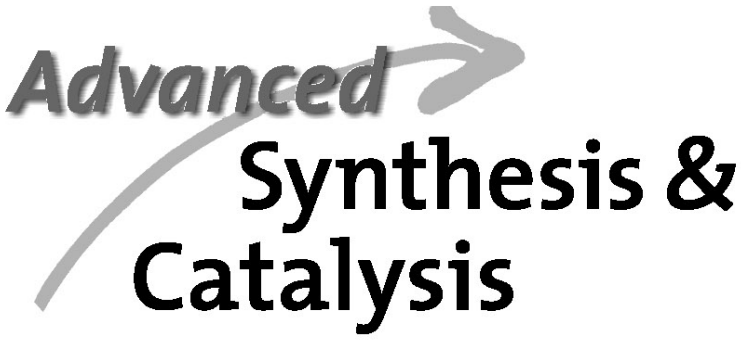

Supporting Information

Efficient Synthesis of 2-Substituted Indoles Based on $Pd(OAc)_2$/*t*-Bu_3P-Catalyzed Alkynylation/Amination of 1,2-Dihalobenzenes

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Department of Chemistry, College of Staten Island and Graduate Center of City University of New York, Staten Island, NY 10314, USA

Supporting Information

General:

NMR spectra were recorded on Varian 200 MHz or 600 MHz spectrometers. All yields reported refer to isolated yields (average of two runs) unless otherwise indicated, and the product purity was estimated to be greater than 95% as determined by ^{1}H NMR.

THF were dried with sodium/benzophenone. Cu(I)I, DPEPhos, Xantphos, and Buchwald's phosphines, PCy_3, *sec*-Bu_3P, and 1,3-bis(2,4,6-trimethylphenyl)imidazolium chloride were purchased from Strem and used directly. *t*-Bu_3P was obtained as a gift from Combiphos Catalysts, Inc., and palladium(II) acetate was obtained as a gift from Frontier Scientific, Inc and used directly. Other chemical reagents were purchase from Aldrich or Alfa Aesar and used directly.

General Procedures for $Pd(OAc)_2$/*t*-Bu_3P-Catalyzed Indole Synthesis from *o*-alkynylchlorobenzene:

Method A: Under N_2 atmosphere, to a mixture of *o*-alkynylchlorobenzene (1 mmol), *t*-BuOK (3 mmol), $Pd(OAc)_2$ (0.03 mmol) and *t*-Bu_3P (0.06 mmol) were added an amine (1.2 mmol) and toluene (2 ml). The resulting mixture was heated to 110-120^{o}C for 14 hours. After quenching with water, the reaction mixture was extracted with ethyl acetate and the organic layer was washed with brine. Rota-evaporation and flash chromatography on silica gel (hexane: ethyl acetate = 5 : 95 to 15 : 85) gave 2-substituted indoles as the product.

Method B: Under N_2 atmosphere, to a mixture of *o*-alkynylchlorobenzene (1 mmol), K_3PO_4 (3 mmol), $Pd(OAc)_2$ (0.03 mmol) and *t*-Bu_3P (0.06 mmol) were added an amine (1.2 mmol) and dimethylacetamide (2 ml). The resulting mixture was heated to 130^{o}C for 14 hours. After quenching with water, the reaction mixture was extracted with ethyl acetate and the organic layer was washed with brine. Rota-evaporation and flash chromatography on silica gel (hexane: ethyl acetate = 5 : 95 to 15 : 85) gave 2-substituted indoles as the product.

General procedure for one-pot, sequential preparation of 2-substituted indoles

Under N_2 atmosphere, to a mixture of 1-iodo-2-chlorobenze (1 mmol), $Pd(OAc)_2$ (0.03 mmol), CuI (0.1 mmol), *t*-BuOK (3 mmol), and *t*-Bu_3P (0.06 mmol) were added an alkyne (1.2 mmol) and toluene (2 ml). After the resulting mixture was heated to 110^{o}C for 1 hour, an amine (1.2 mmol) was added and the mixture was heated at 110^{o}C for

another 12 hrs. After quenching with water, the reaction was extracted with ethyl acetate and the organic layer was washed with brine. Rota-evaporation and flash chromatography on silica gel (hexane: ethyl acetate = 5 : 95 to 15 : 85) gave 2-substituted indoles as the product.

1-Benzyl-2-phenylindole: Following general procedure Method A or B, 270 mg (yield: 95%) was obtained as white solid. ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.698~7.649 (m, 1H), 7.740~7.351 (m, 6H), 7.283~7.116 (m, 6H), 7.054~7.006 (m, 2H), 6.656 (s, 1H), 5.732 (s, 2H). ^{13}C NMR (150.871 MHz, $CDCl_3$): δ 141.788, 138.179, 137.958, 132.674, 129.185, 128.704, 128.514, 128.290, 127.995, 127.121, 125.934, 121.876, 120.524, 120.138, 110.538,102.308, 47.704.

