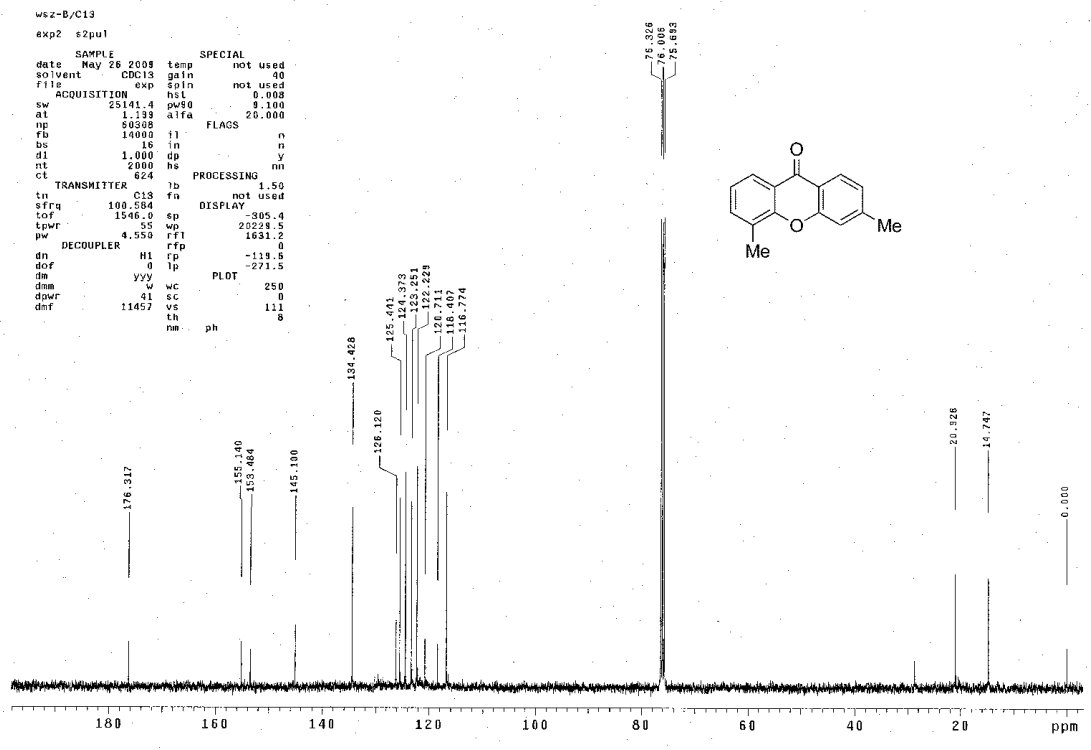

wsz-C
exp1 s2pul
SAMPLE
date May 26 2009
solvent CDCl3
file exp
ACQUISITION
sw 6421.6
at 2.008
np 25686
fb 4000
bs 8
d1 1.000
nt 200
ct 32
TRANSMITTER
tn H1
sfrq 399.975
tof 838.2
tpwr 57
pw 5.850
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 41
dmf 21180
SPECIAL
temp not used
gain 20
spin not used
hst 0.008
pw90 11.700
alfa 0.600
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -82.7
wp 3818.3
rfl 295.9
rfp 0
rp 0.0
lp -4.1
PLOT
wc 250
sc 0
vs 155
th 18
nm cdc ph
8.273
8.259
8.253
8.249
8.040
7.653
7.648
7.632
7.628
7.614
7.610
7.465
7.460
7.448
7.443
7.428
7.407
7.387
7.322
7.301
7.287
7.269
7.186
2.381
1.662
0.000
Me
8.2
8.0
7.8
7.6
7.4
ppm
0.79
0.83
0.98
0.92
0.87
1.83
9
8
7
6
5
4
3
2
1
ppm
2.95

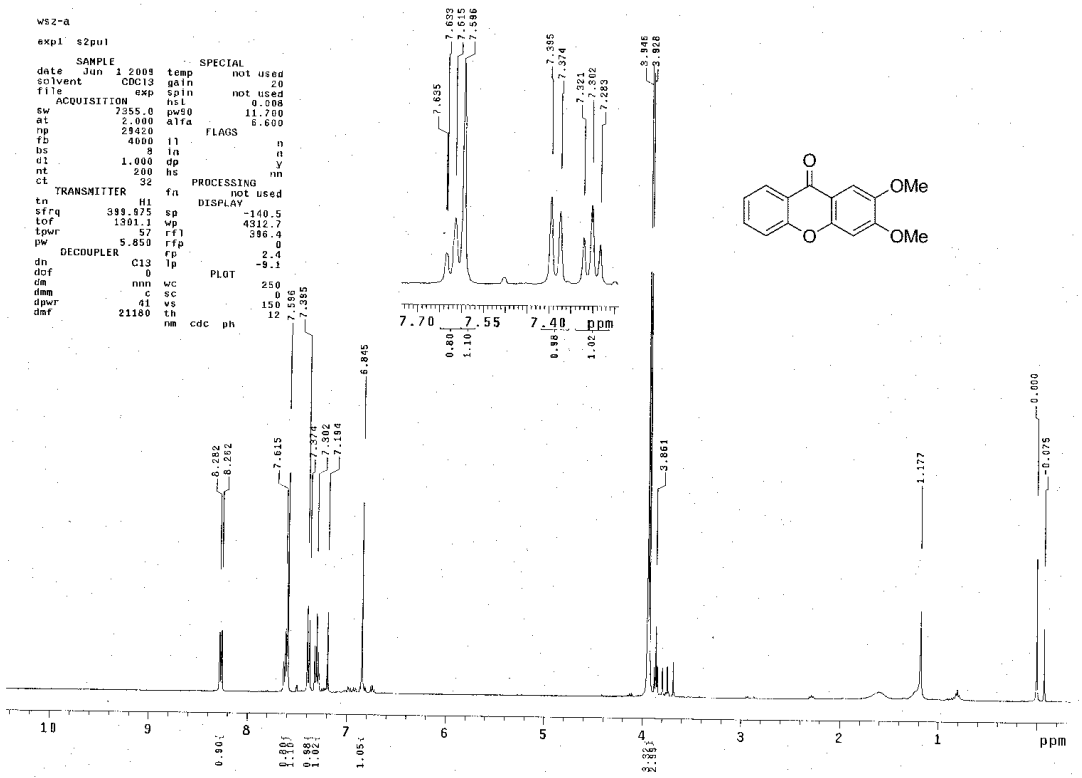

wsz-c/C13
exp2 s2pul
SAMPLE
date May 26 2009
solvent CDCl3
file exp
ACQUISITION
sw 25141.4
at 1.189
np 60308
fb 14000
bs 16
d1 1.000
nt 2000
ct 2000
TRANSMITTER
tn C13
sfrq 100.584
tof 1546.0
tpwr 55
pw 4.550
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 41
dmf 11457
SPECIAL
temp not used
gain 40
spin not used
hst 0.008
pw90 9.100
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.50
fn not used
DISPLAY
sp -804.8
wp 21359.6
rfl 1631.2
rfp 0
rp -138.4
lp -249.4
PLOT
wc 250
sc 0
vs 111
th 8
nm ph
176.272
155.186
153.400
135.053
133.604
132.681
125.715
125.014
122.679
120.810
120.467
116.927
116.721
76.319
76.006
75.686
19.812
0.000
Me
200
180
160
140
120
100
80
60
40
20
ppm

