Transition Metal-Catalyzed Addition Reactions of Arylboronic Acids with Alkyl 2-Formylbenzoates: Efficient Access to Chiral 3-Substituted Phthalides

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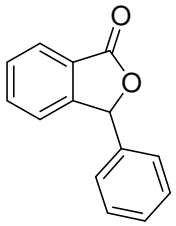
Supporting Information

General: NMR spectra were recorded on Varian 300 MHz or 600 MHz spectrometers. All yields reported refer to isolated yields unless otherwise indicated, and the product purity was estimated to be greater than 95% as determined by ^{1}H NMR. Compounds described in the literature were characterized by comparison of their ^{1}H NMR and ^{13}C NMR spectra to reported data. HPLC analyses were performed using an Agilent 1100 series HPLC system. Optical rotations were measured using a JASCO polarimeter DIP-370.

THF and Toluene were dried and freshly distilled with sodium under nitrogen prior to use. CH_2Cl_2 was dried and freshly distilled with calcium hydride under nitrogen prior to use. Arylboronic acids were obtained as gifts from Frontier Scientific, Inc. Alkyl formylbenzoates were prepared from reported methods.[1,2] Ligand **10a** was purchased from Strem Chemicals and used directly. Other ligands were prepared by following reported methods.[3-6] Other chemical reagents were purchased from Strem Chemicals Aldrich or Alpha Aesar and used directly.

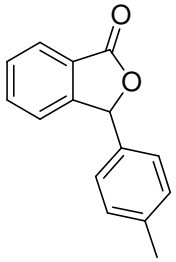
General procedure for palladacycle 1, 2 or platinacycle 3-catalyzed addition reactions of arylboronic acids with methyl 2-formylbenzoate: To a vial containing methyl 2-formylbenzoate (0.5 mmol), arylboronic acid (1.0 mmol), K_3PO_4 (1.5 mmol) and palladacycles **1**, **2** or platinacycle **3** (0.0025 mmol) was added toluene (1 ml). After the mixture was stirred for 12 hrs at room temperature (for palladacycle **1**, **2**-catalyzed reactions) or 80-90 ^{o}C (for platinadacycle **3**-catalyzed reactions), the reaction was quenched by adding small amount of water. Column chromatography on silica gel with diethyl ether/hexane (v/v=20:1) afforded the addition product.

General Procedure for $[Rh(COD)Cl]_2$ Catalyzed Addition Reactions of Arylboronic Acids with Alkyl/benzyl 2-Formylbenzoates: To a vial containing arylboronic acid (0.75 mmol), 2-formylbenzoate (0.5 mmol) and THF (2 mL), $[Rh(COD)Cl]_2$ (0.5 mol %) and K_3PO_4 (5.0 M, 0.3 mL, 1.5 mmol.) were added in sequence. After the mixture was stirred at room temperature for 1-2 hrs, the reaction was quenched by adding water, and then extracted with CH_2Cl_2 (30 mL × 2). After remove solvent, column chromatography on silica gel with ethyl acetate/hexane afforded the product.



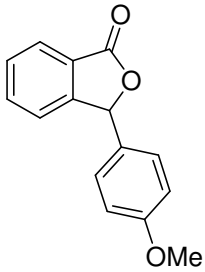
3-Phenylisobenzofuran-1(*3H*)-one[8]

^{1}H NMR ($CDCl_3$, 300 MHz): 7.97 (d, J=7.5 Hz, 1H), 7.65 (dt, J= 1.2, 7.5 Hz, 1H), 7.56 (t, J=7.5 Hz, 1H), 7.26-7.39 (m, 6H), 6.41 (s, 1H) ppm. ^{13}C NMR ($CDCl_3$, 150 MHz): 170.5, 149.7, 136.4, 134.3, 129.34, 129.29, 129.0, 127.0, 125.64, 125.59, 122.8, 82.7 ppm.



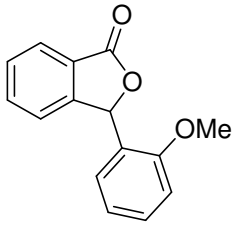
3-*p*-Tolylisobenzofuran-1(*3H*)-one[7]

^{1}H NMR ($CDCl_3$, 300 MHz): 7.96 (d, J=7.5 Hz, 1H), 7.64 (t, J=7.5 Hz, 1H), 7.55 (t, J=7.5 Hz, 1H), 7.32 (d, J=7.5 Hz, 1H), 7.13-7.20 (m, 4H), 6.38 (s, 1H), 2.35 (s, 3H) ppm. ^{13}C NMR ($CDCl_3$, 150 MHz): 170.6, 149.8, 139.3, 134.2, 133.4, 129.6, 129.3, 127.0, 125.7, 125.6, 122.8, 82.8, 21.2 ppm.



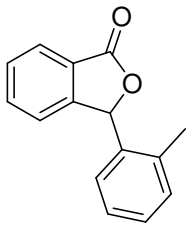
3-(4-Methoxyphenyl)isobenzofuran-1(*3H*)-one [9]

^{1}H NMR ($CDCl_3$, 300 MHz): 7.96 (d, J=7.8 Hz, 1H), 7.66 (t, J=7.2 Hz, 1H), 7.56 (t, J=7.5 Hz,1H), 7.32 (d, J=7.5 Hz, 1H), 7.18 (d, J=9.0 Hz, 2H), 6.89 (d, J=8.7 Hz, 2H), 6.38 (s, 1H), 3.81 (s, 3H) ppm. ^{13}C NMR ($CDCl_3$, 150 MHz): 170.5, 160.4, 149.8, 134.2, 129.3, 128.8, 128.3, 125.9, 125.6, 122.9, 114.3, 82.7, 55.3 ppm.



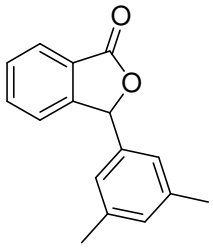
3-(2-Methoxyphenyl)isobenzofuran-1(*3H*)-one [10]

^{1}H NMR ($CDCl_3$, 300 MHz): 7.93 (d, J=7.2 Hz, 1H), 7.61 (dt, J=0.9, 7.2 Hz, 1H), 7.51 (t, J=7.5 Hz, 1H), 7.44 (d, J=7.5 Hz, 1H), 7.30-7.35 (m, 1H), 7.08 (dd, J=1.5, 7.5 Hz, 1H), 6.97 (d, J=8.1 Hz, 1H), 6.91 (t, J=7.8 Hz), 6.87 (s, 1H), 3.91 (s, 3H) ppm. ^{13}C NMR ($CDCl_3$, 75 MHz): 170.9, 156.9, 150.3, 134.0, 130.0, 128.9, 126.7, 125.4, 125.2, 124.8, 122.8, 120.7, 110.9, 77.9, 55.4 ppm.



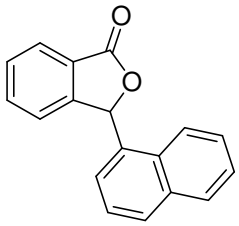
3-*o*-Tolylisobenzofuran-1(*3H*)-one [11]

^{1}H NMR ($CDCl_3$, 600 MHz): 7.96 (d, J=7.8 Hz, 1H), 7.66 (dt, J=1.2, 7.8 Hz, 1H), 7.56 (t, J=7.2 Hz,1H), 7.34 (dd, J=0.6, 7.8 Hz, 1H), 7.20-7.28 (m, 2H), 7.12 (dt, J=1.8, 7.8 Hz, 1H), 6.91 (d, J=7.8 Hz, 1H), 6.67 (s, 1H), 2.49 (s, 3H) ppm. ^{13}C NMR ($CDCl_3$, 75 MHz): 170.6, 149.3, 137.2, 134.2, 134.1, 131.1, 129.4, 129.3, 127.3, 126.41, 126.38, 125.7, 123.1, 80.5, 19.3 ppm.



3-(3, 5-Dimethylphenyl)isobenzofuran-1(*3H*)-one

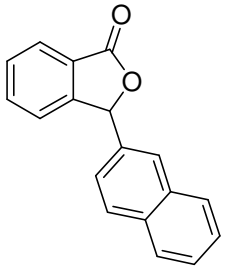
^{1}H NMR ($CDCl_3$, 300 MHz): 7.96 (d, J=7.5 Hz, 1H), 7.64 (t, J=7.2 Hz, 1H), 7.55 (t, J=7.5 Hz, 1H), 7.34 (d, J=7.5 Hz, 1H), 6.99 (s, 1H), 6.87 (s, 2H), 6.33 (s, 1H), 2.29 (s, 6H) ppm. ^{13}C NMR ($CDCl_3$, 75 MHz): 170.6, 149.9, 138.6, 136.2, 134.2, 130.9, 129.2, 125.6, 124.6, 122.8, 82.9, 21.2 ppm. FT-IR (cm^{-1}): 3092(w), 3022(w), 2973(w), 2917(w), 1750(s), 1612(m), 1600(m), 1467(s), 1334(m), 1300(m), 1282(s), 1209(s), 1190(m), 1159(m), 1102(m), 1062(s), 984(s), 937(s), 909(m), 867(m), 841(s), 804(m), 761(s), 719(s), 706(s), 689(s), 671(m), 631(m). MS (EI) m/z: 238 (M^+, 48.6), 223(100), 179(11.8), 133(53.2), 105(30.4), 104(31.2), 77(25.8), 76(14.3), 51(9.1).



3-(Naphthalen-1-yl)isobenzofuran-1(*3H*)-one [12]

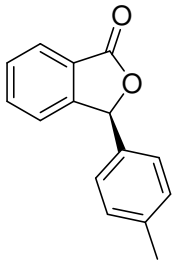
^{1}H NMR ($CDCl_3$, 300 MHz): 8.23 (d, J=8.1 Hz, 1H), 8.01 (d, J=6.9 Hz, 1H), 7.93 (d, J=7.5 Hz, 1H), 7.87 (d, J=8.4 Hz, 1H), 7.55-7.66 (m, 4H), 7.36-7.44 (m, 2H), 7.24-7.27

(m, 2H) ppm. ^{13}C NMR ($CDCl_3$, 150 MHz): 170.5, 149.3, 134.1, 134.0, 131.9, 131.3, 129.9, 129.5, 129.0, 127.0, 126.2, 126.1, 126.0, 125.2, 124.5, 123.2, 122.9, 79.6 ppm.

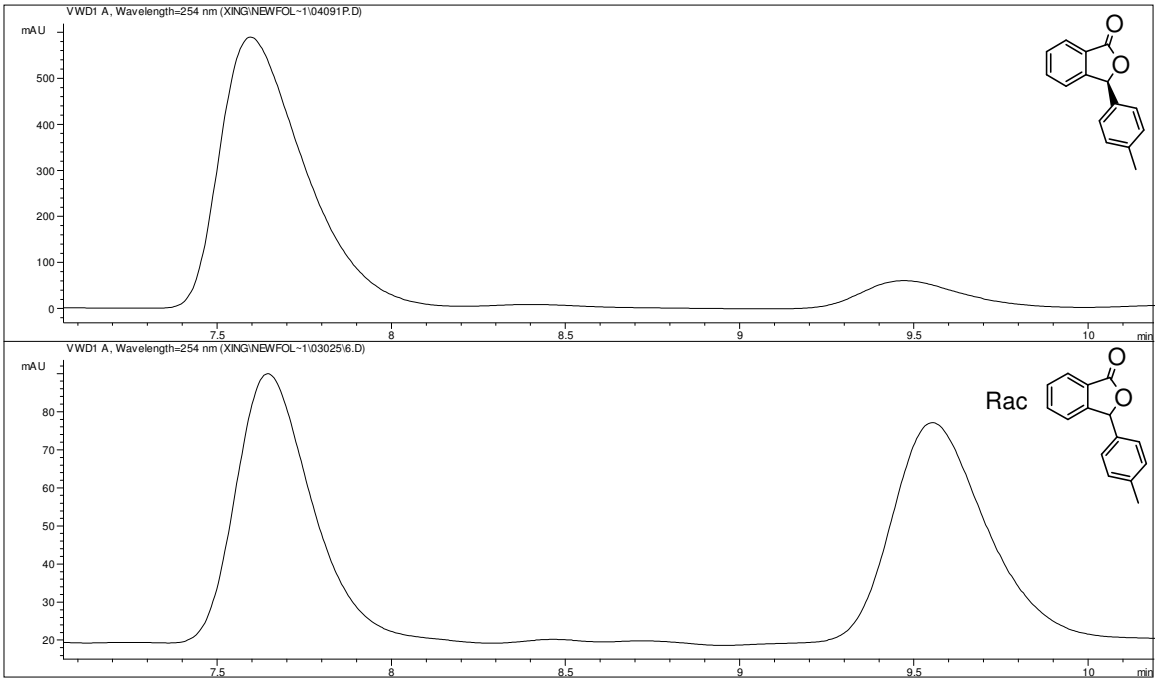


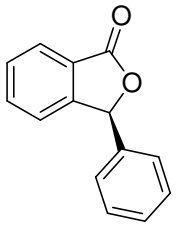
3-(Naphthalen-3-yl)isobenzofuran-1(*3H*)-one[13]
^{1}H NMR ($CDCl_3$, 300 MHz): 8.01 (d, J=7.5 Hz, 1H), 7.82-7.85 (m, 4H), 7.65 (dt, J=1.2, 7.5 Hz, 1H), 7.51-7.60 (m, 3H), 7.35 (d, J=7.5 Hz, 1H), 7.22-7.26 (m, 1H), 6.57(s, 1H) ppm. ^{13}C NMR ($CDCl_3$, 150 MHz): 170.6, 149.7, 134.4, 133.7, 133.6, 133.1, 129.4, 129.1, 128.1, 127.8, 126.8, 126.71, 126.69, 125.7, 125.6, 123.8, 122.9, 82.9 ppm.