1,2-Diphenylindole: Following the general procedure Method B, 262 mg (yield: 97%) was obtained as white solid. ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.709~7.664 (m, 1H), 7.414~7.149 (m, 13H), 6.805 (s, 1H). ^{13}C NMR (150.871 MHz, $CDCl_3$): δ 140.679, 138.965, 138.474, 132.495, 129.199, 128.855, 128.230, 128.104, 127.991, 127.233, 127.131, 122.301, 120.668, 120.496, 110.579, 103.673.

1-(4-Methoxyphenyl)-2-phenylindole: Following the general procedure Method B, 258 mg (yield: 95%) was obtained as white solid. ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.695~7.561 (m, 1H), 7.289~7.131(m, 10H), 6.916 (d, J = 8.6 Hz, 2H), 6.780 (s, 1H). ^{13}C NMR (150.871 MHz, $CDCl_3$): δ 158.523, 140.851, 139.341, 132.590, 131.298, 129.132, 128.876, 128.125, 128.065, 127.198, 122.150, 120.458, 114.430, 110.624, 103.105, 55.420.

1-Octyl-2-phenylindole: Following the general procedure Method A, 308 mg (yield: 99%) was obtained as colorless oil. ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.630 (d, J = 7.8 Hz, 1H), 7.465~7.353 (m, 6H), 7.257~7.083(m, 2H), 6.509 (s, 1H), 4.122 (t, J = 7.7 Hz, 2H), 1.677 (t, J = 7.0 Hz, 2H), 1.151(m, 9H), 0.847 (t, J = 7.0 Hz, 3H). ^{13}C NMR (150.871 MHz, $CDCl_3$): δ 141.324, 137.326, 133.317, 129.417, 128.416, 128.814, 127.840, 121.416, 120.521, 119.675, 110.021, 101.995, 43.923, 31.699, 29.881, 29.052, 28.993, 26.721, 22.579, 14.059.

1-Benzyl-2-hexylindole: Following the general procedure Method A in half mmol scale, 147 mg (yield: 99%) was obtained as colorless oil. ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.588~7.543 (m, 1H), 7.277~7.042(m, 6H), 6.948~6.903 (m, 2H), 6.335 (s, 1H), 5.277 (s, 2H), 2.641 (t, J = 7.6 Hz, 2H), 1.669 (m, 2H), 1.430~1.216 (m, 6H), 0.861 (t, J = 6.5 Hz,3H). ^{13}C NMR (150.871 MHz, $CDCl_3$): δ 141.391, 137.968, 137.108, 128.662, 128.142, 127.142, 125.864, 120.710, 119.752, 119.432, 109.238, 99.327, 46.310, 31.598, 29.038, 28.371, 26.683, 22.540, 14.048.