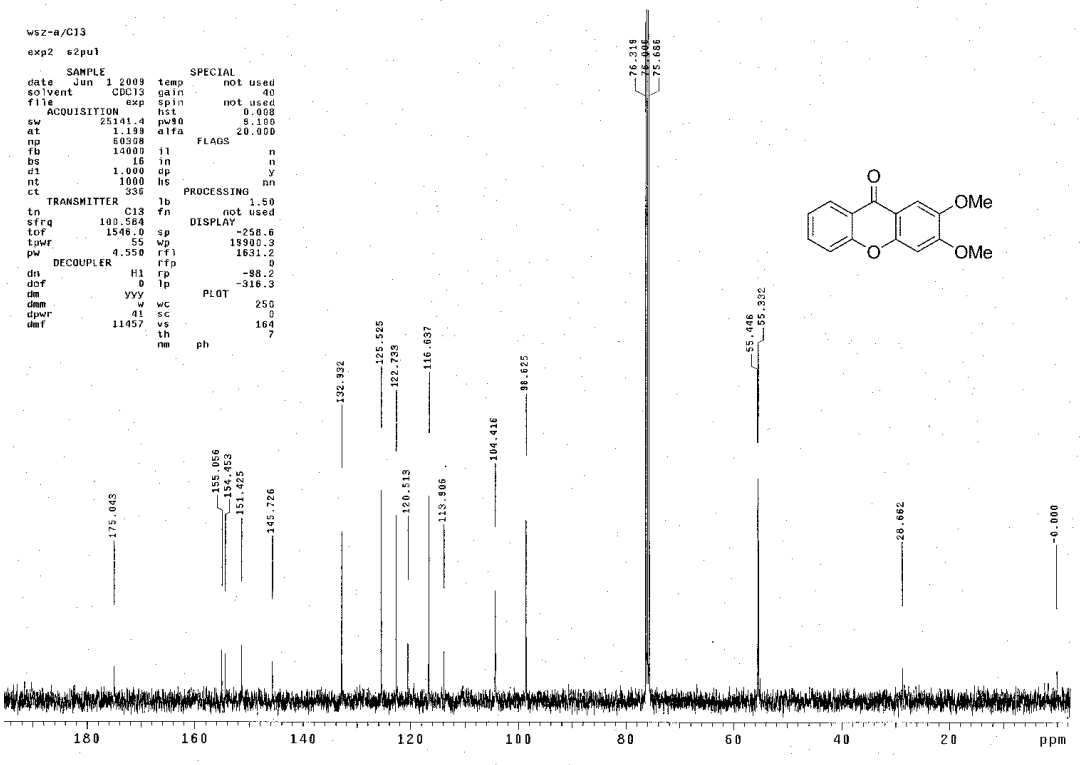

wsz-a
exp1 s2pul
SAMPLE
date May 26 2009
solvent CDCl3
file exp
ACQUISITION
sw 6421.6
at 2.000
np 25686
fb 4000
bs 8
d1 1.000
nt 200
ct 40
TRANSMITTER
tn H1
sfrq 399.975
tof 936.2
tpwr 57
pw 5.850
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 41
dmf 21180
SPECIAL
temp not used
gain 20
spin not used
hst 0.008
pw90 11.700
alfa 6.600
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -82.7
wp 3818.9
rfl 295.9
rfp 0
rp 0.0
lp -4.1
PLOT
wc 250
sc 0
vs 89
th 12
nm cdc ph
8.114
8.094
8.037
8.035
7.479
7.461
7.444
7.440
7.361
7.340
7.196
7.187
7.176
7.157
2.475
2.393
1.598
0.000
8.2
7.8
7.4
7.0 ppm
9
8
7
6
5
4
3
2
1
ppm
O
Me
O
Me

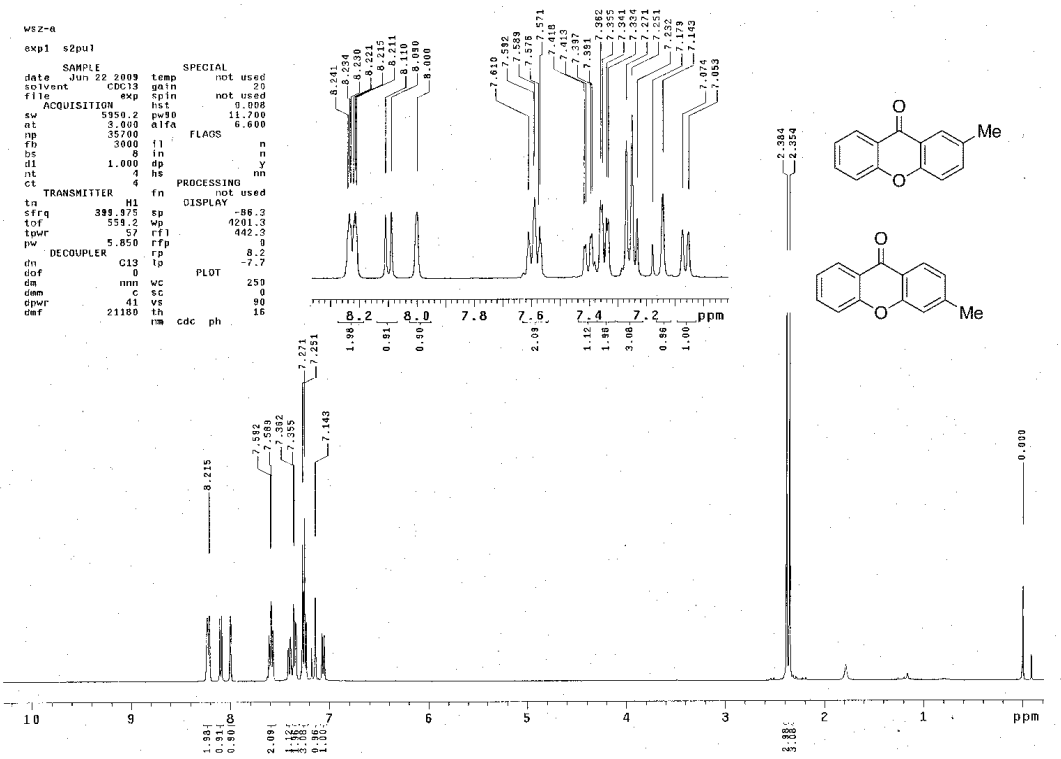

wsz-A/C13
exp2 s2pul
SAMPLE
date May 26 2009
solvent CDCl3
file exp
ACQUISITION
sw 25141.4
at 1.199
np 60308
fb 14000
bs 16
d1 1.000
nt 2000
ct 656
TRANSMITTER
tn C13
sfrq 100.584
tof 1546.0
tpwr 55
pw 4.550
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 41
dmf 11457
SPECIAL
temp not used
gain 40
spin not used
hst 0.008
pw90 9.100
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.50
fn not used
DISPLAY
sp -244.8
wp 19188.8
rfl 1831.2
rfp 0
rp -103.4
lp -293.6
PLOT
wc 250
sc 0
vs 111
th 7
nm ph
176.607
153.561
153.284
134.901
134.473
132.597
126.181
124.959
124.397
123.168
120.612
120.246
116.782
76.319
76.006
75.686
19.820
14.777
0.000
180
160
140
120
100
80
60
40
20
ppm
O
Me
O
Me