General Procedure for $[Rh(C_2H_4)_2Cl]_2$/(*R*)-Ligand Catalyzed Addition Reactions of Arylboronic Acids with Aromatic and Aliphatic Aldehydes: To a schlenk flask, $[Rh(C_2H_4)_2Cl]_2$ (0.5 mmol%) and ligand (3 mmol%) were mixed in 3mL toluene and stirred under nitrogen at room temperature for 1 hour. Boronic acid (0.75 mmol) and K_3PO_4 (5M, 0.1-0.15 mL) was added and cooled to 0 ^{o}C, then 2-formylbenzoate (0.5 mmol) was injected. The reaction mixture was stirred at 0 ^{o}C for 12-24 hours. The reaction was quenched by adding brine, and then extracted with ethyl acetate (50 mL). After remove solvent, column chromatography on silica gel with ethyl acetate/hexane afforded the product. Enantiomerical excess were check by chiral HPLC. Absolute configuration was determined based on reference.[14, 15]



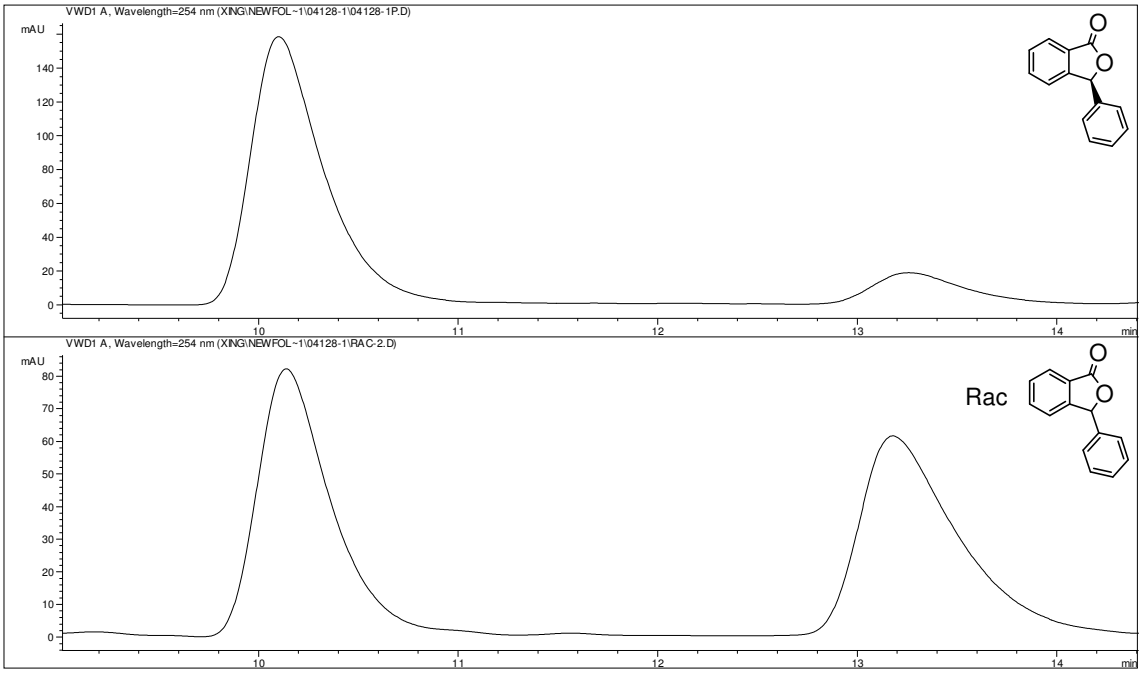
(*S*)-3-*p*-Tolylisobenzofuran-1(*3H*)-one [14]
$[\alpha]_D$ = + 10.3 ^{o} (c = 0.101, $CHCl_3$); 77% e. e. (HPLC condition: Chiralcel OD column, Hexanes/2-Propanol=80/20, 1.0mL/min, 254 nm UV detector, $t_{R\ major}$= 7.59 min, $t_{R\ minor}$= 9.47 min). ^{1}H NMR ($CDCl_3$, 600 MHz): 7.96 (d, J=7.2 Hz, 1H), 7.63-7.66 (m, 1H), 7.55 (t, J=7.2 Hz, 1H), 7.32 (d, J=7.8 Hz, 1H), 7.15-7.19 (m, 4H), 6.38 (s, 1H), 2.35 (s, 3H) ppm. ^{13}C NMR ($CDCl_3$, 150 MHz): 170.6, 149.8, 139.4, 134.3, 133.4, 129.6, 129.3, 127.1, 125.8, 125.6, 122.9, 82.8, 21.2 ppm.

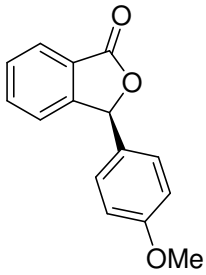




(S)-3-Phenylisobenzofuran-1(*3H*)-one [7, 14]

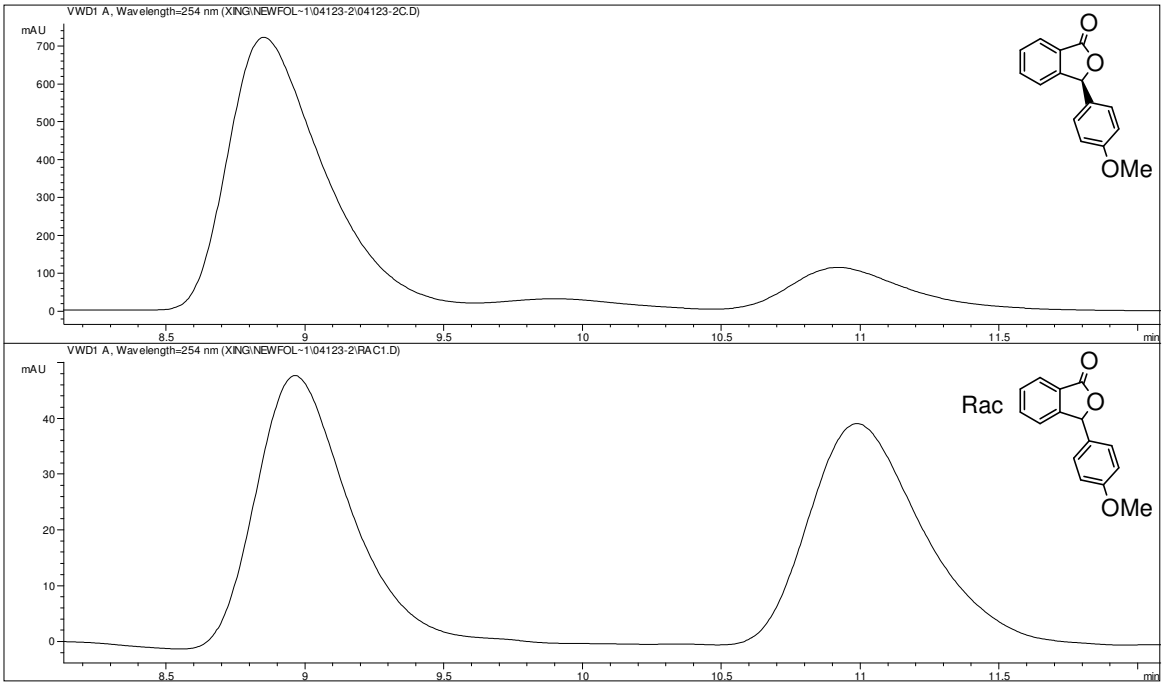
$[\alpha]_D$ = + 45.3° (c = 0.101, $CHCl_3$). 71% ee (HPLC condition: Chiralcel OD column, Hexanes/2-Propanol=85/15, 1.0mL/min, 254 nm UV detector, t_{Rmajor}= 10.10 min, t_{Rminor}= 13.26 min). ^1H NMR ($CDCl_3$, 300 MHz): 7.97 (d, J=7.5 Hz, 1H), 7.65 (dt, J=1.2 Hz, 7.5 Hz, 1H), 7.56 (t, J=7.5 Hz, 1H), 7.27-7.39 (m, 6H), 6.41 (s, 1H), 2.35 (s, 3H) ppm. ^{13}C NMR ($CDCl_3$, 150 MHz): 170.5, 149.7, 136.4, 134.3, 129.4, 129.3, 129.0, 127.0, 125.7, 125.6, 122.8, 82.7 ppm.

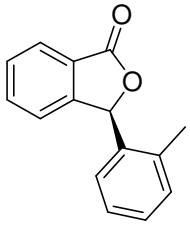




(*S*)-3-(4-Methoxyphenyl)isobenzofuran-1(*3H*)-one [14]

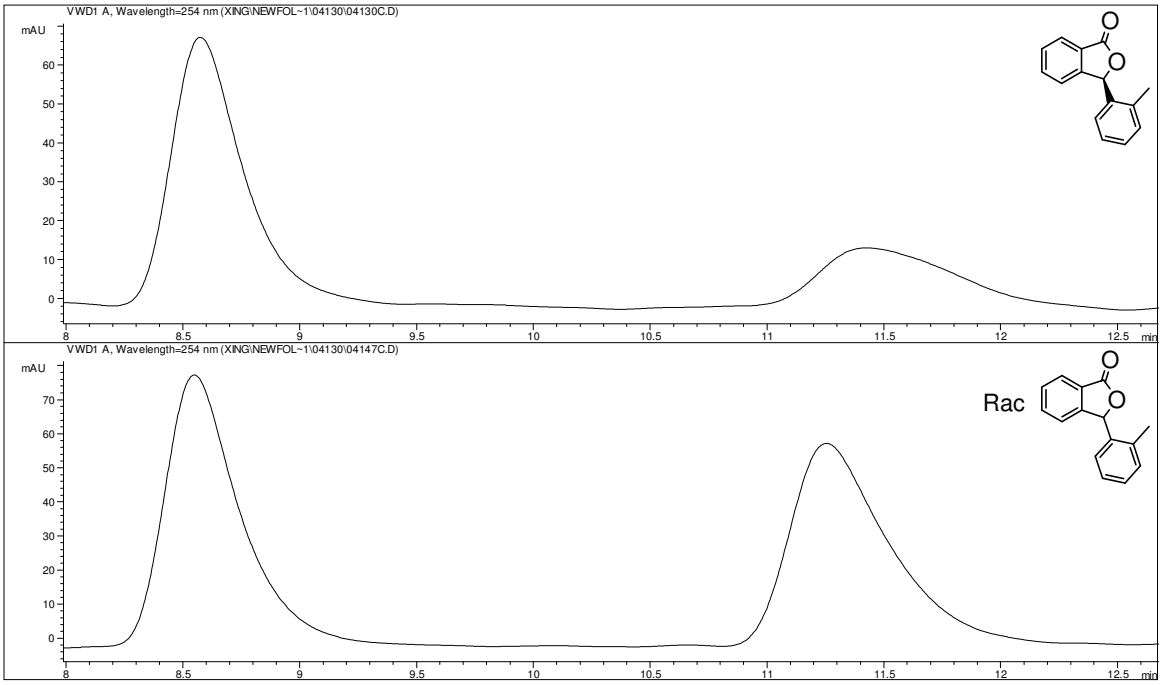
$[\alpha]_D$ = -27.8° (c = 0.101, $CHCl_3$). 67% ee (HPLC condition: Chiralcel OD column, Hexanes/2-Propanol=75/25, 1.0mL/min, 254 nm UV detector, t_{Rmajor}= 9.31 min, t_{Rminor}= 11.59 min). ^{1}H NMR ($CDCl_3$, 300 MHz): 7.96 (d, J=7.8 Hz, 1H), 7.65 (dt, J=1.2, 7.5 Hz, 1H), 7.56 (t, J=7.5 Hz,1H), 7.32 (d, J=7.5 Hz, 1H), 7.18 (d, J=8.4 Hz, 2H), 6.89 (d, J=9.0 Hz, 2H), 6.37 (s, 1H), 3.81 (s, 3H) ppm. ^{13}C NMR ($CDCl_3$, 150 MHz): 170.5, 160.4, 149.7, 134.2, 129.3, 128.8, 128.3, 125.9, 125.6, 122.9, 114.3, 82.7, 55.3 ppm.