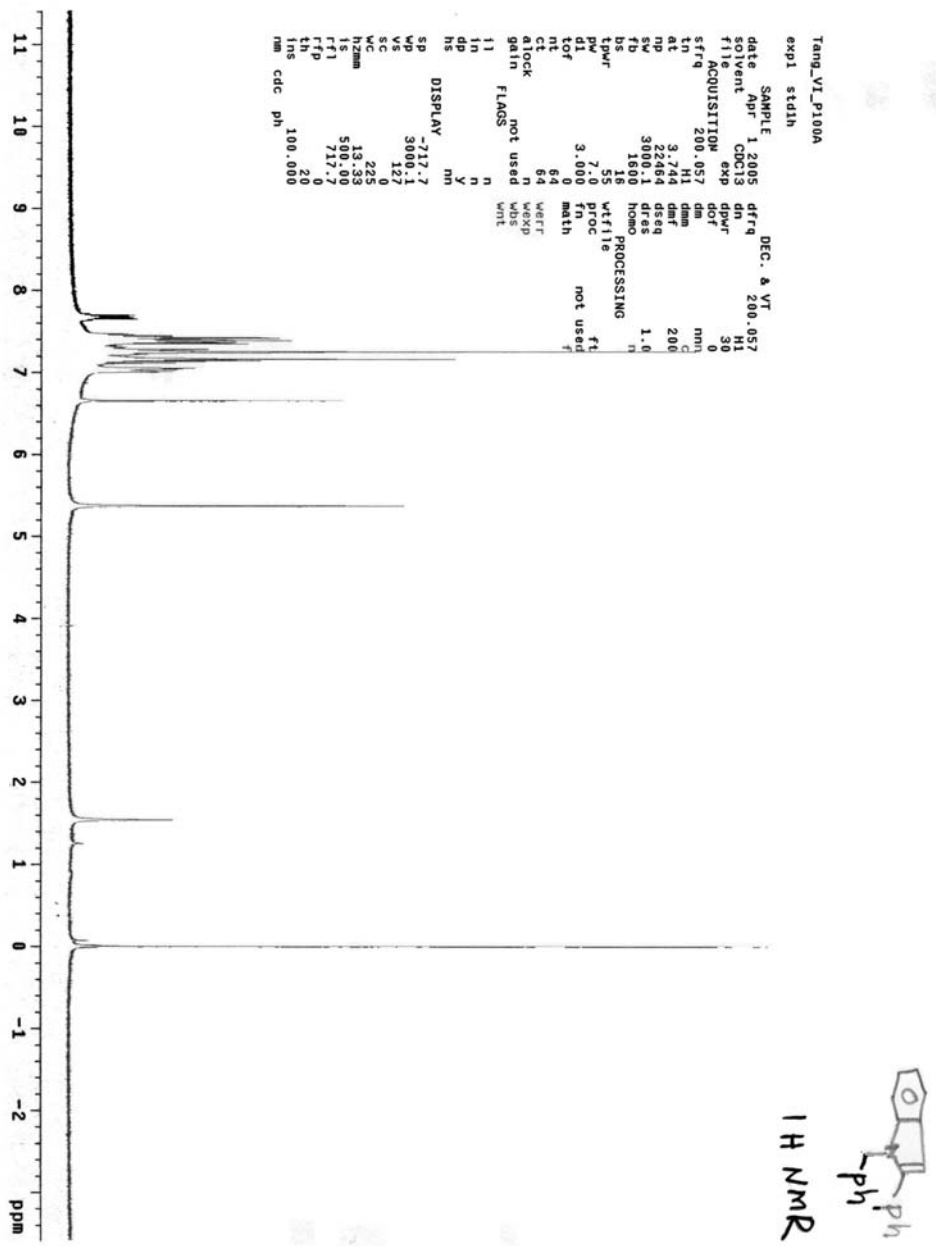
2-Hexyl -1-octylindole: Following the general procedure Method A in half mmol scale, 115 mg (yield: 74%) was obtained as colorless oil. ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.524 (d, J=9 Hz, 1H), 7.280~6.997 (m, 3H), 6.235 (s, 1H), 4.024 (t, J = 7.6 Hz, 2H), 2.700 (t, J = 7.6 Hz, 2H), 1.819~1.670 (m, 4H), 1.476~1.261 (m, 16H), 0.941~0.841 (m, 6H). ^{13}C NMR (150.871 MHz, $CDCl_3$): δ 141.054, 136.490, 128.061, 120.247, 119.696, 118.994,

109.003, 98.520, 43.137, 31.787, 31.717, 30.183, 29.319, 29.228, 29.186, 28.536, 27.108, 26.714, 22.614, 14.083, 14.059.

2-Butyl -1-octylindole: Following the general procedure Method A in half mmol scale, 128 mg (yield: 90%) was obtained as colorless oil. ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.521 (d, J = 8.8 Hz, 1H), 7.234 (d, J = 7.8 Hz, 1H), 7.164~6.994 (m, 2H), 6.234 (s, 1H), 4.017 (t, J = 7.6 Hz, 2H), 2.702 (t, J = 7.6 Hz, 2H), 1.770~1.656 (m, 4H), 1.522~1.262 (m, 12H), 1.011~0.844(m, 6H). ^{13}C NMR (150.871 MHz, $CDCl_3$): δ 140.963, 136.494, 128.061, 120.247, 119.689, 118.994, 108.992, 98.537, 43.112, 31.780, 30.681, 30.172, 29.312, 29.175, 27.093, 26.384, 22.610, 22.593, 14.052, 13.939.

1-Benzyl-2-butylindole: Following the general procedure Method A, 224 mg (yield: 85%) was obtained as colorless oil. ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.592 ~7.529 (m, 1H), 7.258~7.049 (m, 6H), 6.970~6.930 (m, 2H), 6.342 (s, 1H), 5.318 (s, 2H), 2.666 (t, J = 7.6 Hz, 2H), 1.671 (m, 2H), 1.398 (m, 2H), 0.901 (t, J = 7.2 Hz, 3H). ^{13}C NMR (150.871 MHz, $CDCl_3$): δ 141.316, 137.928, 137.065, 128.632, 128.095, 127.112, 125.827, 120.677, 119.712, 119.396, 109.205, 99.291, 46.281, 30.480, 26.344, 22.399, 13.808.