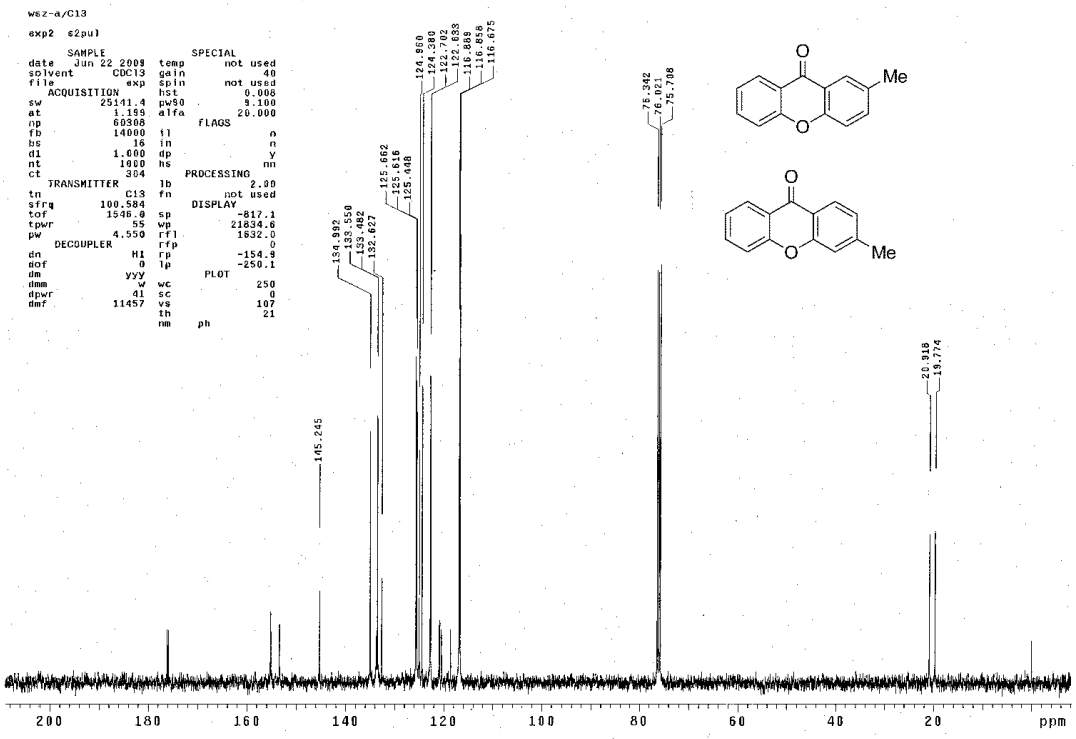

ws2-B
exp1 s2pul
SAMPLE
date May 26 2009
solvent CDCl3
file exp
ACQUISITION
sw 6421.6
at 2.000
np 25686
fb 4000
bs 8
d1 1.000
nt 200
ct 32
TRANSMITTER
tn H1
sfrq 399.975
tof 938.2
tpwr 57
pw 5.850
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 41
dmf 21160
SPECIAL
temp not used
gain 20
spin not used
hst 0.008
pw90 11.700
alfa 6.600
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -82.7
wp 3818.9
rfl 297.1
rfp 0
rp 0.0
lp -4.1
PLOT
wc 250
sc 0
vs 155
th 20
nm cdc ph
8.139
8.114
8.101
8.098
8.080
8.078
7.465
7.463
7.447
7.445
7.242
7.184
7.166
7.108
7.106
7.104
7.086
7.084
2.459
2.421
1.625
0.000
O
O
Me
Me
8.1
7.9
7.7
7.5
7.3
ppm
9
8
7
6
5
4
3
2
1
ppm

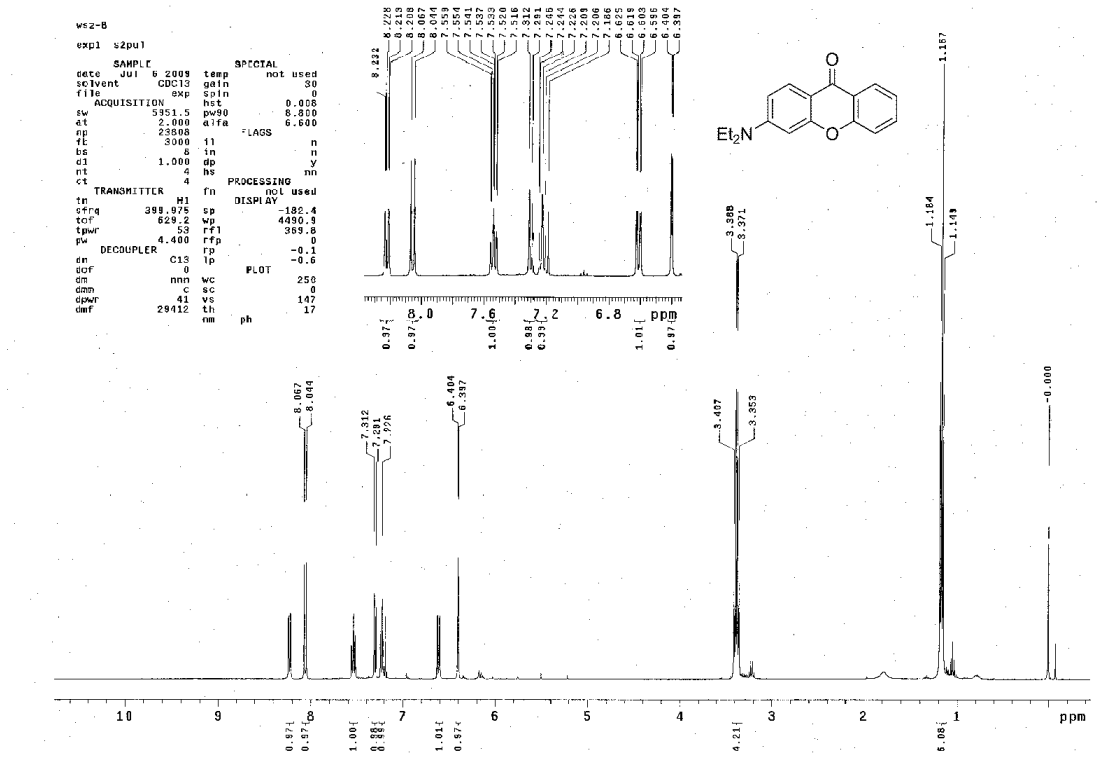

wsz-B/C13
exp2 s2pul
SAMPLE
date May 26 2009
solvent CDCl3
file exp
ACQUISITION
sw 25141.4
at 1.199
np 60308
fb 14000
bs 16
d1 1.000
nt 2000
ct 624
TRANSMITTER
tn C13
sfrq 100.584
tof 1546.0
tpwr 55
pw 4.550
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 41
dmf 11457
SPECIAL
temp not used
gain 40
spin not used
hst 0.008
pw90 9.100
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.50
fn not used
DISPLAY
sp -305.4
wp 20228.5
rfl 1631.2
rfp 0
rp -119.5
lp -271.5
PLOT
wc 250
sc 0
vs 111
th 8
nm ph
176.317
156.140
153.484
145.100
134.428
126.120
125.401
124.373
123.251
122.229
120.711
118.407
116.774
77.326
77.005
76.683
20.926
14.747
0.000
O
O
Me
Me
180
160
140
120
100
80
60
40
20
ppm