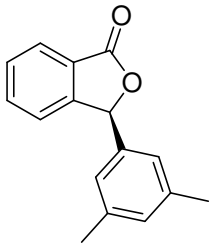




(*S*)-3-*o*-Tolylisobenzofuran-1(*3H*)-one

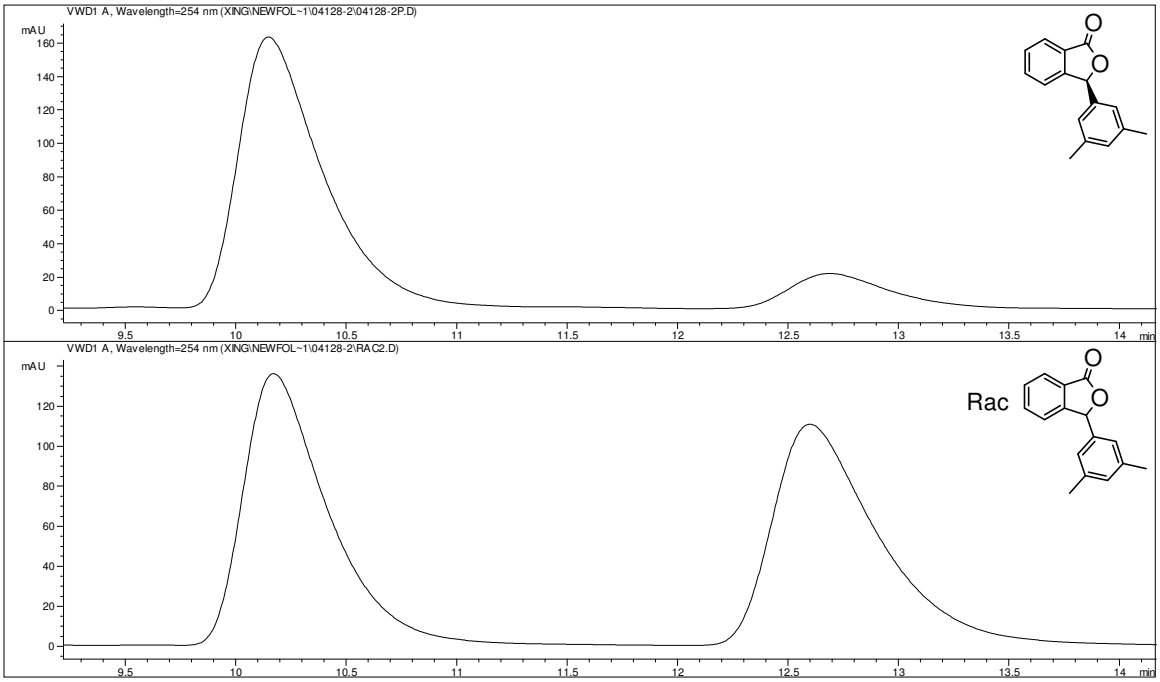
$[\alpha]_D$ = -55.3° (c = 0.103, $CHCl_3$). 63% ee (HPLC condition: Chiralcel OD column, Hexanes/2-Propanol=80/20, 1.0mL/min, 254 nm UV detector, t_{Rmajor}= 8.57 min, t_{Rminor}= 11.43 min). ^1H NMR ($CDCl_3$, 600 MHz): 7.98 (d, J=7.2 Hz, 1H), 7.67 (t, J=7.8 Hz, 1H), 7.58 (t, J=7.2 Hz,1H), 7.35 (d, J=7.8 Hz, 1H), 7.26-7.29 (m, 2H), 7.12-7.15 (m, 1H), 6.92 (d, J=7.8 Hz, 1H), 6.69 (s, 1H), 2.50 (s, 3H) ppm. ^{13}C NMR ($CDCl_3$, 75 MHz): 170.6, 149.3, 137.2, 134.2, 134.1, 131.1, 129.4, 129.30, 127.3, 126.4, 126.4, 125.8, 123.0, 80.5, 19.3 ppm.

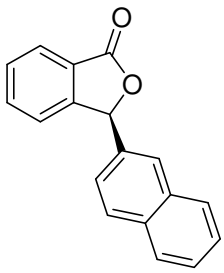




(*S*)-3-(3, 5-Dimethylphenyl)isobenzofuran-1(*3H*)-one

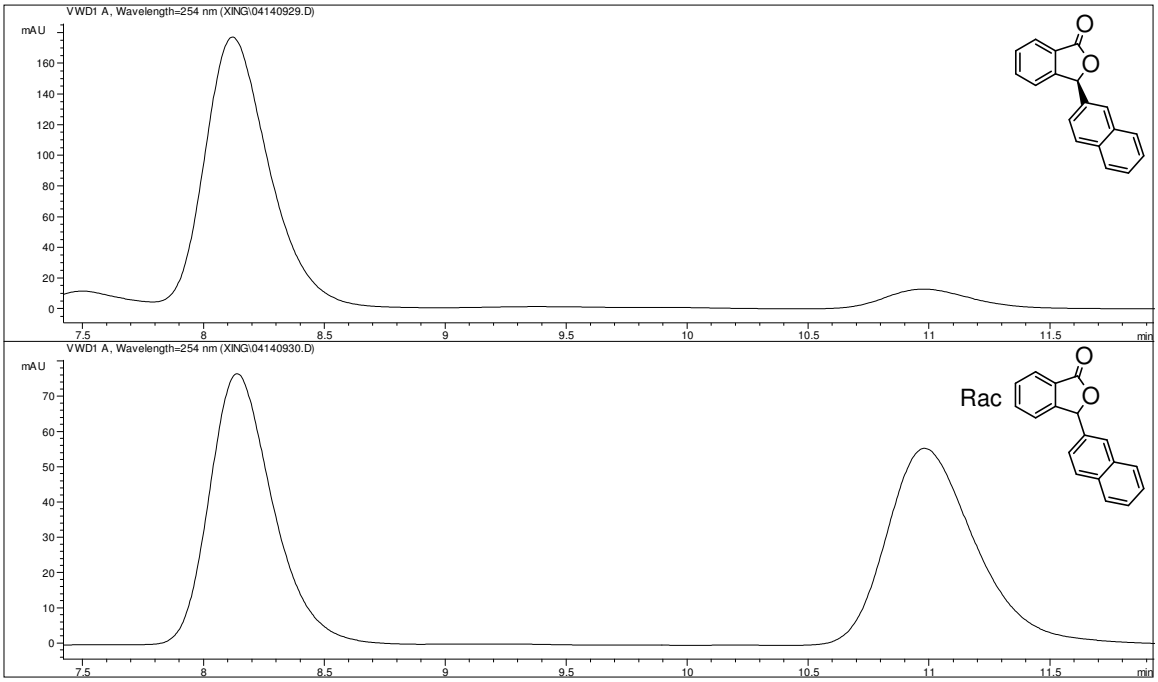
$[\alpha]_D$ = +18.8^o (c = 0.072, $CHCl_3$).66.5% ee (HPLC condition: Chiralcel OD column, Hexanes/2-Propanol=95/5, 1.0mL/min, 254 nm UV detector, t_{Rmajor}= 10.15 min, t_{Rminor}= 12.69 min). ^{1}H NMR ($CDCl_3$, 300 MHz): 7.96 (d, J=7.5 Hz, 1H), 7.65 (t, J=7.5 Hz, 1H), 7.55 (t, J=7.5 Hz, 1H), 7.34 (d, J=7.8 Hz, 1H), 7.00 (s, 1H), 6.87 (s, 2H), 6.33 (s, 1H), 2.30 (s, 6H) ppm. ^{13}C NMR ($CDCl_3$, 150 MHz): 170.7, 149.9, 138.7, 136.2, 134.3, 130.9, 129.3, 125.6, 125.6, 124.6, 122.8, 82.9, 21.3 ppm.





(*S*)-3-(Naphthalen-3-yl)isobenzofuran-1(*3H*)-one[13]

$[\alpha]_D$ = +97.9^o (c = 0.101, $CHCl_3$). 83% ee (HPLC condition: Chiralcel OD column, Hexanes/2-Propanol=65/35, 1.0mL/min, 254 nm UV detector, t_{Rmajor}= 8.12 min, t_{Rminor}= 10.98 min). ^{1}H NMR ($CDCl_3$, 600 MHz): 8.09 (d, J=7.8 Hz, 1H), 7.83-7.86 (m, 4H), 7.65 (t, J=7.2 Hz, 1H), 7.58 (t, J=7.2 Hz, 1H), 7.51-7.54 (m, 2H), 7.35 (d, J=7.8 Hz, 1H), 7.23-7.25 (m, 1H), 6.57(s, 1H) ppm. ^{13}C NMR ($CDCl_3$, 150 MHz): 170.6, 149.7, 134.4, 133.7, 133.6, 133.1, 129.5, 129.1, 128.1, 127.8, 126.8, 126.72, 126.71, 125.8, 125.7, 123.8, 122.9, 82.9 ppm.



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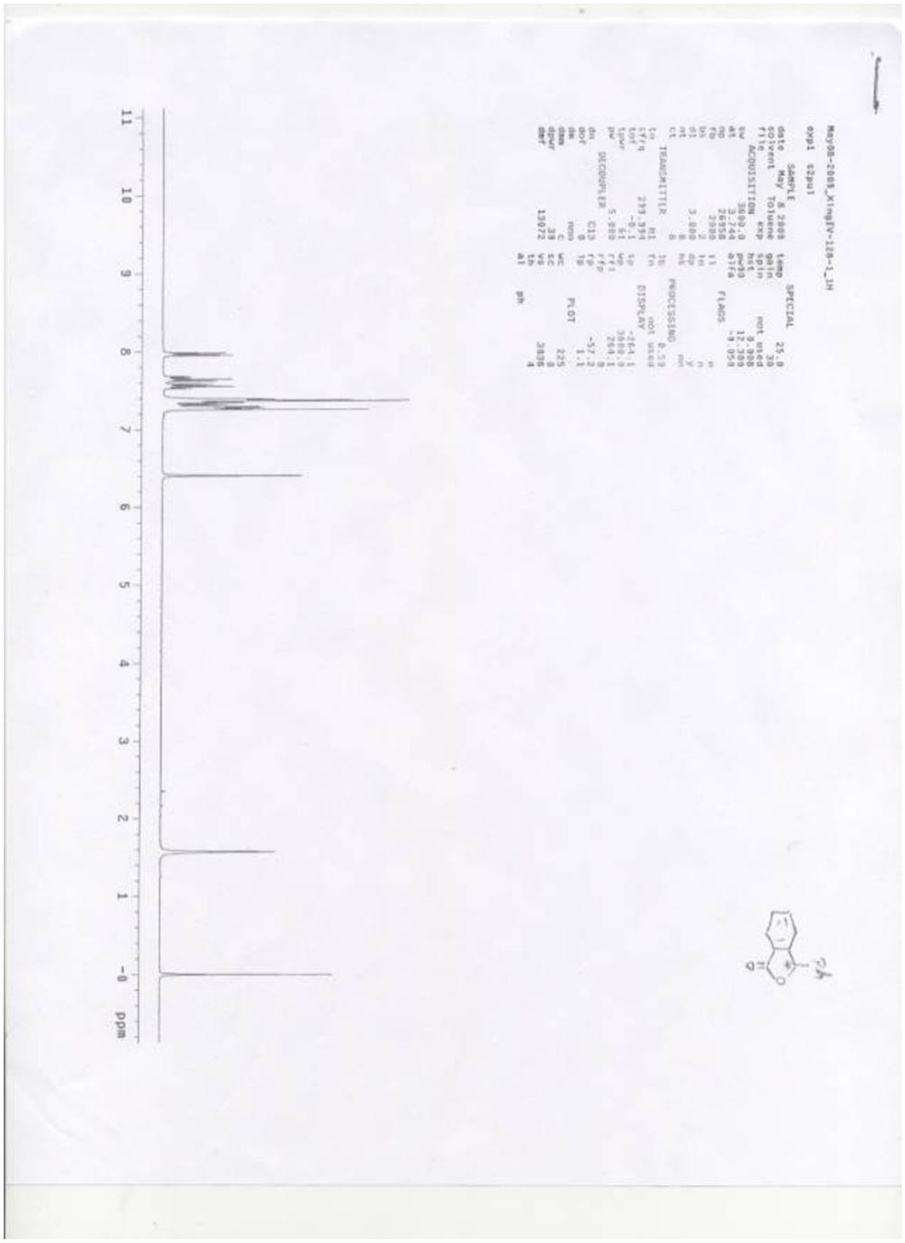