1-Hexyldecyl-2-phenyl indole: Following "General procedure for one-pot, sequential preparation of 2-substituted indoles", 196mg (yield: 47%) was obtained as colorless oil. ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.611 (d, J = 7.2 Hz, 1H), 7.482~7.327 (m, 5H), 7.237~7.068(m, 3H), 6.496(s, 1H), 4.089 (t, J = 7.6 Hz, 2H), 1.648 (t, J = 6.6 Hz, 2H), 1.256~1.129 (m, 26H), 0.876(t, J = 6 Hz, 3H). ^{13}C NMR (50.310 MHz, $CDCl_3$): δ 141.695, 137.767, 133.740, 129.812, 128.819, 128.637, 128.219, 121.842, 120.962, 120.105, 110.429, 102.451, 44.293, 32.371, 30.286, 30.119, 30.051, 29.945, 29.823, 29.459, 27.131, 23.135, 14.565.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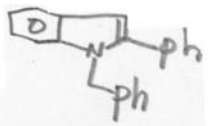

^{13}C NMR

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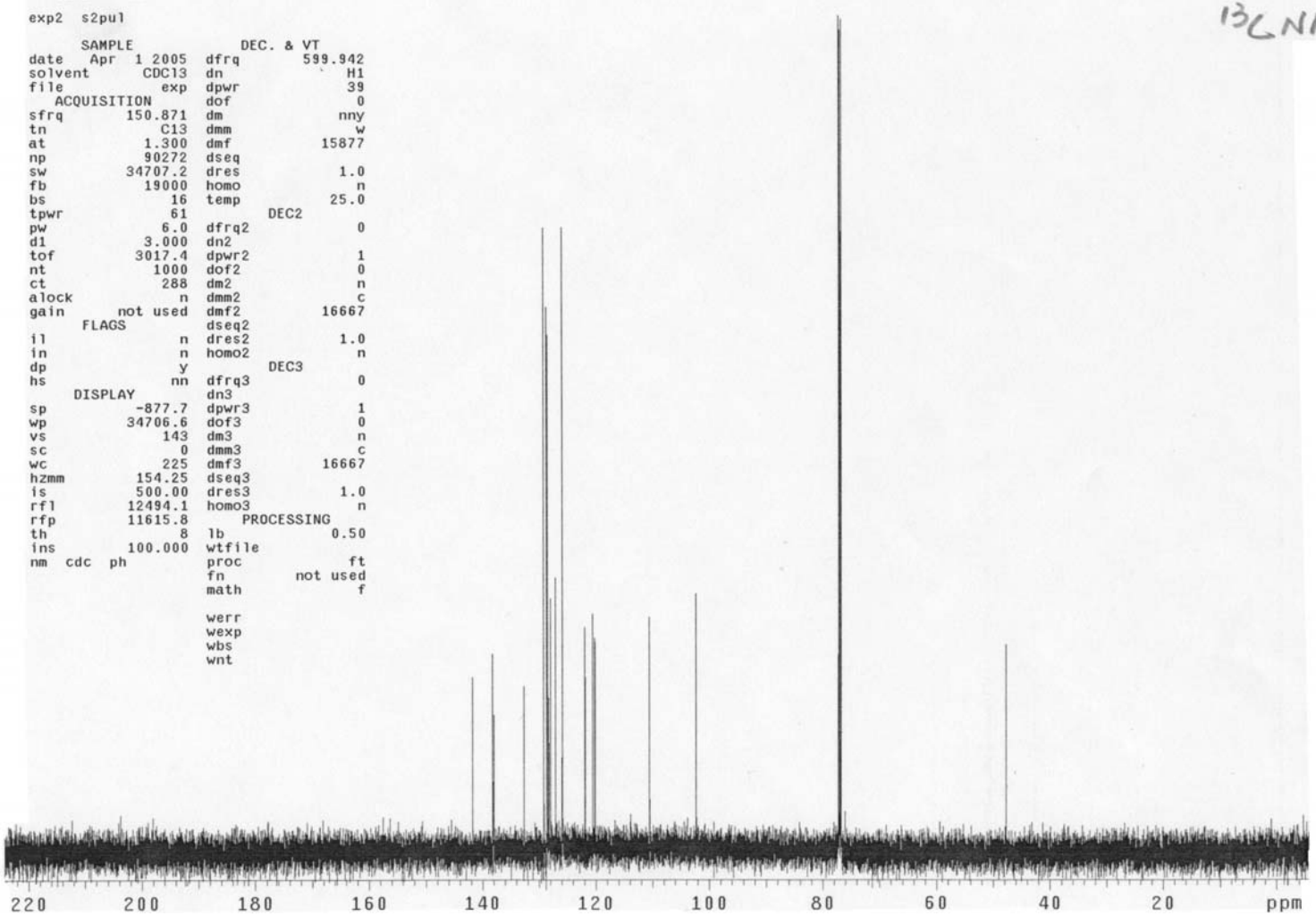