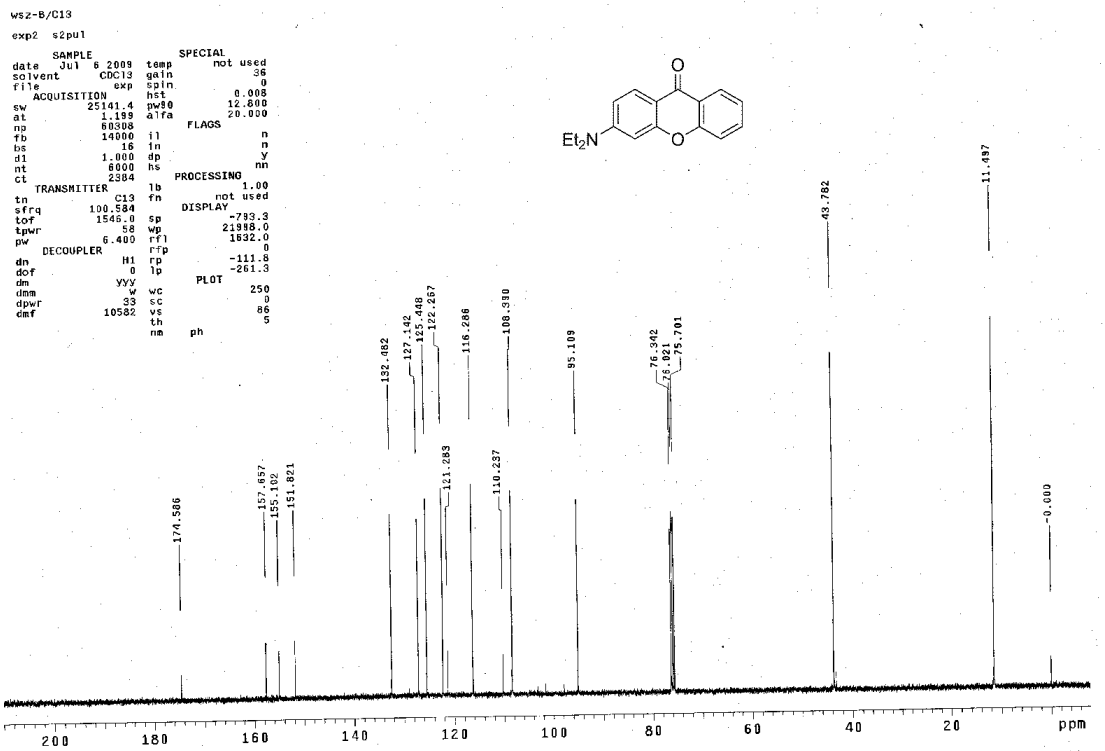

wsz-a
exp1 s2pul
SAMPLE
date Jun 1 2009
solvent CDCl3
file exp
ACQUISITION
sw 7355.8
at 2.000
np 29420
fb 4000
bs 8
d1 1.000
nt 200
ct 32
TRANSMITTER
tn H1
sfrq 399.975
tof 1301.1
tpwr 57
pw 5.850
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 41
dmf 21180
SPECIAL
temp not used
gain 20
spin not used
hst 0.008
pw90 11.700
alfa 6.600
FLAGS
PROCESSING
fn not used
DISPLAY
sp -140.5
wp 4312.7
rfl 396.4
rfp 0
rp 2.4
lp -9.1
PLOT
wc 250
sc 0
vs 150
th 12
nm cdc ph
7.70 7.55 7.40 ppm
OMe
OMe
ppm

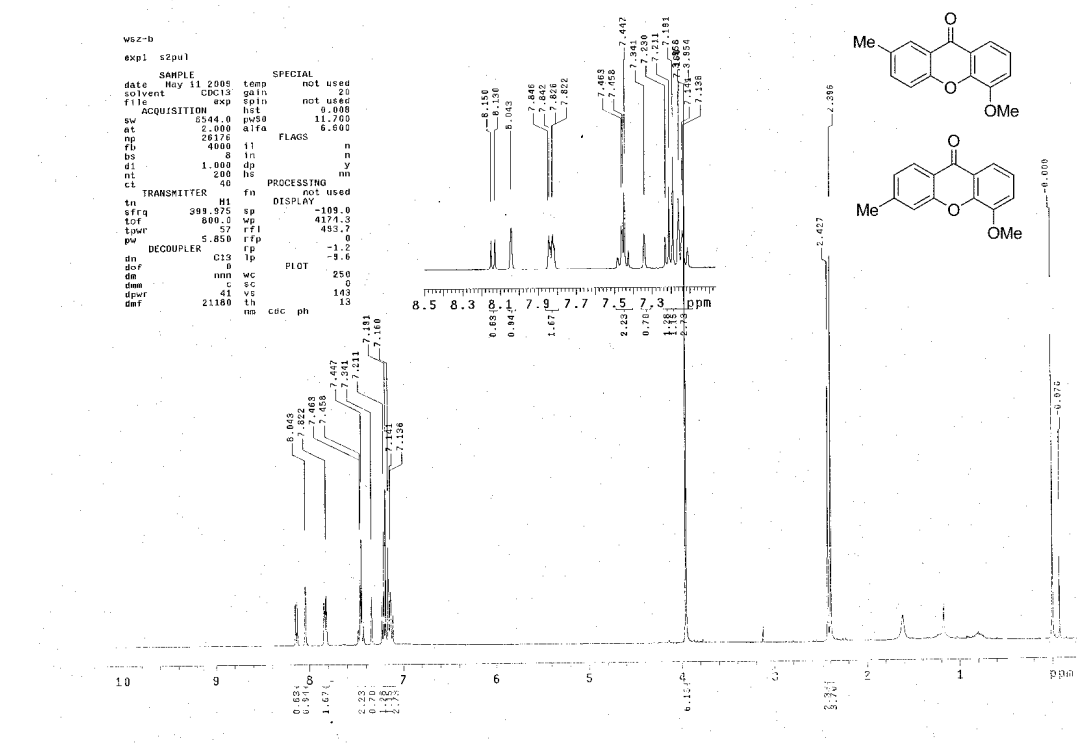

wsz-a/C13
exp2 s2pul
SAMPLE
date Jun 1 2009
solvent CDCl3
file exp
ACQUISITION
sw 25141.4
at 1.199
np 60308
fb 14000
bs 16
d1 1.000
nt 1000
ct 338
TRANSMITTER
tn C13
sfrq 100.584
tof 1546.0
tpwr 55
pw 4.550
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 41
dmf 11457
SPECIAL
temp not used
gain 40
spin not used
hst 0.008
pw90 5.100
alfa 20.000
FLAGS
PROCESSING
lb 1.50
fn not used
DISPLAY
sp -258.6
wp 19900.3
rfl 1631.2
rfp 0
rp -88.2
lp -316.3
PLOT
wc 250
sc 0
vs 164
th 7
nm ph
OMe
OMe
ppm