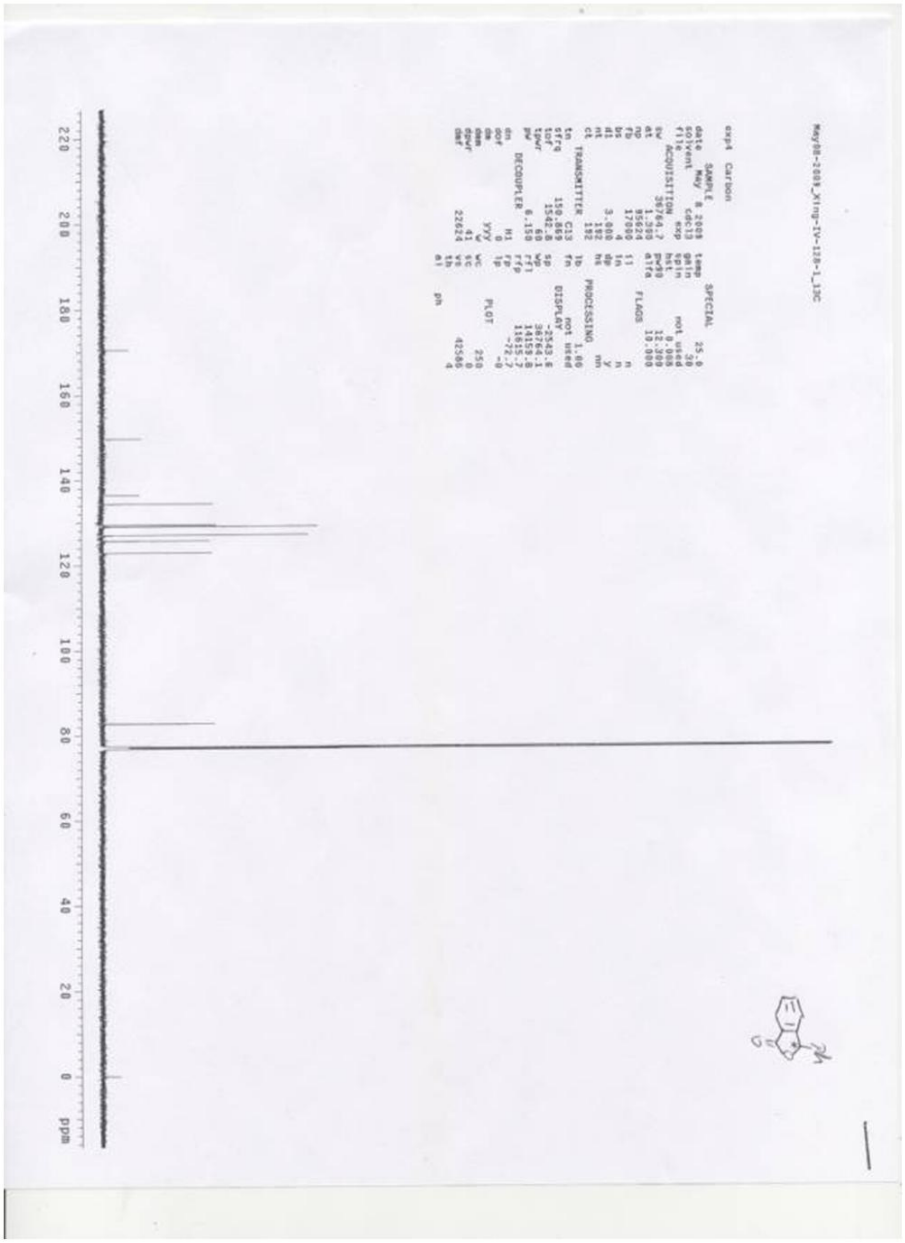
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exp4 Carbon
SAMPLE
date 2008
solvent May
file exp
ACQUISITION
PROCESSING
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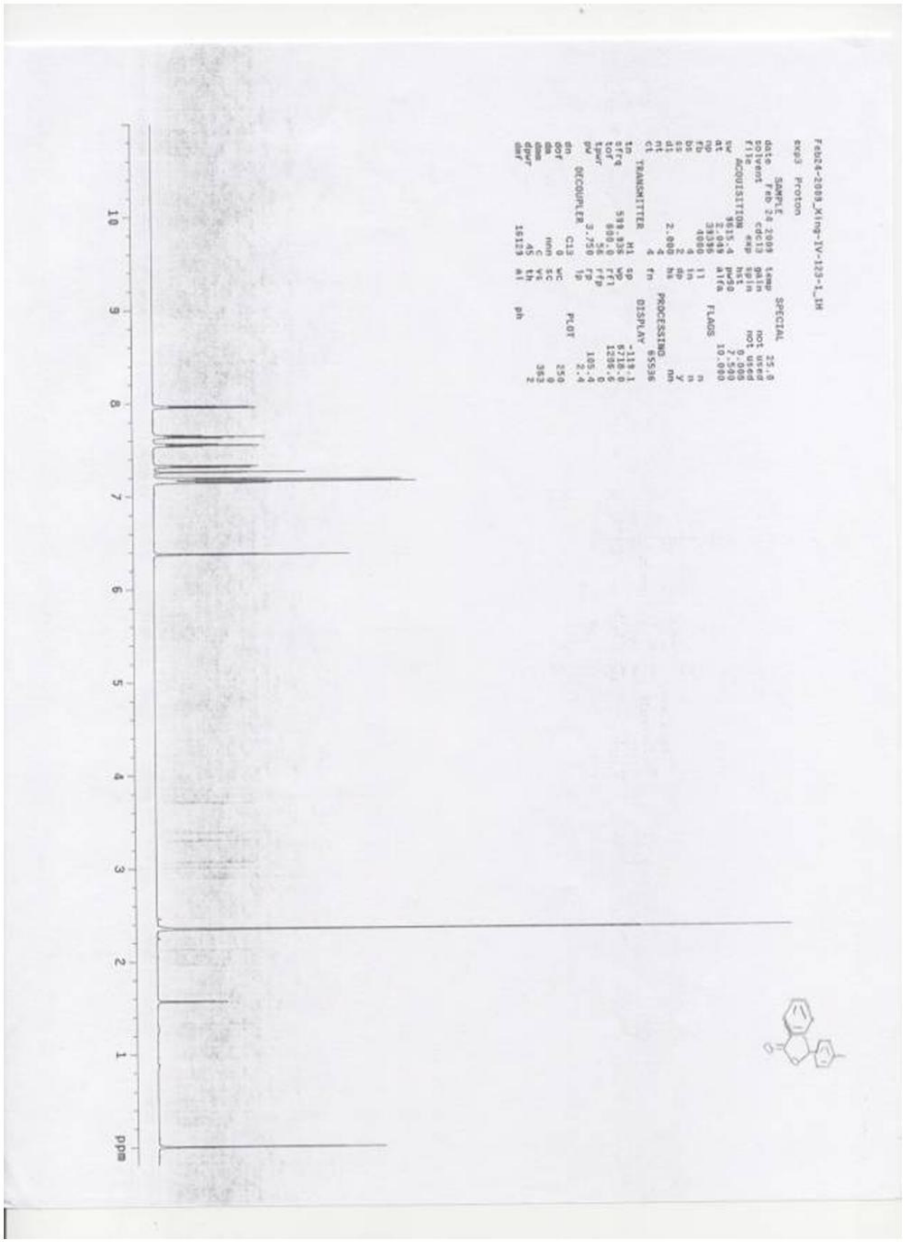
DISPLAY
DECOUPLER
220
200
180
160
140
120
100
80
60
40
20
0
ppm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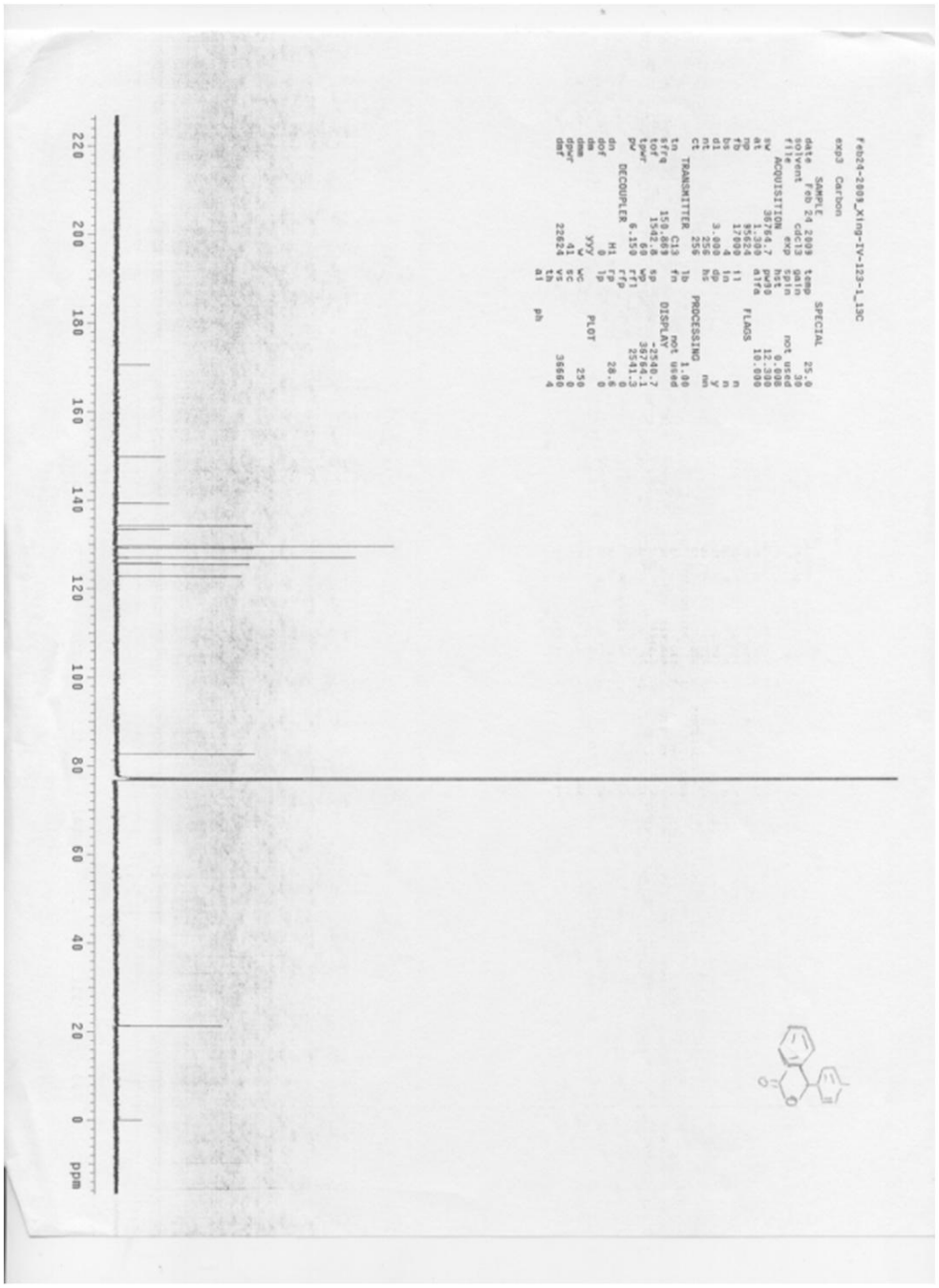
Feb24-2009_Xing-IV-123-1_13C
exp3 Carbon
SAMPLE
date Feb 24 2009
solvent CDCl3
file
ACQUISITION
sw 38764.7
at 1.013
np 85624
fb 17000
bs
nt 256
ct 256
TRANSMITTER
tn C13
sfrq 150.883
tof 1542.8
tpwr 60
pw 6.150
DECOUPLER
dn H1
dof 0
dm yyy
dmm c
dmf 22624
temp 25.0
gain 30
spin not used
hst 0.008
pwp 12.300
dtyp 10.000
FLAGS
in n
dp y
hs nn
PROCESSING
lb 1.00
fn not used
DISPLAY
sp -2540.7
wp 38764.1
rfl 2541.3
rfp 0
rp 20.6
lp 0
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ph
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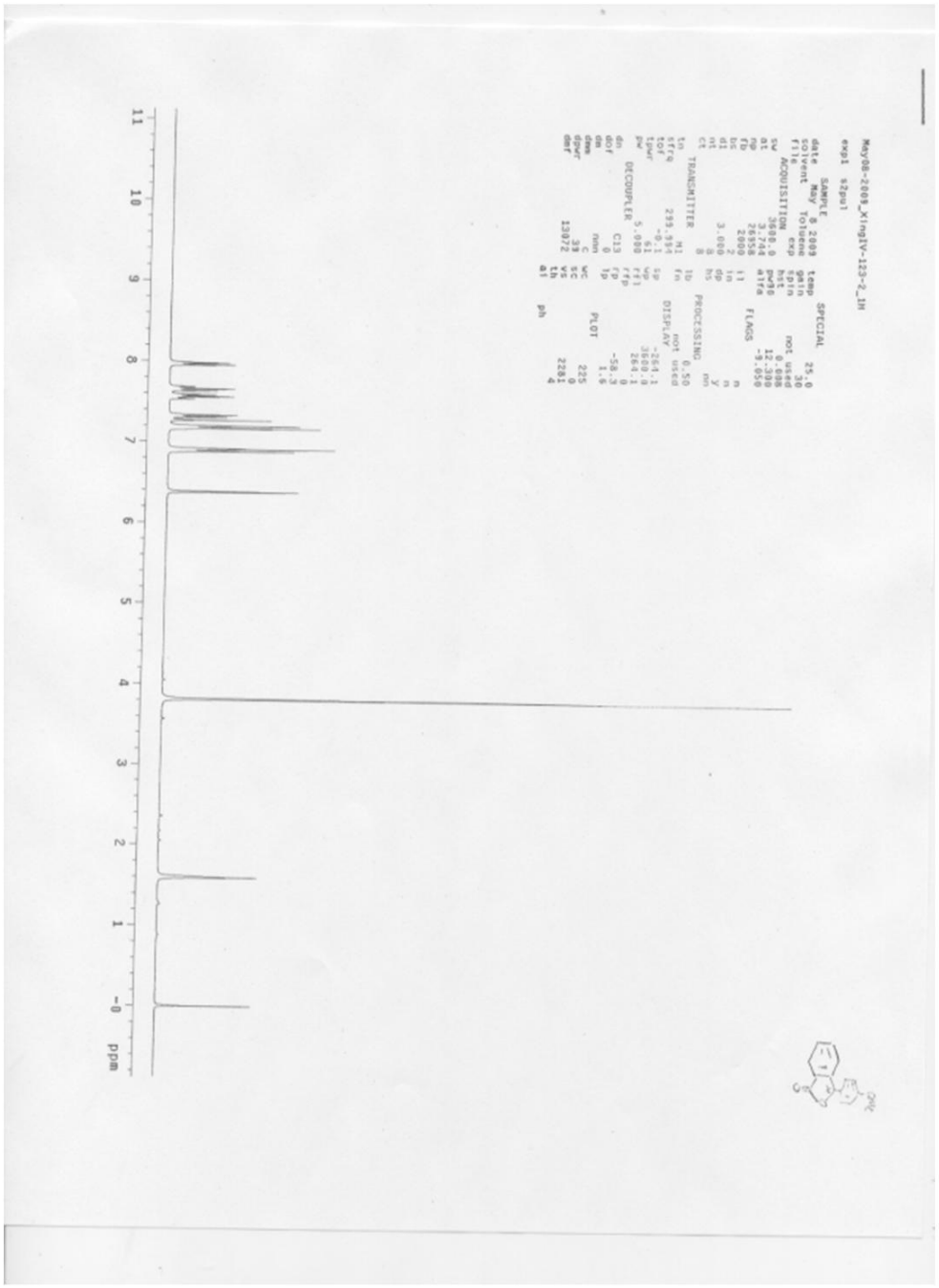
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solvent Toluene