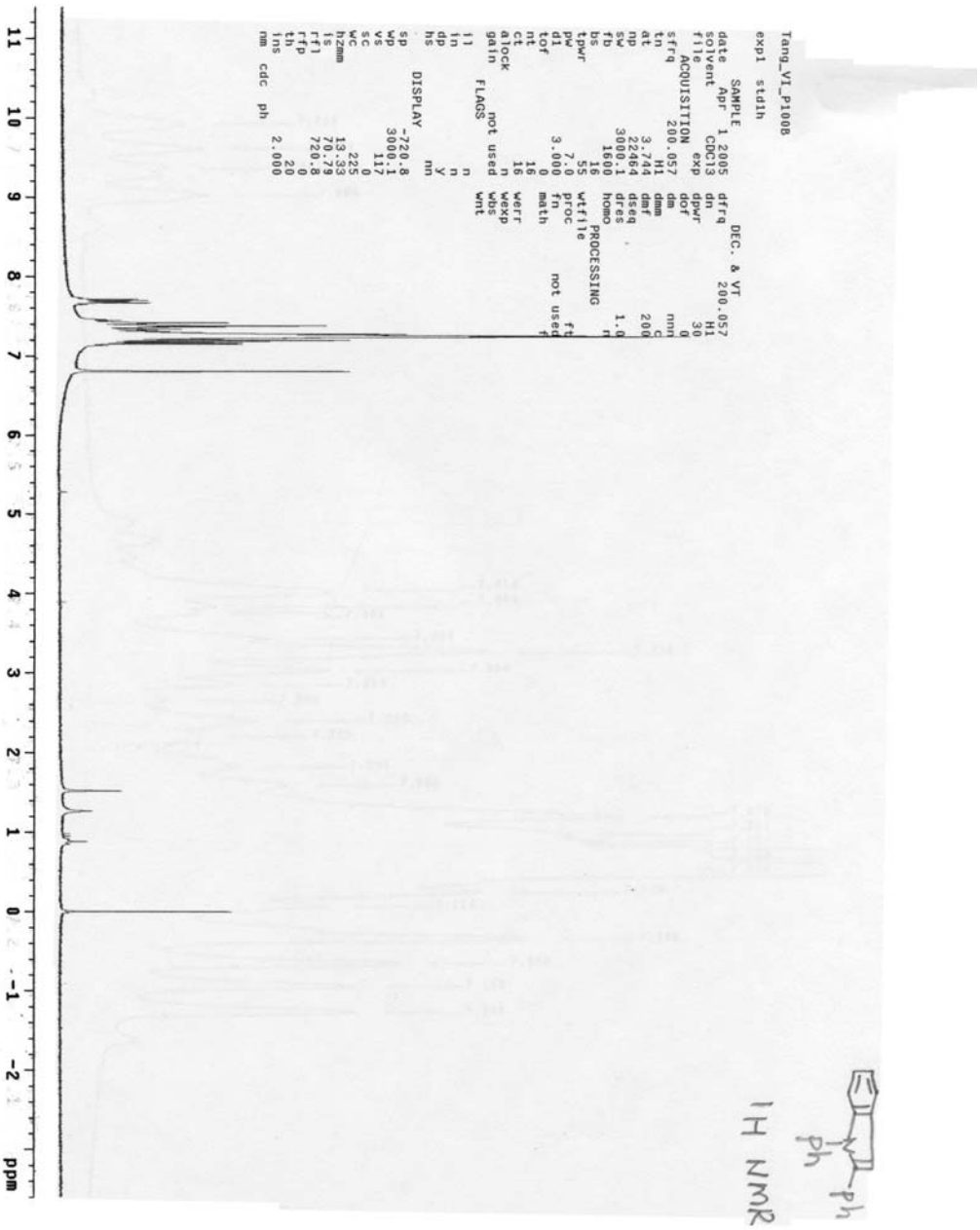
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