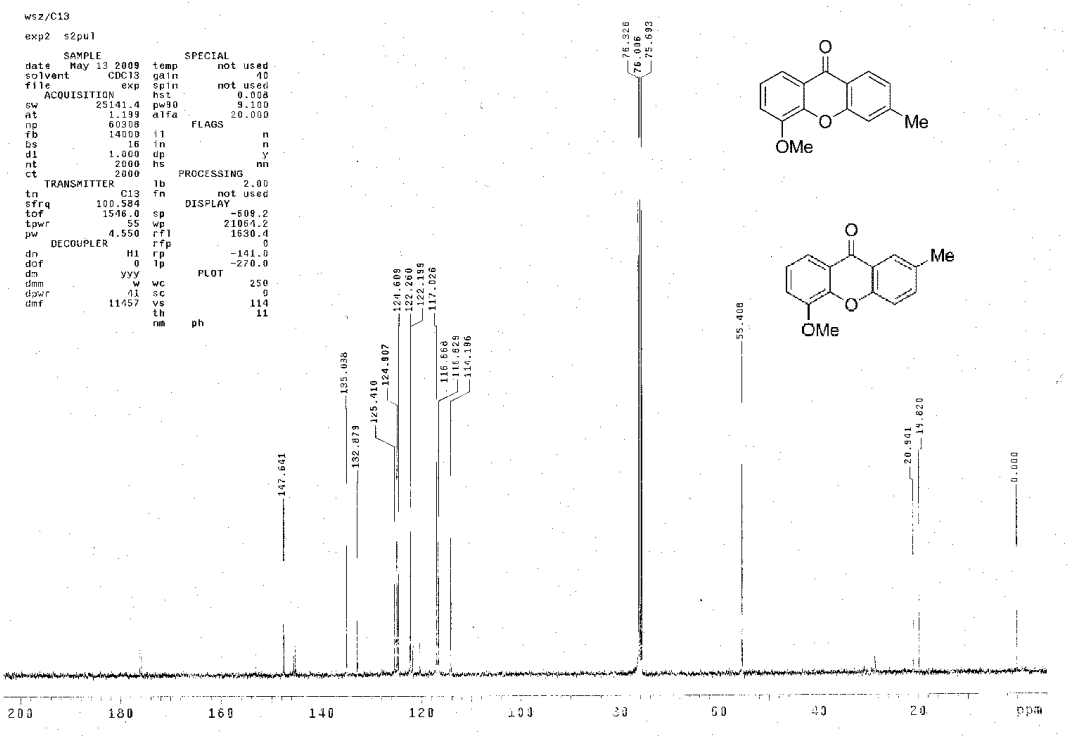

wsz-a
exp1 s2pul
SAMPLE
date Jun 22 2009
solvent CDCl3
file exp
ACQUISITION
sw 5950.2
at 3.000
np 35700
fb 3000
bs 8
d1 1.000
nt 4
ct 4
TRANSMITTER
tn H1
sfrq 399.975
tof 559.2
tpwr 57
pw 5.850
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 41
dmf 21180
SPECIAL
temp not used
gain 20
spin not used
hst 0.008
pw90 11.700
alfa 6.600
FLAGS
fl n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -86.3
wp 4201.3
rfl 442.3
rfp 0
rp 8.2
lp -7.7
PLOT
wc 250
sc 0
vs 90
th 16
nm cdc ph
8.241
8.234
8.230
8.221
8.215
8.211
8.110
8.090
8.000
7.610
7.592
7.589
7.575
7.571
7.418
7.413
7.382
7.381
7.355
7.341
7.334
7.251
7.232
7.179
7.143
7.074
7.053
2.384
2.354
0.000
Me
O
8.2
8.0
7.8
7.6
7.4
7.2
ppm
1.98
0.91
0.50
2.09
1.12
1.98
3.08
0.98
1.00
10
9
8
7
6
5
4
3
2
1
ppm

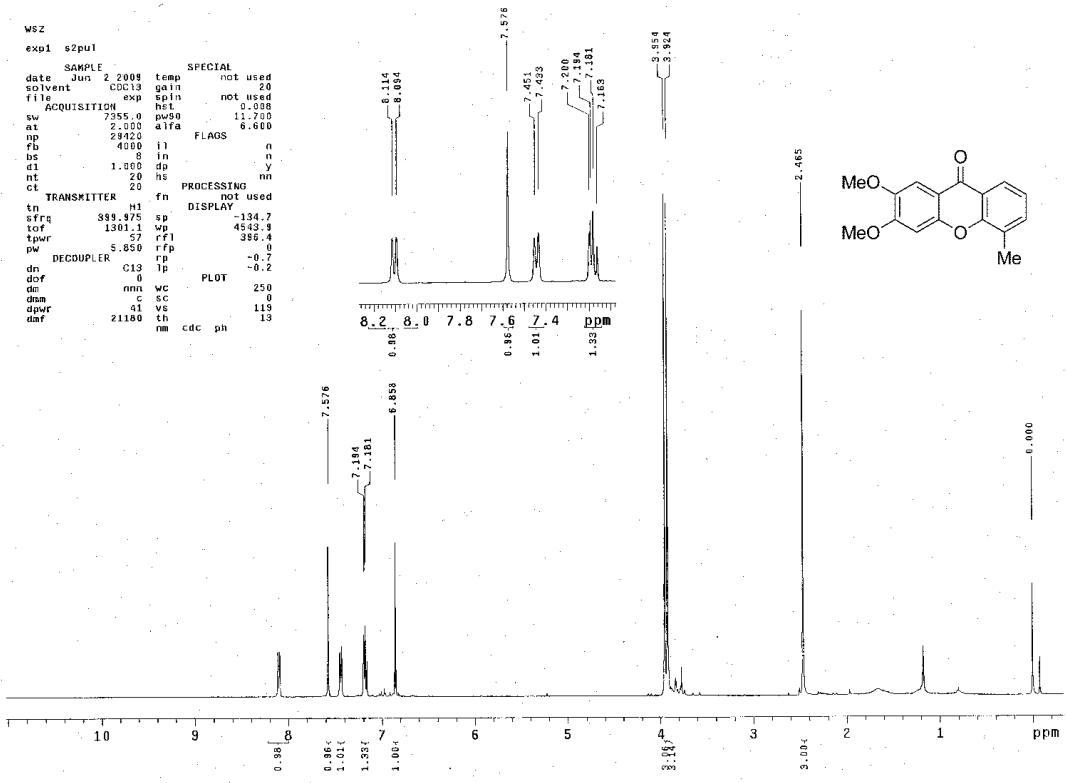

wsz-a/C13
exp2 s2pul
SAMPLE
date Jun 22 2009
solvent CDCl3
file exp
ACQUISITION
sw 25141.4
at 1.199
np 60308
fb 14000
bs 16
d1 1.000
nt 1000
ct 304
TRANSMITTER
tn C13
sfrq 100.584
tof 1546.0
tpwr 55
pw 4.550
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 41
dmf 11457
SPECIAL
temp not used
gain 40
spin not used
hst 0.008
pw90 9.100
alfa 20.000
FLAGS
fl n
in n
dp y
hs nn
PROCESSING
lb 2.00
fn not used
DISPLAY
sp -817.1
wp 21834.6
rfl 1632.0
rfp 0
rp -154.9
lp -250.1
PLOT
wc 250
sc 0
vs 107
th 21
nm ph
145.245
134.992
133.550
133.482
132.627
125.662
125.616
125.448
124.950
124.380
122.702
120.883
118.858
116.675
76.342
76.021
75.708
20.918
19.774
Me
O
200
180
160
140
120
100
80
60
40
20
ppm

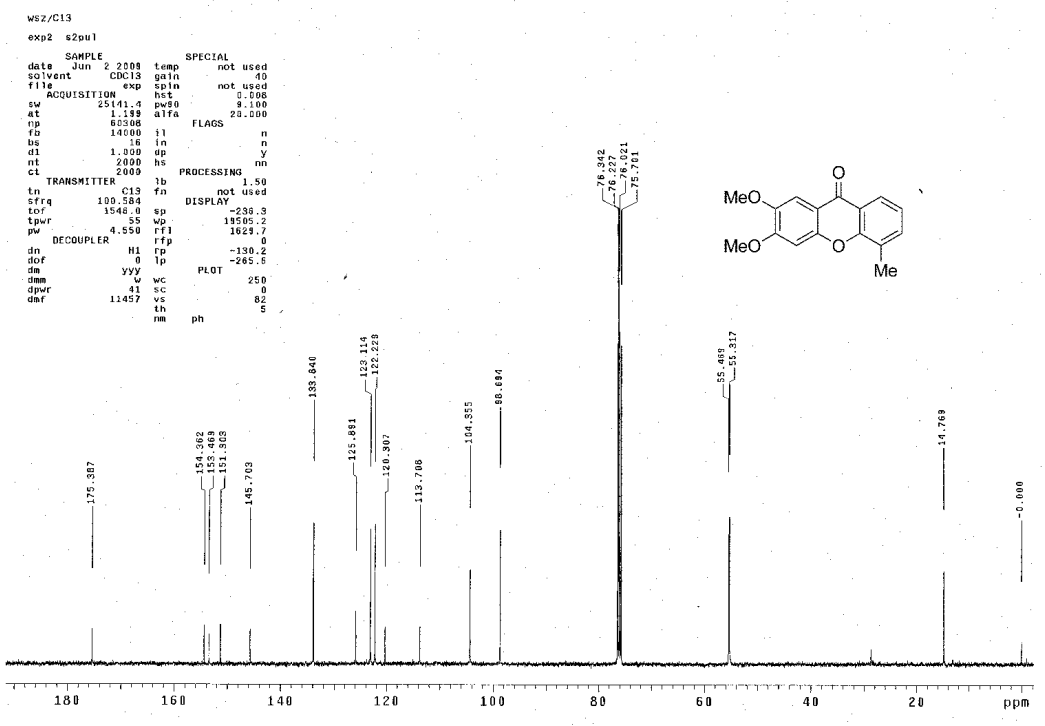

wsz-B
exp1 s2pul
SAMPLE
date Jul 6 2009
solvent CDCl3
file exp
ACQUISITION
sw 5951.5
at 2.000
np 23808
fb 3000
bs 8
d1 1.000
nt 4
ct 4
TRANSMITTER
tn H1
sfrq 399.975
tof 629.2
tpwr 53
pw 4.400
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 41
dmf 29412
SPECIAL
temp not used
gain 30
spin 0
hst 0.008
pw90 8.800
alfa 6.600
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -182.4
wp 4490.5
rfl 369.8
rfp 0
rp -0.1
lp -0.6
PLOT
wc 250
sc 0
vs 147
th 17
nm ph
Et2N
ppm