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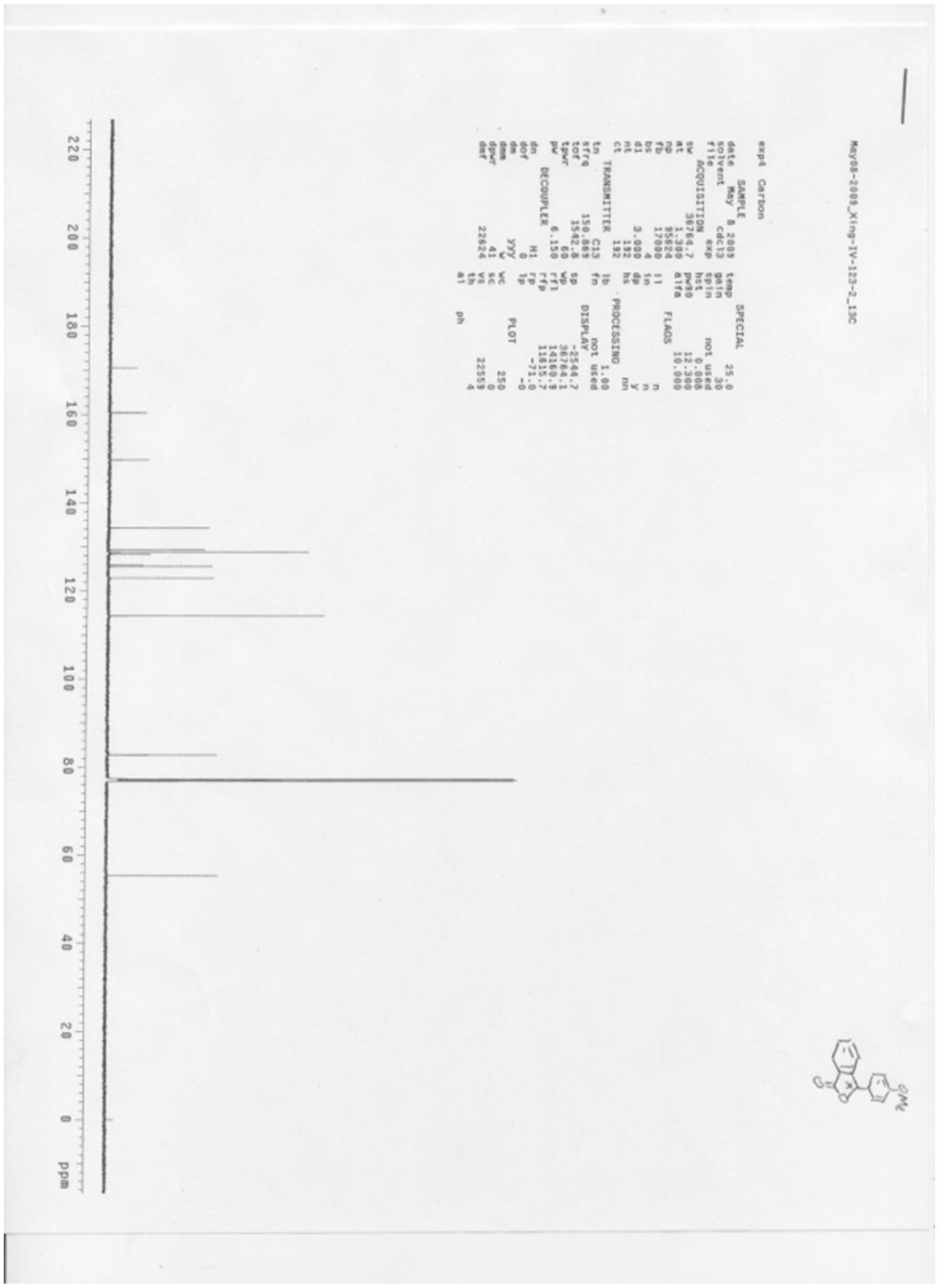
FLAGS
PROCESSING
DISPLAY
PLOT
ppm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date 8 2009
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np 35824
fb 17000
bs
wb
dl 3.000
nt 192
ct 192
TRANSMITTER
tn C13
sfrq 150.869
tof 1542.8
tpwr 60
pw 6.150
DECOUPLER
dn H1
dof 0
dm yyy
dmm y
dpwr 41
dres 2262
df 2262
SPECIAL
temp 25.0
gain 30
spin not used
hst 0.000
alfa 10.000
FLAGS
n
y
y
nn
fb not used
PROCESSING
wft 1.00
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ph
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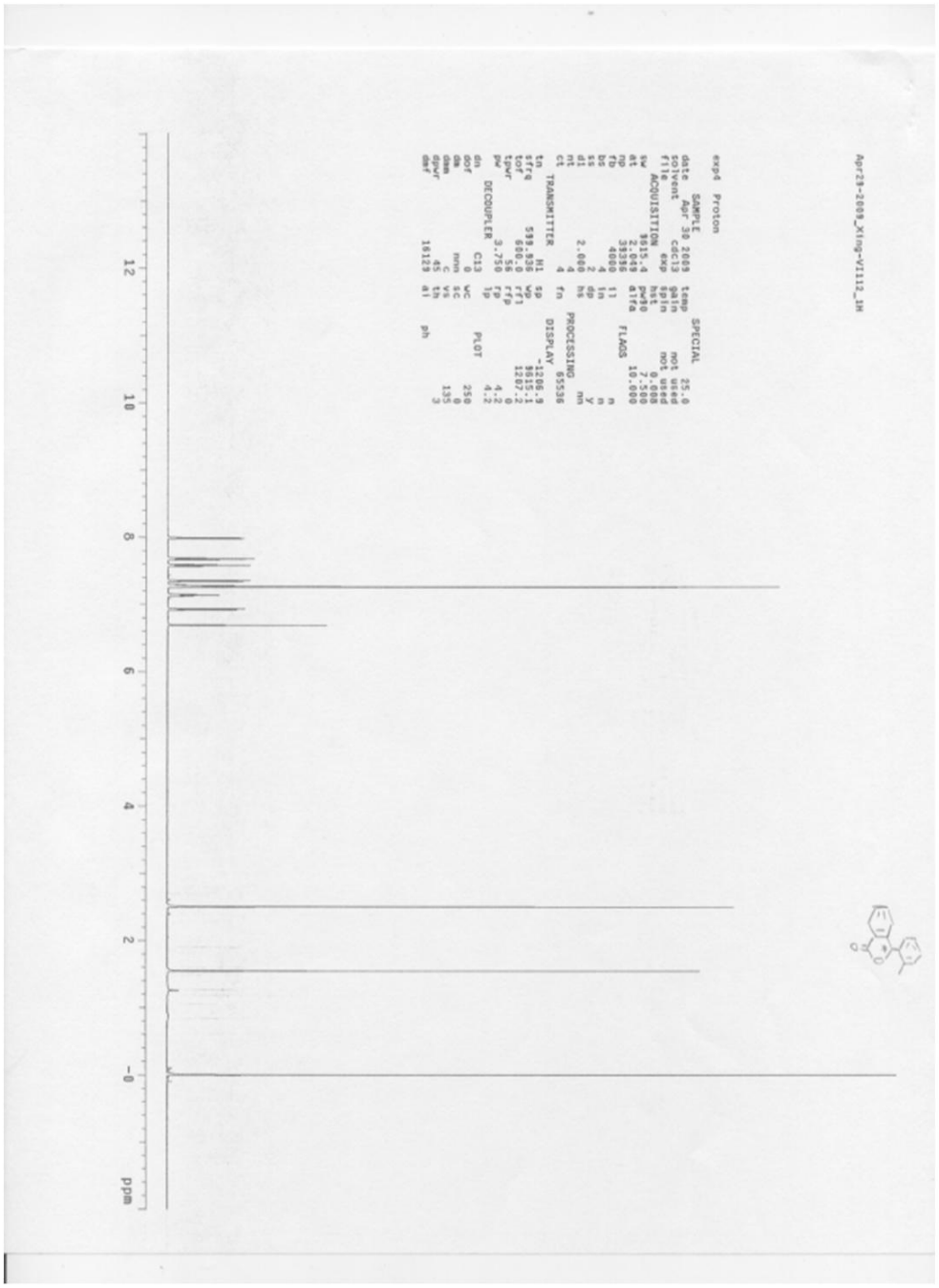
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SAMPLE
date Apr 30 2003
solvent cdcl3
file
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sw
at
fb
bs
ss
nt
ct
TRANSMITTER
tn H1
sfrq 599.881
tof 600.0
tpwr 56
pw 3.750
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dmf 45
16129
ph
temp SPECIAL
spin not used
hst not used
pw90 7.500
d1fa 10.000
FLAGS
n
y
nn
PROCESSING