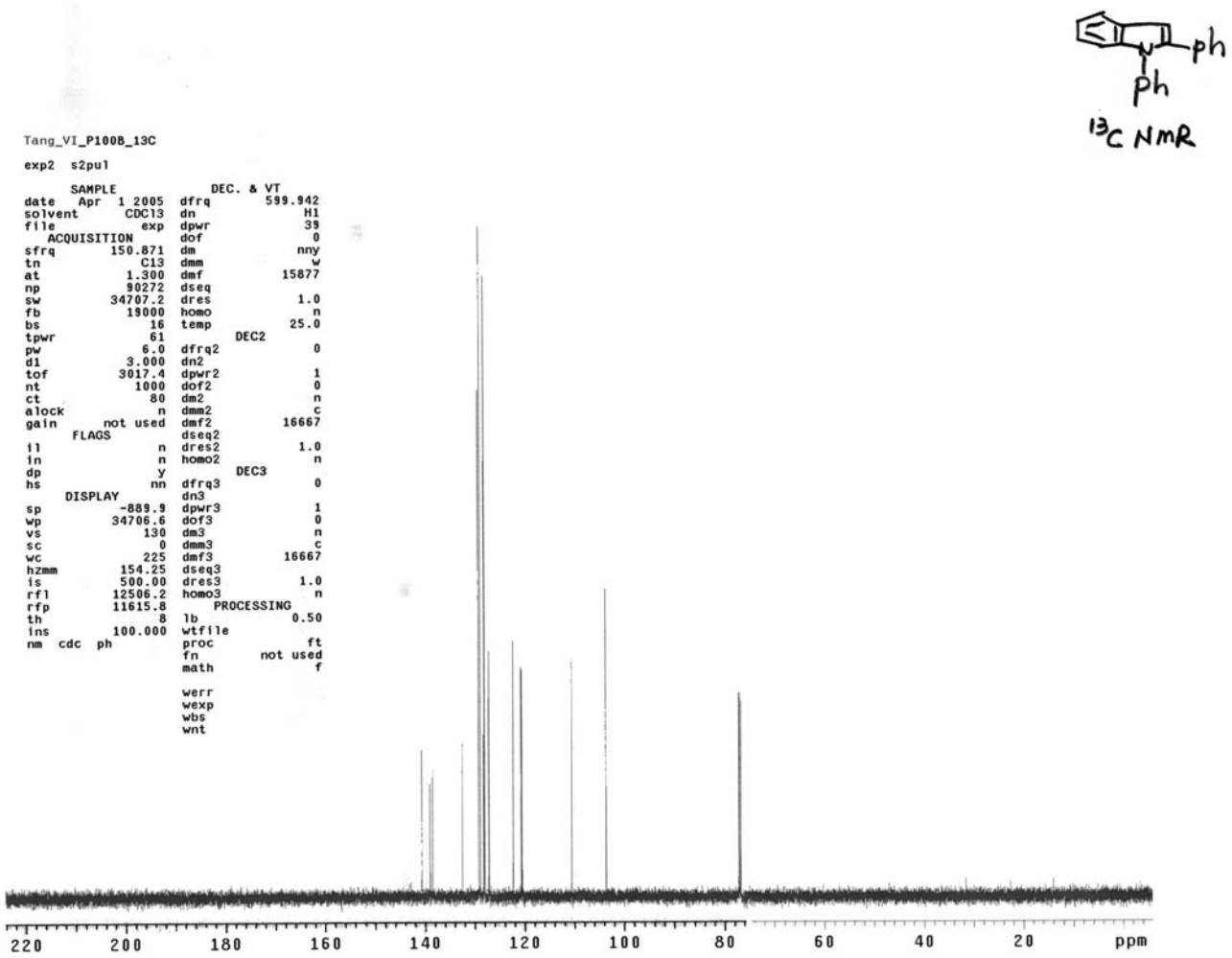
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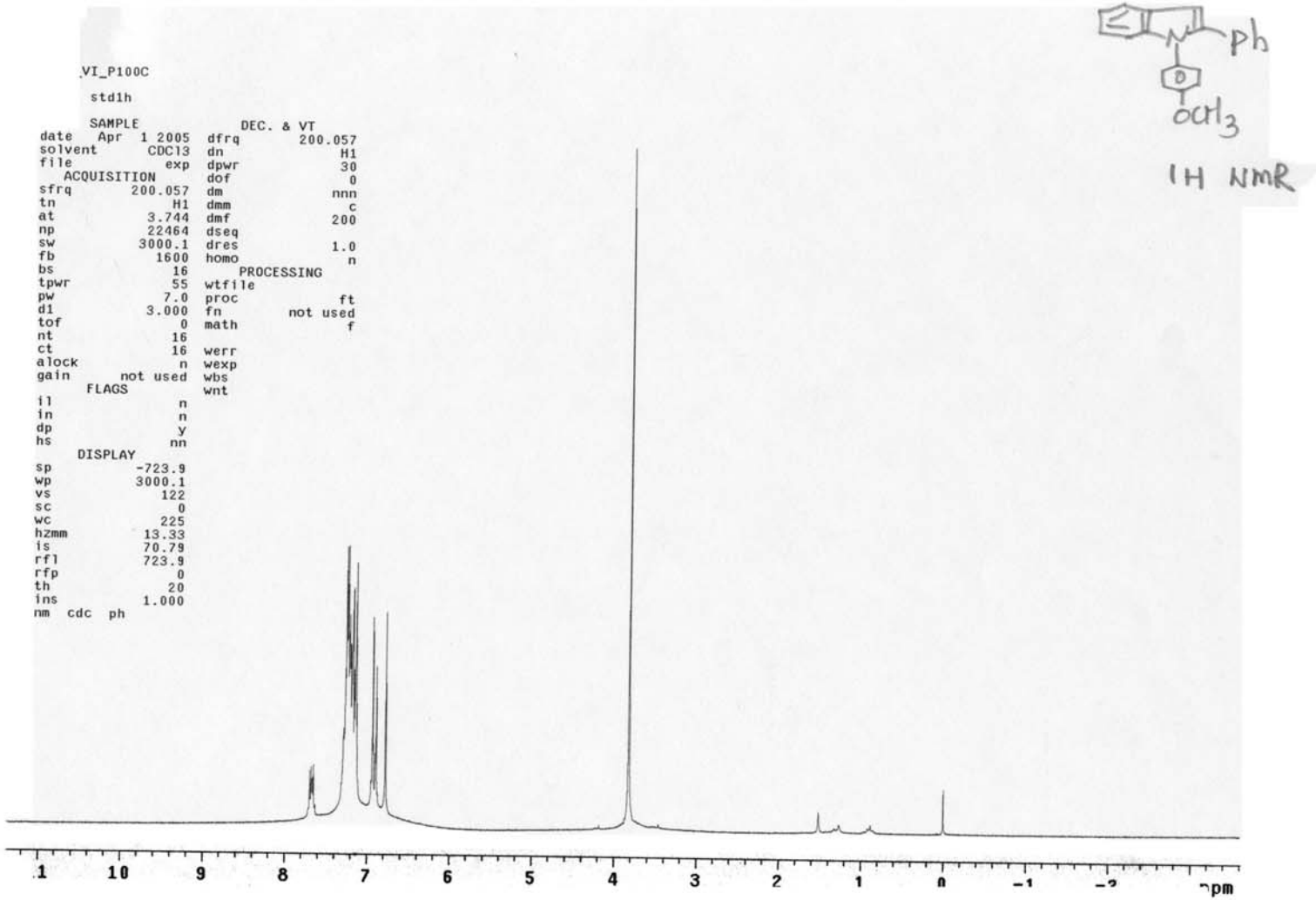