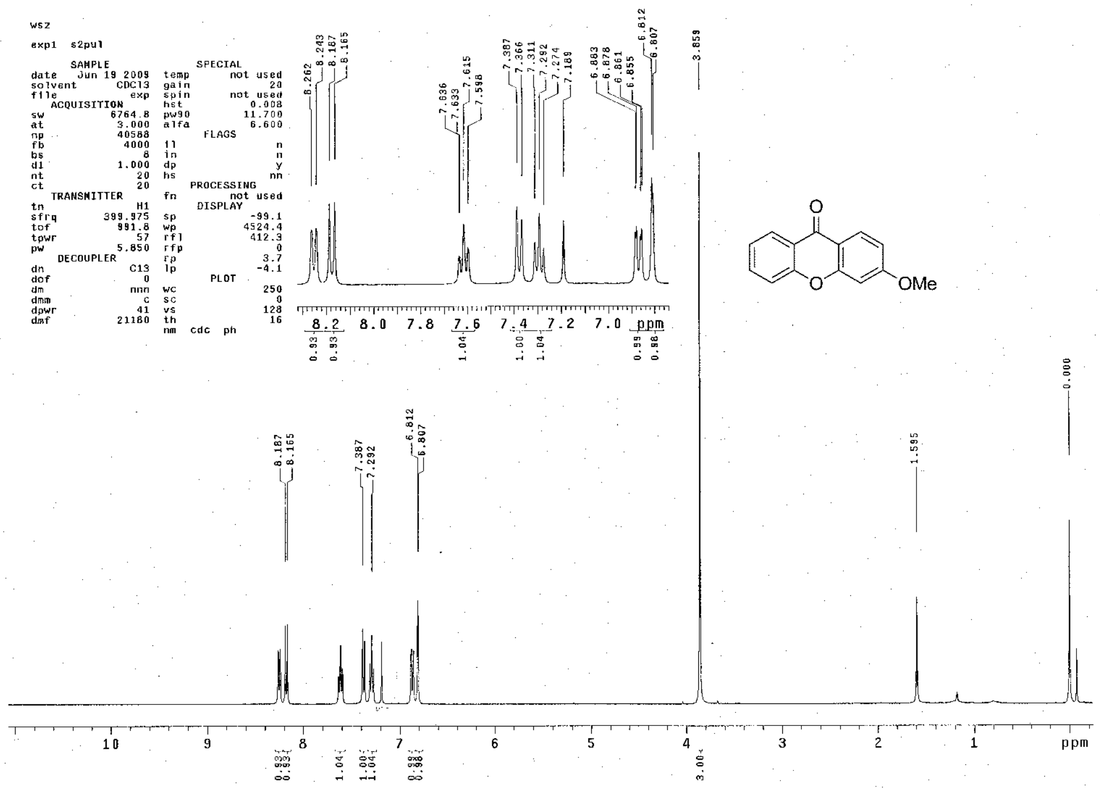

wsz-B/C13
exp2 s2pul
SAMPLE
date Jul 6 2009
solvent CDCl3
file exp
ACQUISITION
sw 25141.4
at 1.199
np 60308
fb 14000
bs 16
d1 1.000
nt 6000
ct 2384
TRANSMITTER
tn C13
sfrq 100.584
tof 1545.0
tpwr 58
pw 6.400
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 33
dmf 10582
SPECIAL
temp not used
gain 36
spin 0
hst 0.008
pw90 12.600
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn not used
DISPLAY
sp -783.3
wp 21988.0
rfl 1632.0
rfp 0
rp -111.8
lp -261.3
PLOT
wc 250
sc 0
vs 86
th 5
nm ph
Et2N
ppm

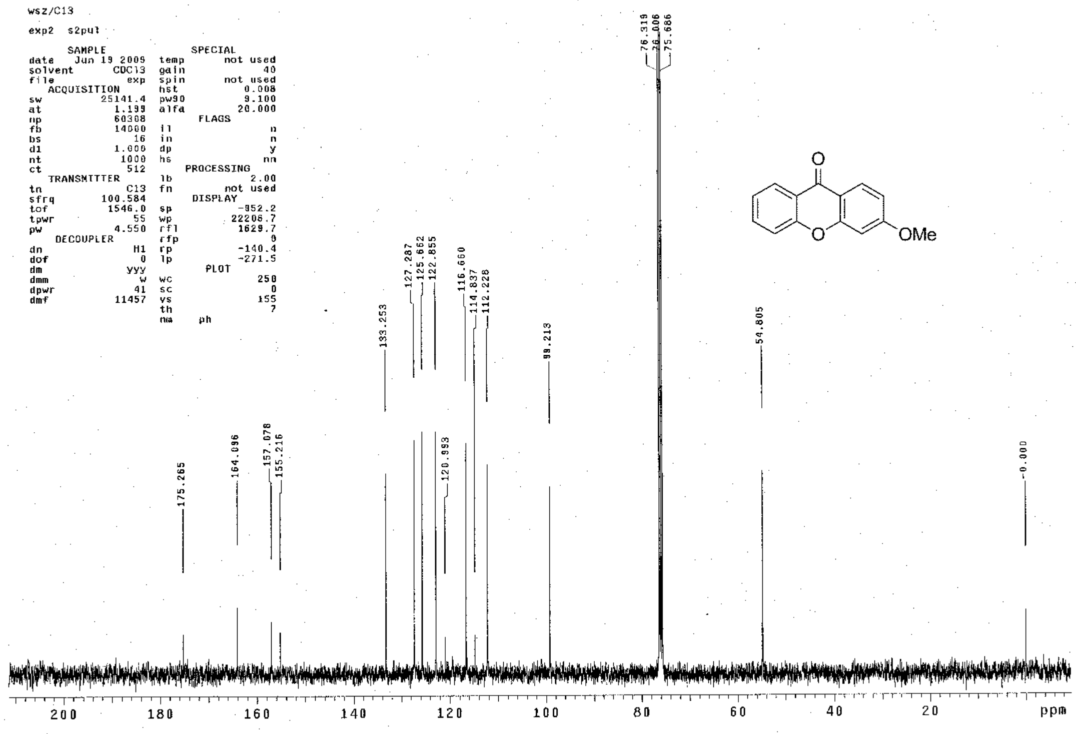

wsz-b
expl s2pul
SAMPLE
date May 11 2009
solvent CDCl3
file exp
ACQUISITION
sw 6544.0
at 2.000
np 26176
fb 4000
bs 8
d1 1.000
nt 200
ct 40
TRANSMITTER
tn H1
sfrq 399.975
tof 600.0
tpwr 57
pw 5.850
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 41
dmf 21180
SPECIAL
temp not used
gain 20
spin not used
hst 0.008
pw90 11.700
alfa 6.600
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -109.0
wp 4171.3
rfl 493.7
rfp 0
rp -1.2
lp -9.6
PLOT
wc 250
sc 0
vs 143
th 13
nm cdc ph
8.5 8.3 8.1 7.9 7.7 7.5 7.3 ppm
10 9 8 7 6 5 4 3 2 1 ppm
Me
OMe