DISPLAY
sp -120.3
wp 1157.1
rfl 1207.0
rfp 4.2
lp 4.2
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sc 0
vs 135
th 3
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ph
12
10
8
6
4
2
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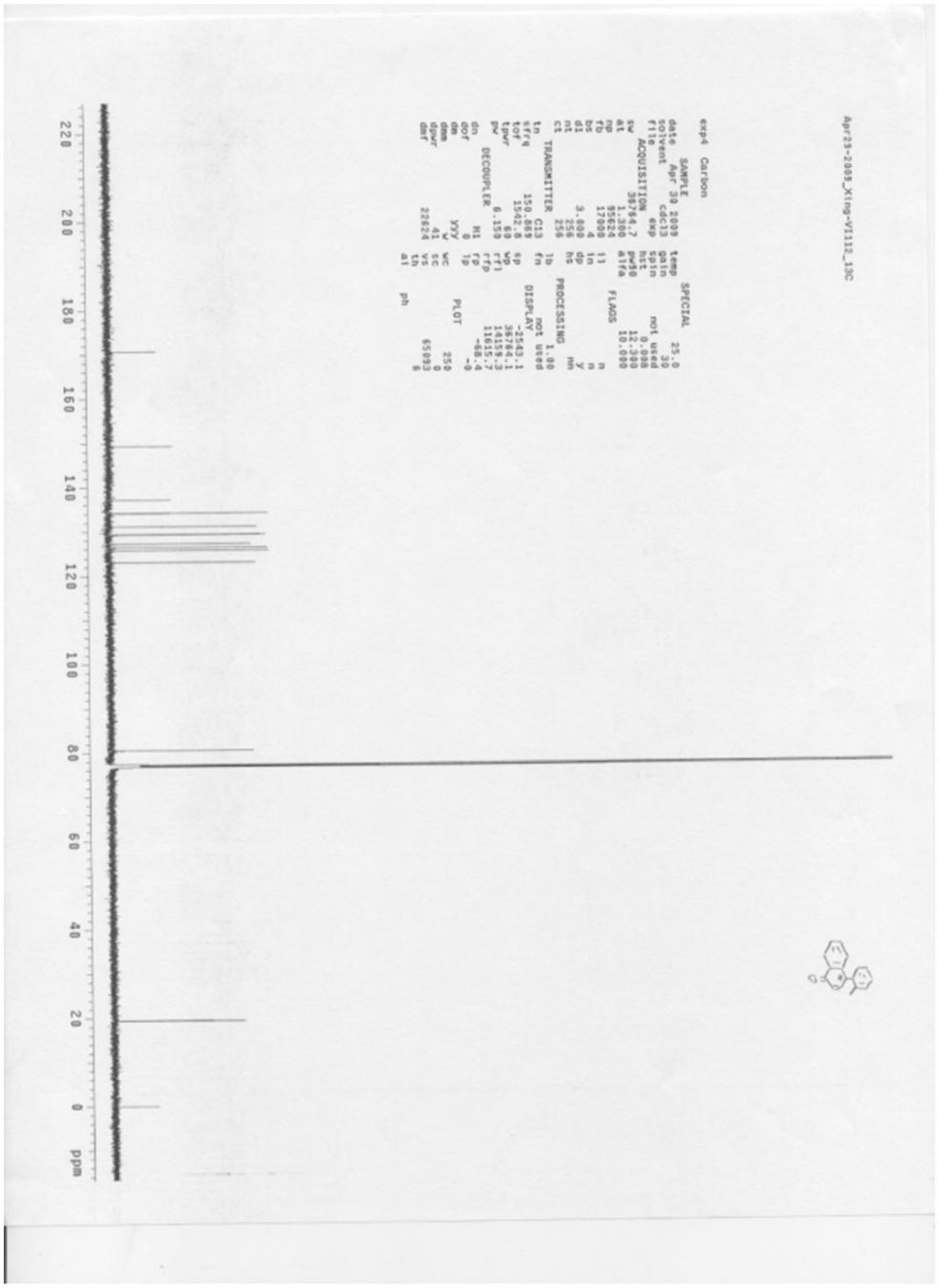
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exp4 Carbon
SAMPLE
date Apr 30 2009
solvent cdcl3
file exp
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sw 38744.7
at 1.000
np 65024
fb 17000
bs 4
d1 2.000
nt 256
ct 256
TRANSMITTER
tn C13
sfrq 150.858
tof 19428.8
tpwr 69
pw 6.150
DECOUPLER
dn H1
dof 0
dm yyy
dmm c
dmf 22624
dof
SPECIAL
temp 25.0
spin not used
hst 0.008
pw90 12.300
alfa 10.000
FLAGS
in n
dp y
hs nn
PROCESSING
lb 1.00
fn DISPLAY not used
sp -2543.1
wp 36764.1
rf1 14155.3
rfp 1161.2
lp -588.9
PLOT
sc 250
wc 65093
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th
al
ph
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60
40
20
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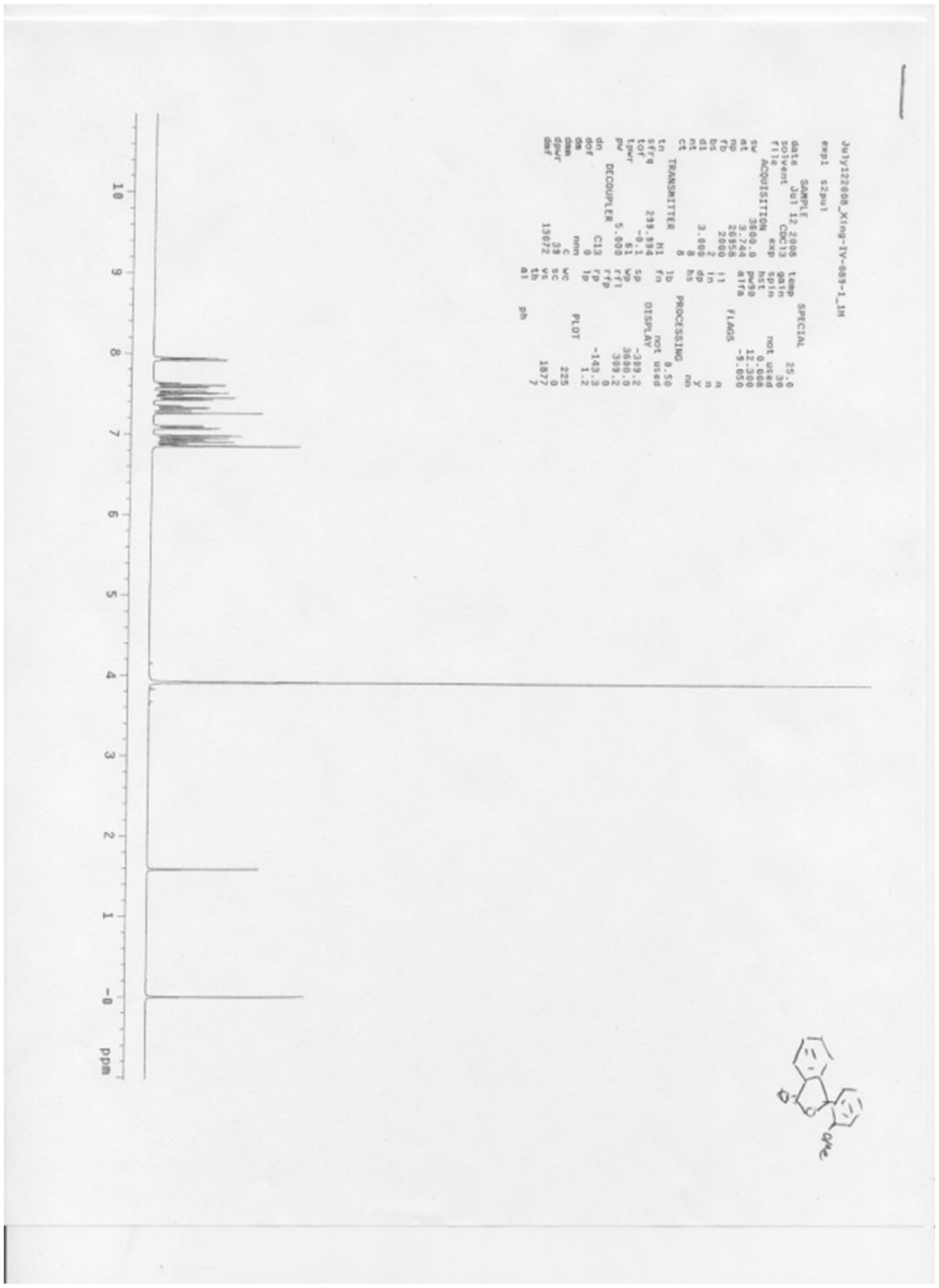
ppm