^{13}C NMR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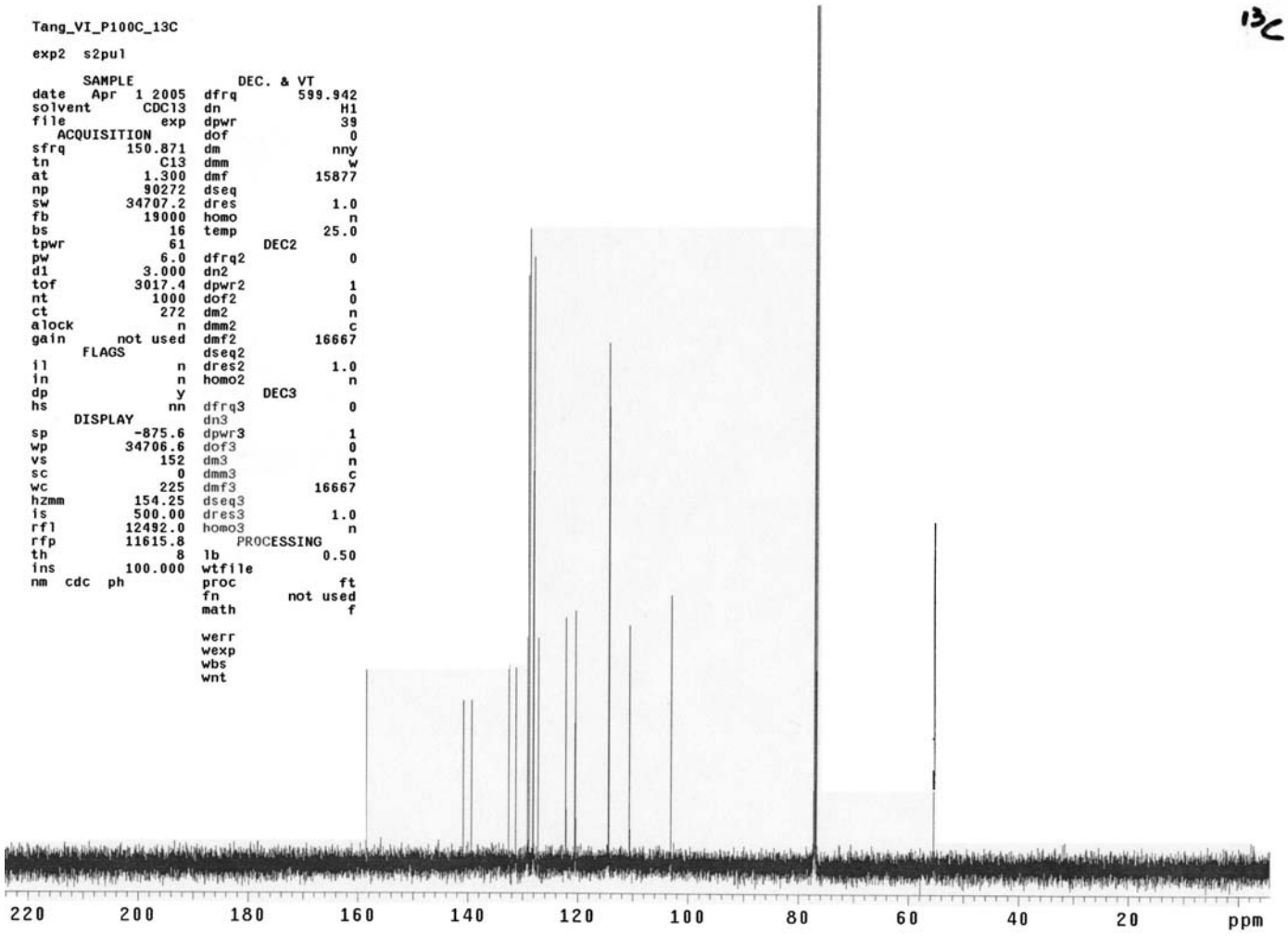