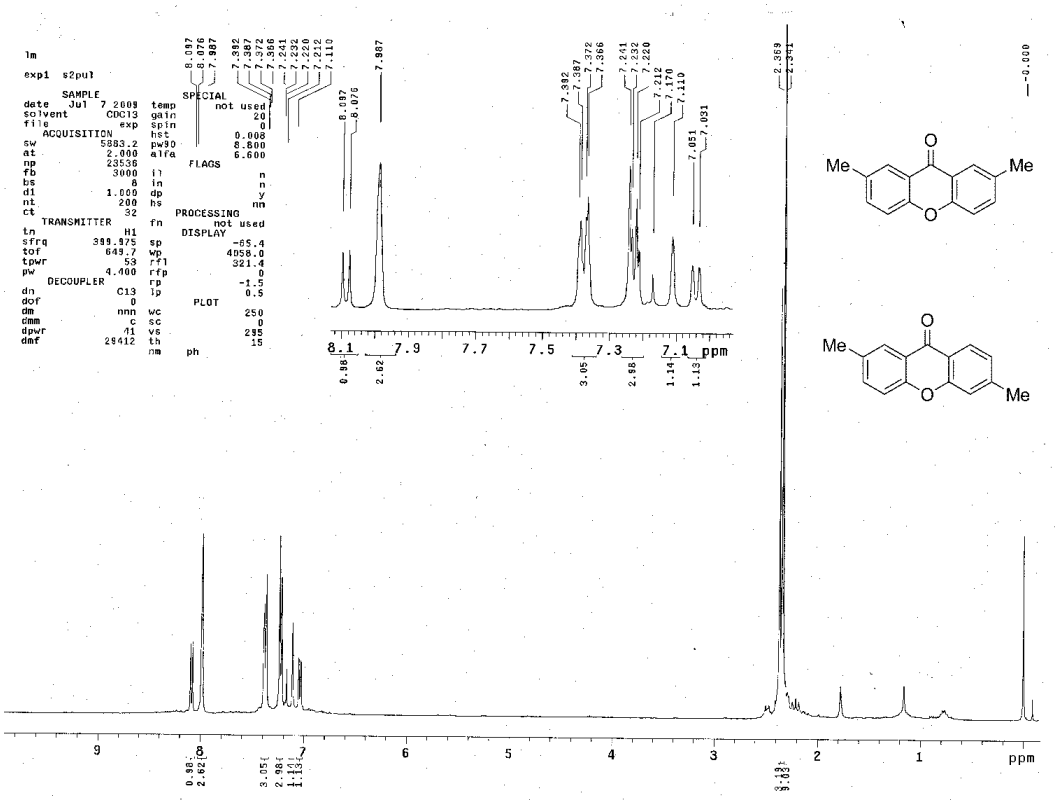

wsz/C13
exp2 s2pul
SAMPLE
date May 13 2009
solvent CDCl3
file exp
ACQUISITION
sw 25141.4
at 1.199
np 60308
fb 14000
bs 16
d1 1.000
nt 2000
ct 2000
TRANSMITTER
tn C13
sfrq 100.584
tof 1546.0
tpwr 55
pw 4.550
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 41
dmf 11457
SPECIAL
temp not used
gain 40
spin not used
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 -609.2
wp 21064.2
rfl 1630.4
rfp 0
rp -141.0
lp -270.0
PLOT
wc 250
sc 0
vs 114
th 11
nm ph
200 180 160 140 120 100 80 60 40 20 ppm
Me
OMe

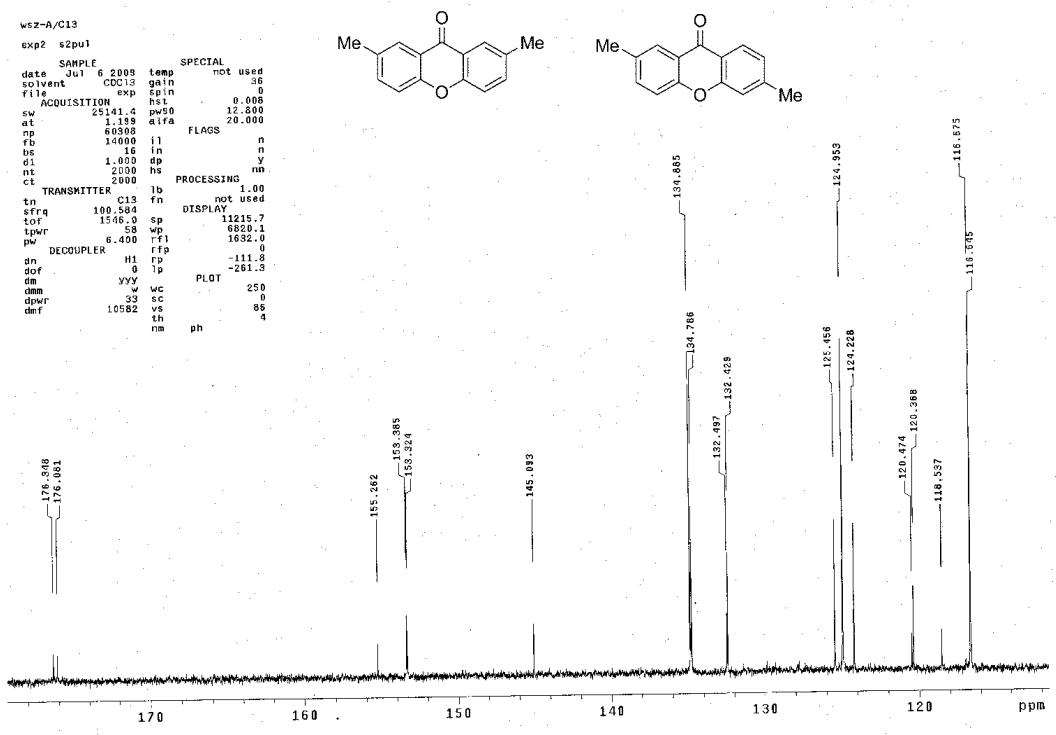

wsz
exp1 s2pul
SAMPLE
date Jun 2 2009
solvent CDCl3
file exp
ACQUISITION
sw 7355.0
at 2.000
np 28420
fb 4000
bs 8
d1 1.000
nt 20
ct 20
TRANSMITTER
tn H1
sfrq 399.975
tof 1301.1
tpwr 57
pw 5.850
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 41
dmf 21180
SPECIAL
temp not used
gain 20
spin not used
hst 0.008
pw90 11.700
alfa 6.600
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -134.7
wp 4543.9
rfl 386.4
rfp 0
rp -0.7
lp -0.2
PLOT
wc 250
sc 0
vs 119
th 13
nm cdc ph
8.114
8.094
7.576
7.451
7.433
7.200
7.194
7.181
7.163
3.954
3.924
2.465
0.000
MeO
MeO
O
Me
ppm