July122008_Xing-IV-089-1_1H
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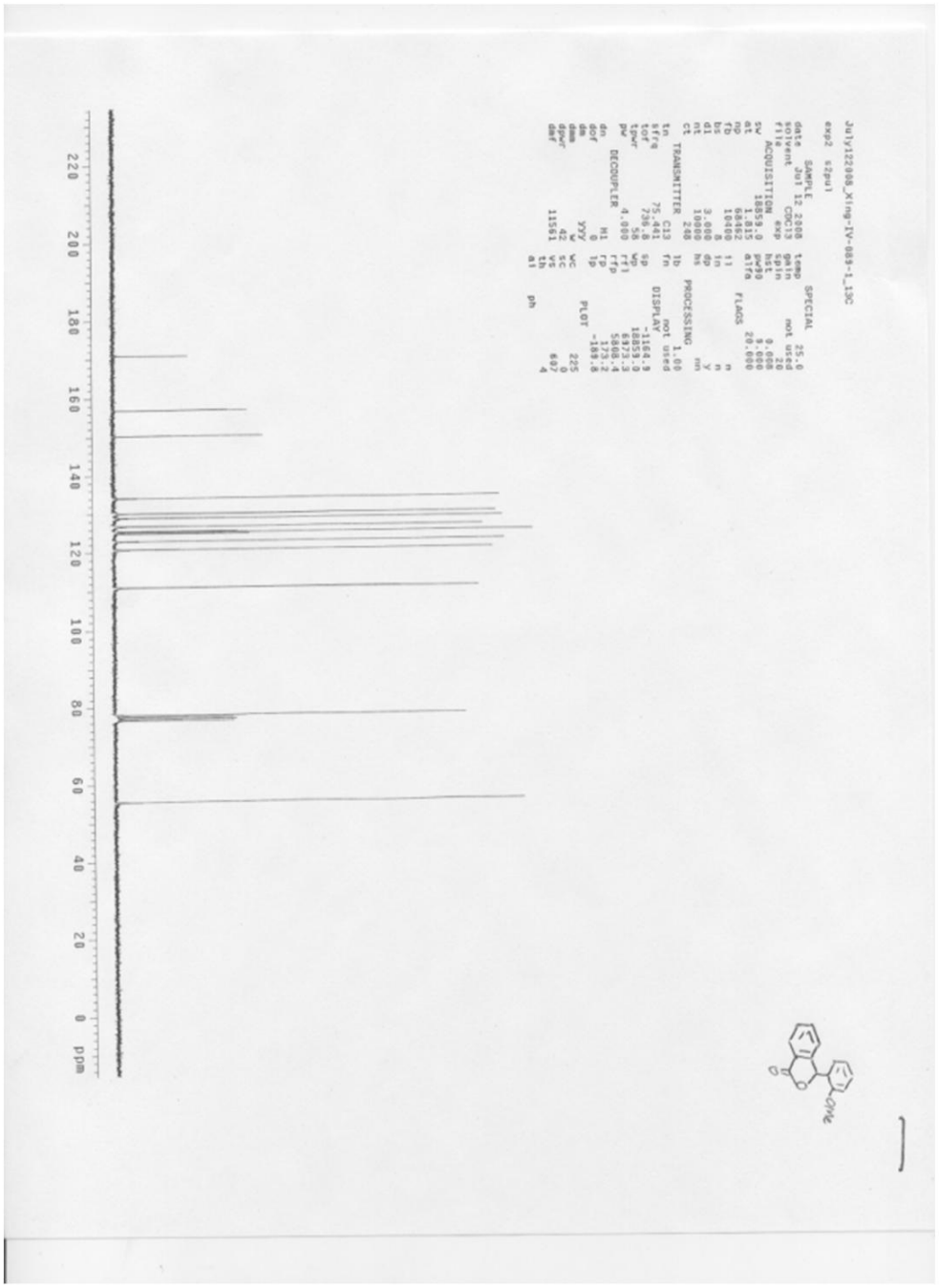
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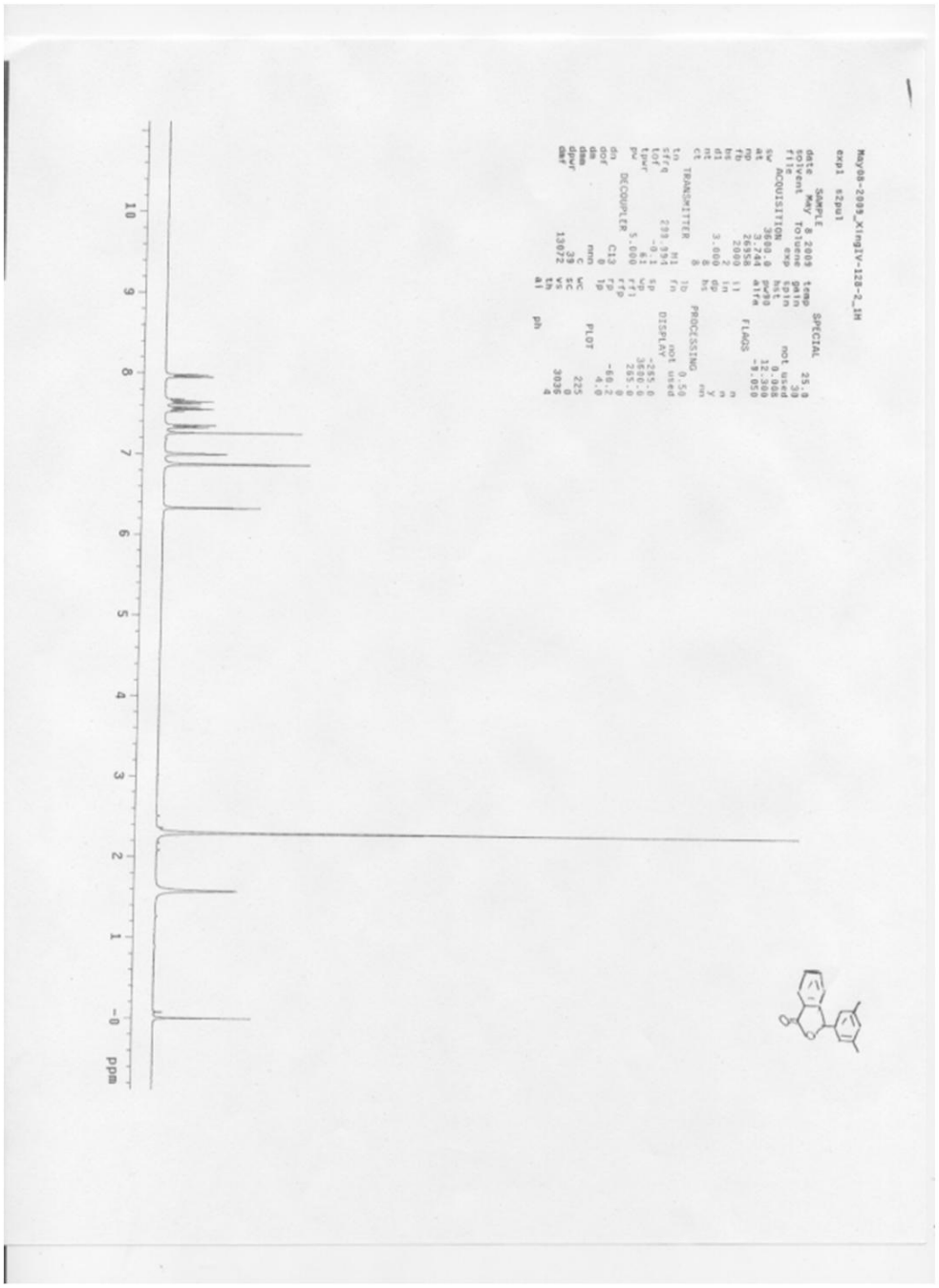
120
100
80
60
40
20
0
ppm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5
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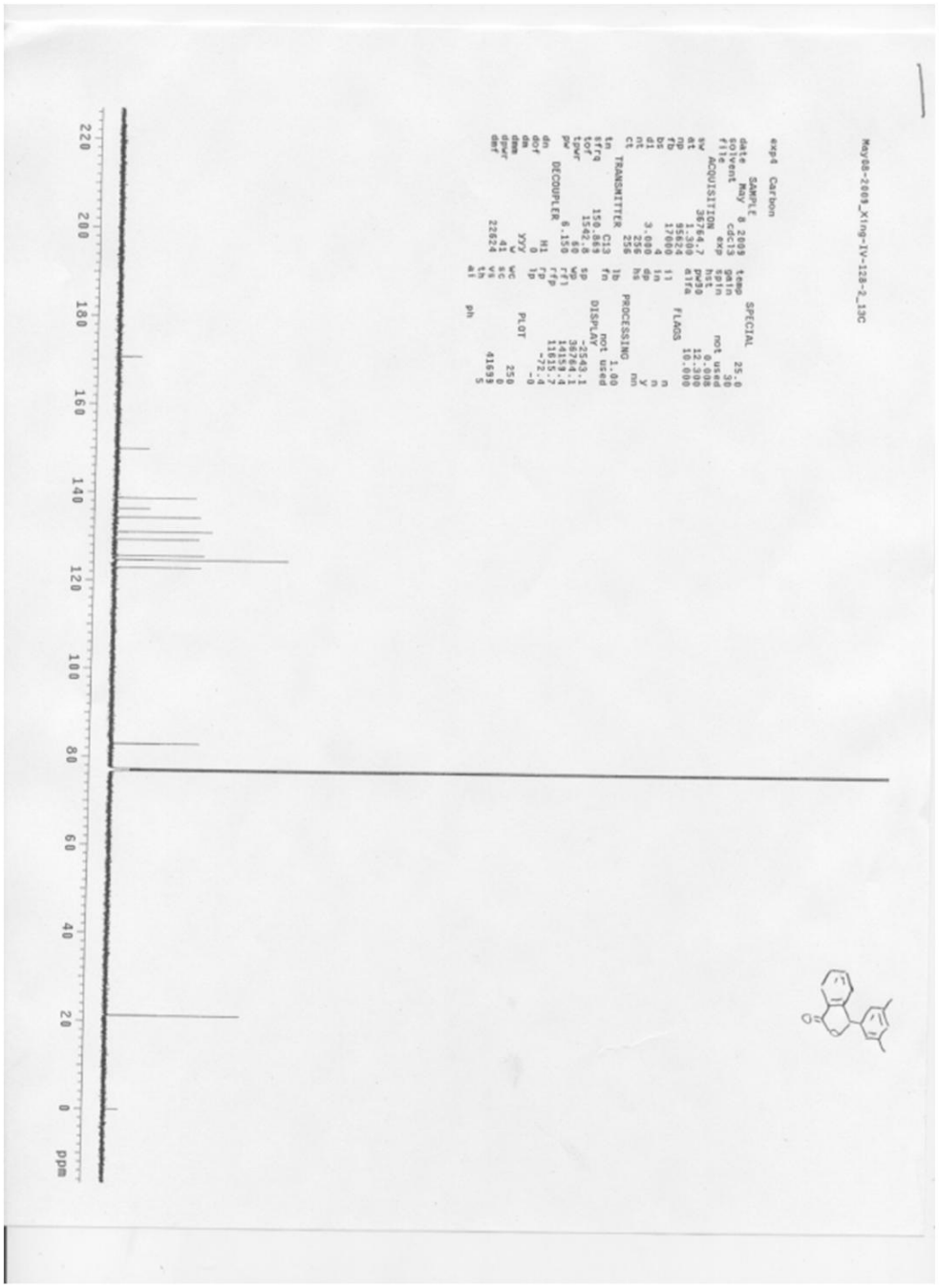
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SAMPLE
date May 8 2008
solvent CDCl3
file exp
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sw 38764.1
at 1.000
np 95824
fb 17000
bs 4
bs 3.000
nt 256
ct 256
TRANSMITTER
tn C13
sfrq 150.913
tof 1542.8
tpwr 60
pw 6.150
DECOUPLER
dn H1
dof 0
dm yyy
dmm c
dmf 22624
dpwr 41
dof 0
SPECIAL
temp 25.0
spin 20
hst 0.008
pw90 12.300
atfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn not used
DISPLAY
sp -2543.1
wp 38764.1
rfl 14159.4
rfp 11615.7
rp -72.8
lp 259.0
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sc 5
ph
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200
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40
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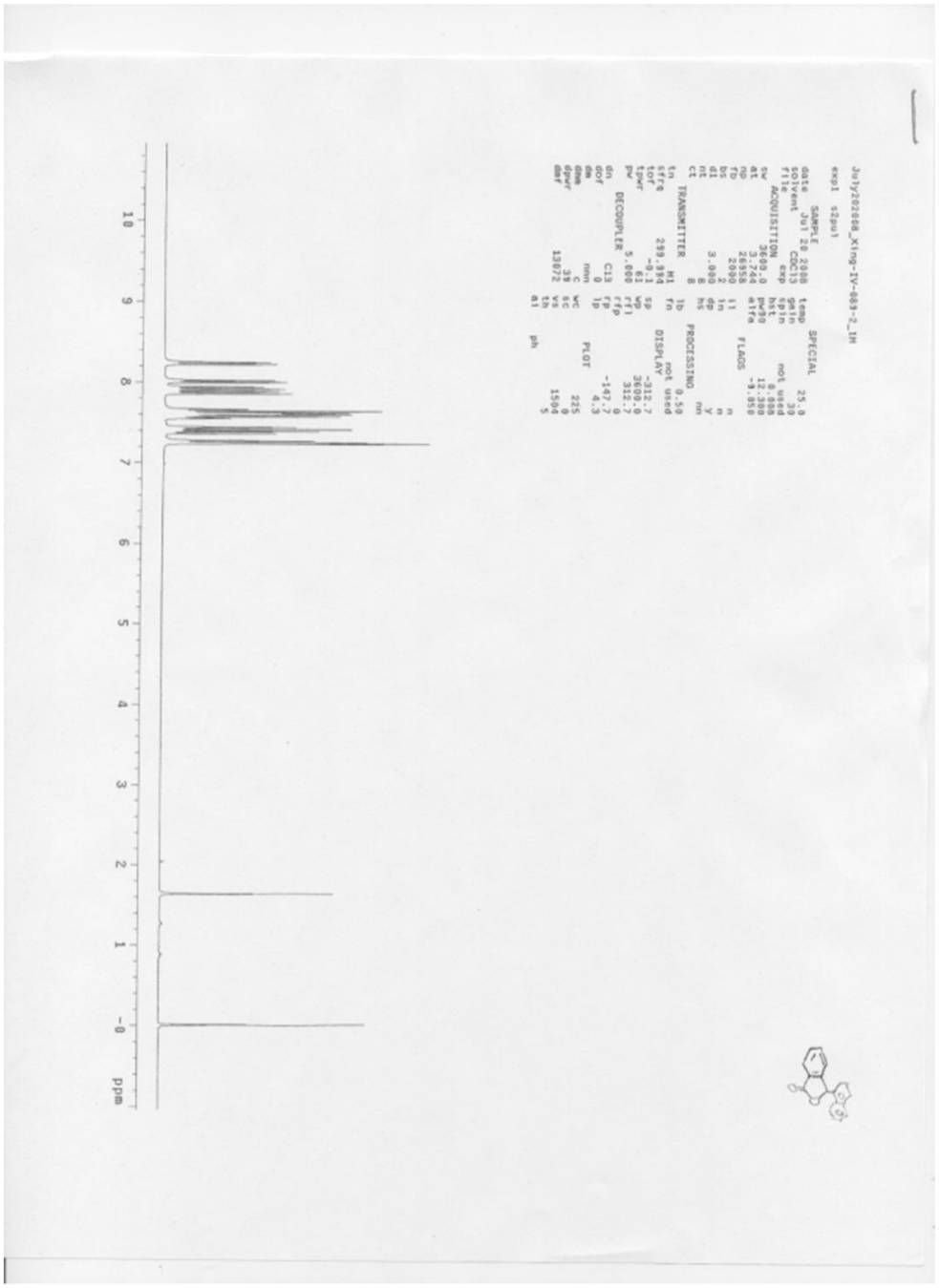
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6
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3
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1
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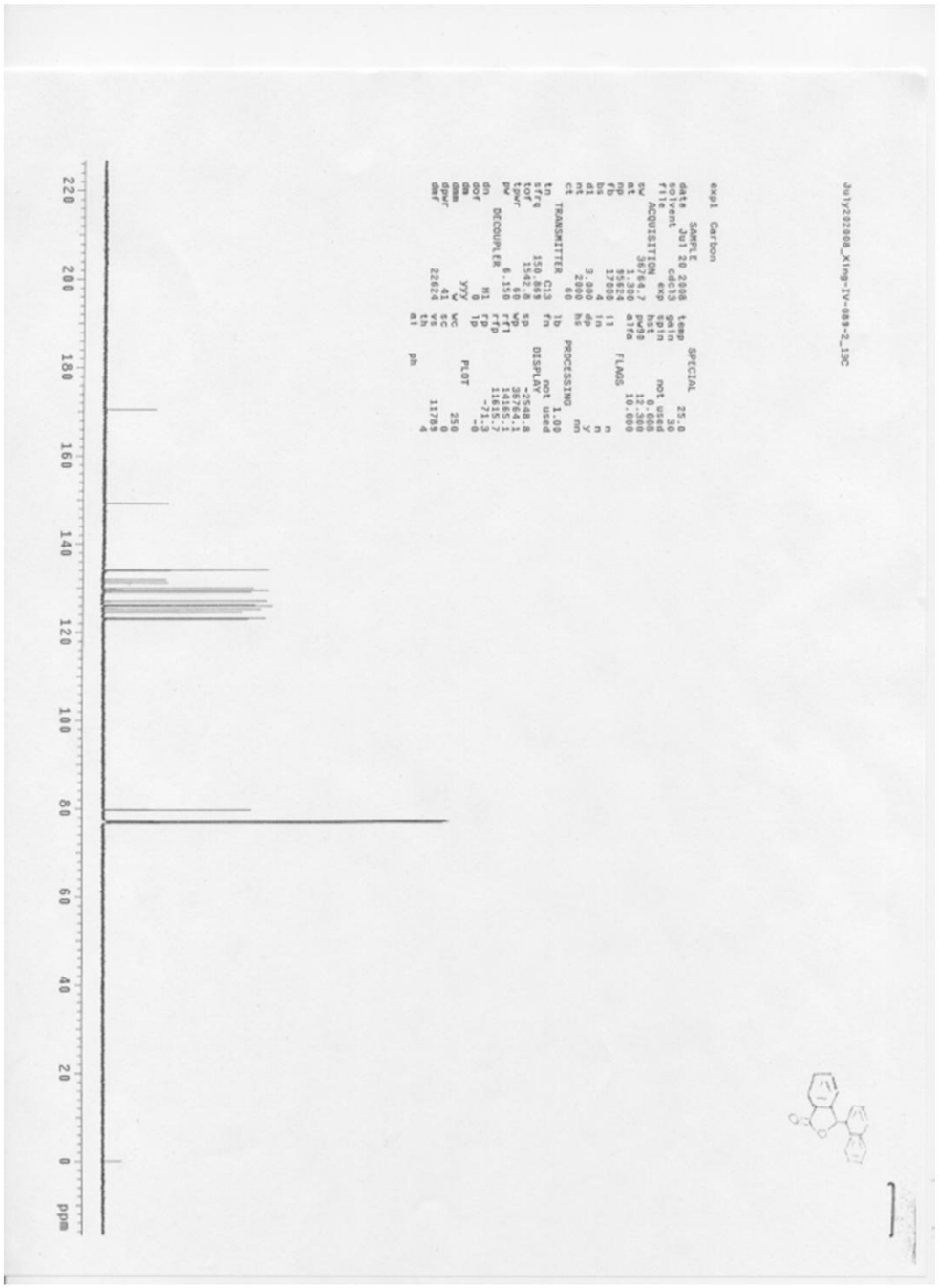
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expl Carbon
SAMPLE
date Jul 20 2008
solvent cdcl3
file acquisition
ACQUISITION
PROCESSING
DISPLAY
DECOUPLER
PLOT