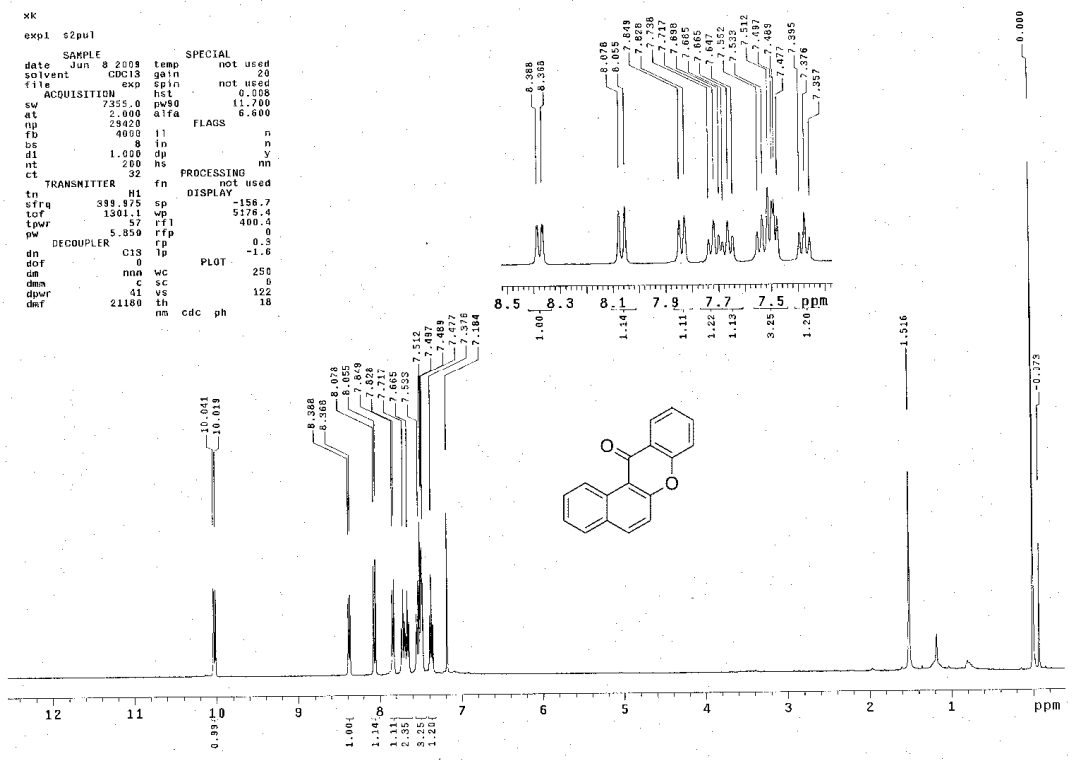

wsz/C13
exp2 s2pul
SAMPLE
date Jun 2 2009
solvent CDCl3
file exp
ACQUISITION
sw 25141.4
at 1.189
np 60308
fb 14000
bs 16
d1 1.000
nt 2000
ct 2000
TRANSMITTER
tn C13
sfrq 100.584
tof 1548.0
tpwr 55
pw 4.550
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 41
dmf 11457
SPECIAL
temp not used
gain 40
spin not used
hst 0.008
pw90 9.100
alfa 28.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.50
fn not used
DISPLAY
sp -236.3
wp 19505.2
rfl 1629.7
rfp 0
rp -130.2
lp -265.8
PLOT
wc 250
sc 0
vs 82
th 5
nm ph
78.342
76.227
76.021
75.761
133.840
123.114
122.228
125.891
120.307
104.555
88.684
55.469
55.317
154.362
153.468
151.968
145.703
113.706
175.387
14.769
-0.000
MeO
MeO
O
Me
ppm

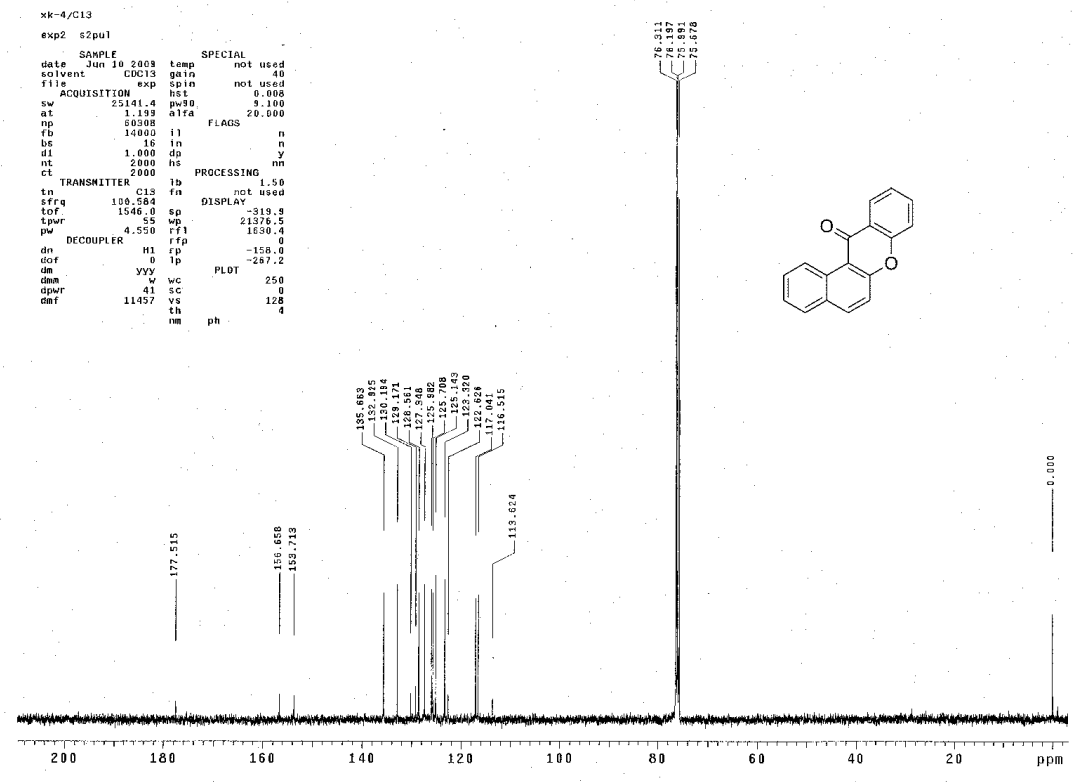

wsz
exp1 s2pul
OMe
3.859
1.595
0.000
ppm


wsz/C13
exp2 s2pul
OMe
175.265
164.096
157.078
155.216
133.253
127.287
125.662
122.855
120.993
116.660
114.837
112.228
99.213
54.805
-0.000
ppm


Me
Me
O
Me
Me
O
ppm


Me
Me
O
Me
Me
O
ppm


xk
expl s2pul
SAMPLE
date Jun 8 2009
solvent CDCl3
file exp
ACQUISITION
sw 7355.0
at 2.000
np 29420
fb 4000
bs 8
d1 1.000
nt 200
ct 32
TRANSMITTER
tn H1
sfrq 399.875
tof 1301.1
tpwr 57
pw 5.850
DECOUPLER
dn C13
dof 0
dm nna
dmm c
dpwr 41
dmf 21180
SPECIAL
temp not used
gain 20
spin not used
hst 0.008
pw90 11.700
alfa 6.600
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -156.7
wp 5176.4
rfl 400.4
rfp 0
rp 0.3
lp -1.6
PLOT
wc 250
sc 0
vs 122
th 18
nm cdc ph
ppm


xk-4/C13
exp2 s2pul
SAMPLE
date Jun 10 2009
solvent CDCl3
file exp
ACQUISITION
hst 0.008
sw 25141.4
pw90 9.100
at 1.199
alfa 20.000
np 60308
FLAGS
fb 14000
il n
bs 16
in n
d1 1.000
dp y
nt 2000
hs nn
ct 2000
PROCESSING
TRANSMITTER
lb 1.50
tn C13
fn not used
sfrq 100.584
DISPLAY
tof 1546.0
sp -319.8
tpwr 55
wp 21376.5
pw 4.550
rfl 1830.4
DECOUPLER
rfp 0
dn H1
rp -158.0
dof 0
lp -267.2
dm yyy
PLOT
dmm w
wc 250
dpwr 41
sc 0
dmf 11457
vs 128
th 4
nm ph
ppm