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200
180
160
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60
40
20
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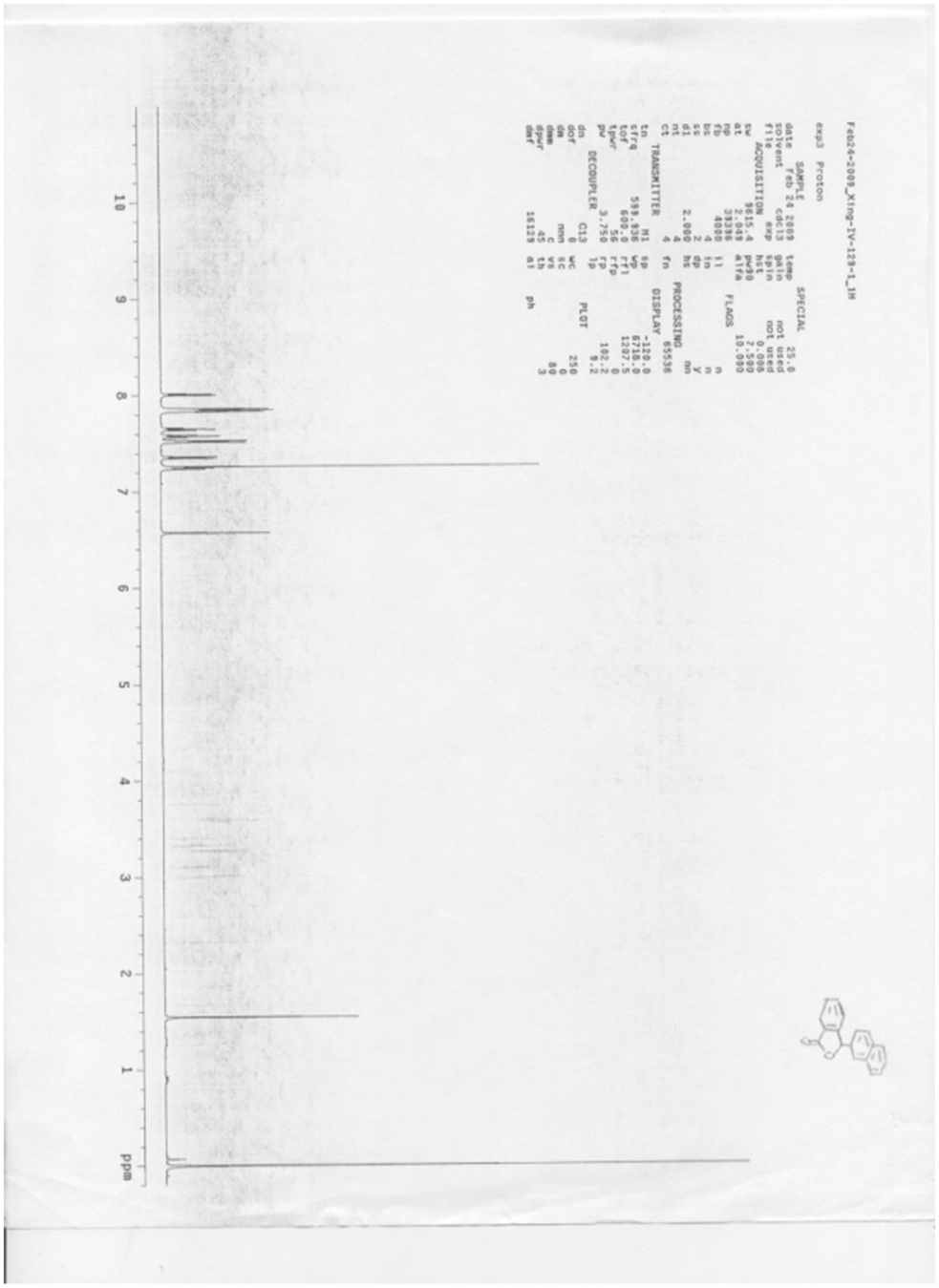
ppm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date Feb 24 2009
solvent cdcl3
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at 2.049
np 39386
fb 4096
bs 2
ss 4
d1 2.000
ct 4
TRANSMITTER
tn H1
sfrq 599.8
tof 600.0
tpwr 56
pw 3.750
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 45
dmf 16125
ph
temp 25.0
gain not used
exp not used
hst 0.000
pw90 7.500
alfa 10.000
FLAGS
ii n
in n
dp y
hs nn
PROCESSING
fn 65536
DISPLAY
sp -120.0
wp 7187.5
rfl 1287.5
rfp 0
rp 102.2
lp 9.2
PLOT
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sc 0
vs 80
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al
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9
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7
6
5
4
3
2
1
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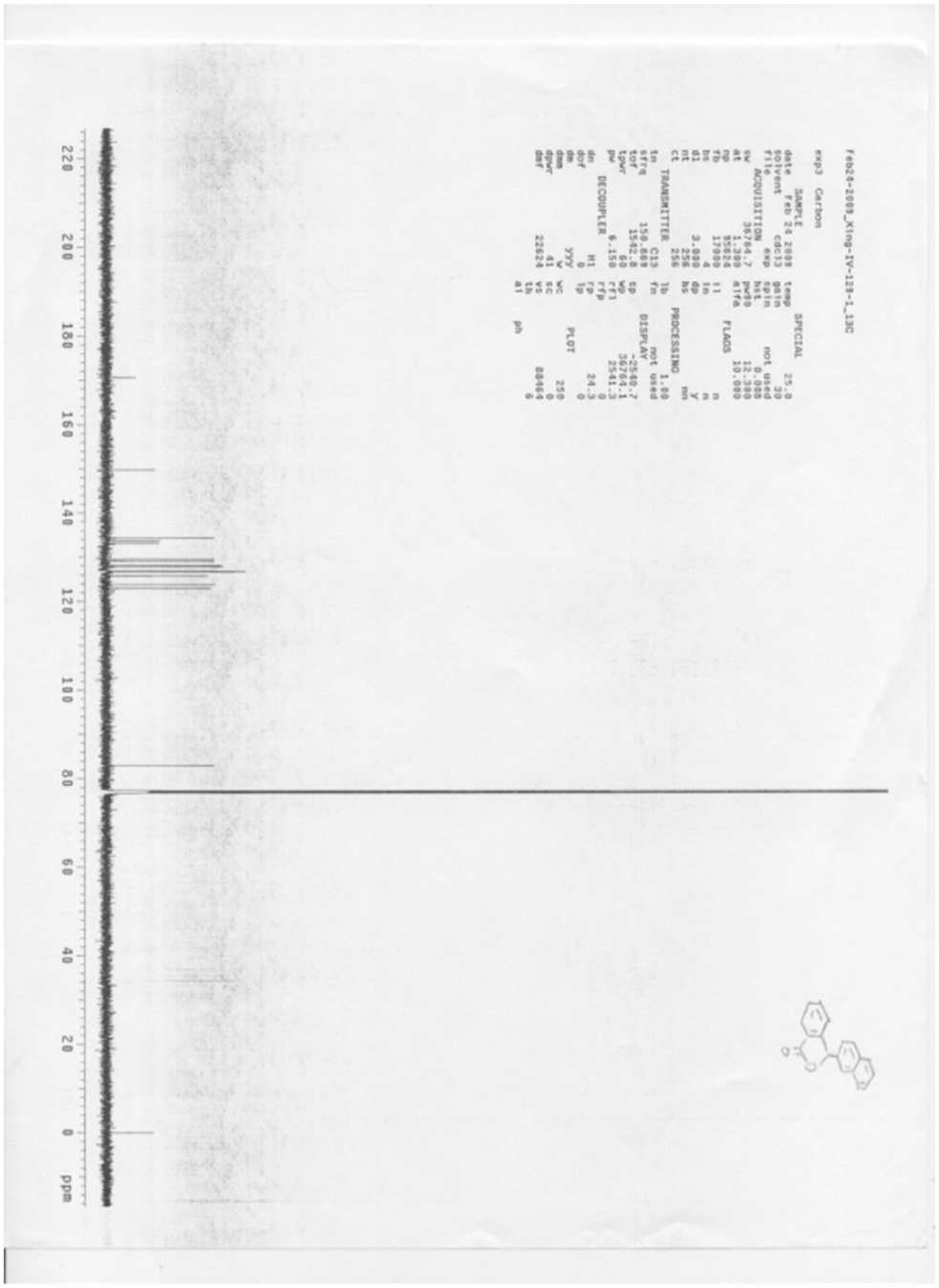
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exp3 Carbon
SAMPLE SPECIAL
date Feb 24 2009 temp 25.0
solvent cdcl3 gain 39
file hp not used
ACQUISITION PROCESSING
sw 38764.7 pw90 12.300
at 1.3900 alfa 10.000
fb 95824 FLAGS
bs 17904 il n
d1 3.000 dp y
nt 256 hs nn
ct 256
TRANSMITTER PROCESSING
tn C13 lb 1.00
sfrq 150.868 fn not used
tof 1542.8 sp DISPLAY -2540.7
tpwr 60 wp 35241.3
pw 6.150 rfl 2541.3
DECOUPLER rfp 0
dn H1 rp 24.3
dof 0 lp 0
dm yyy PLOT
dmm y wc 250
dmf 41 sc 0
dpwr 22624 vs 88464
dmf vs 0
ai
ph
220 200 180 160 140 120 100 80 60 40 20 0 ppm