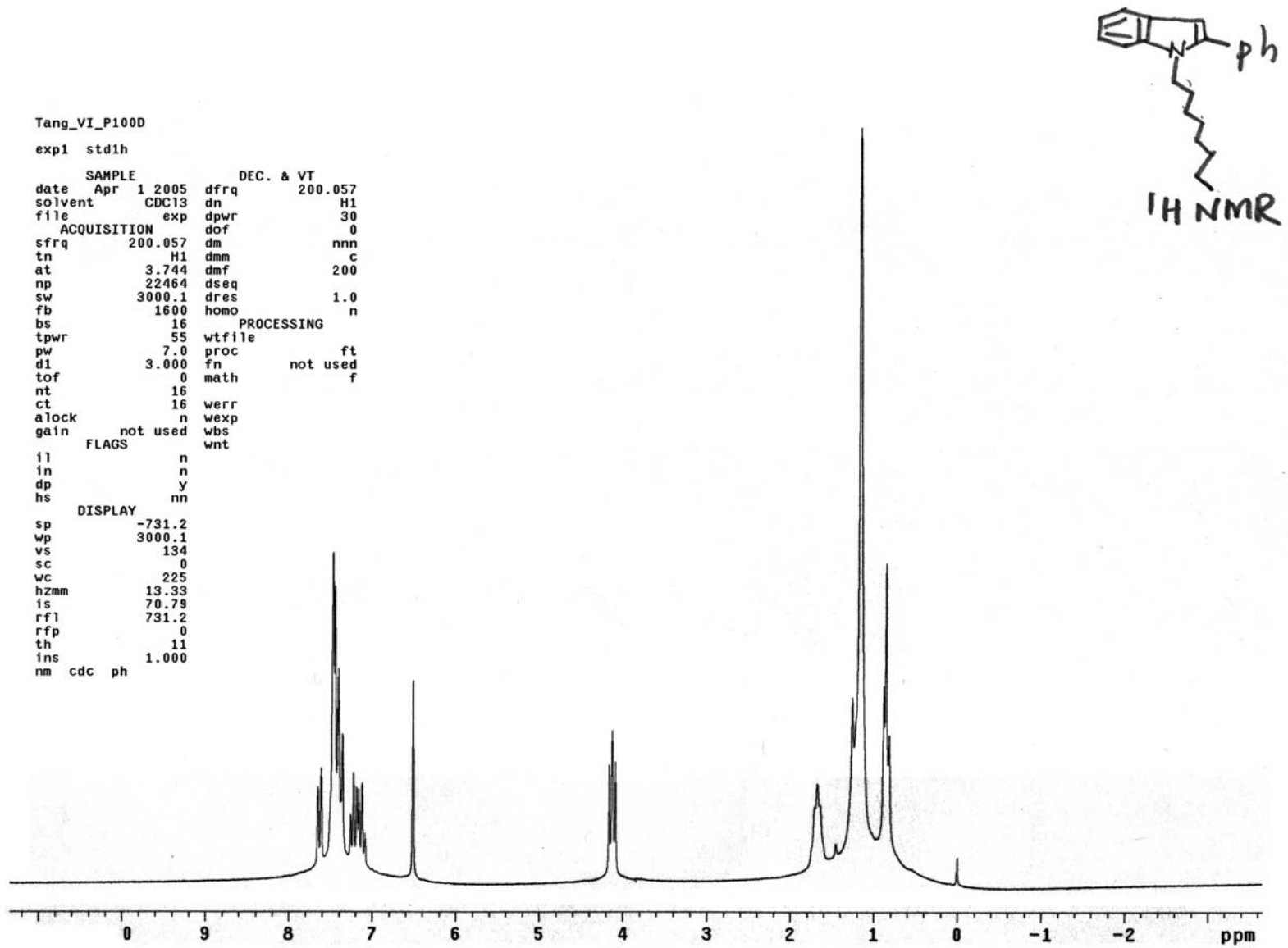
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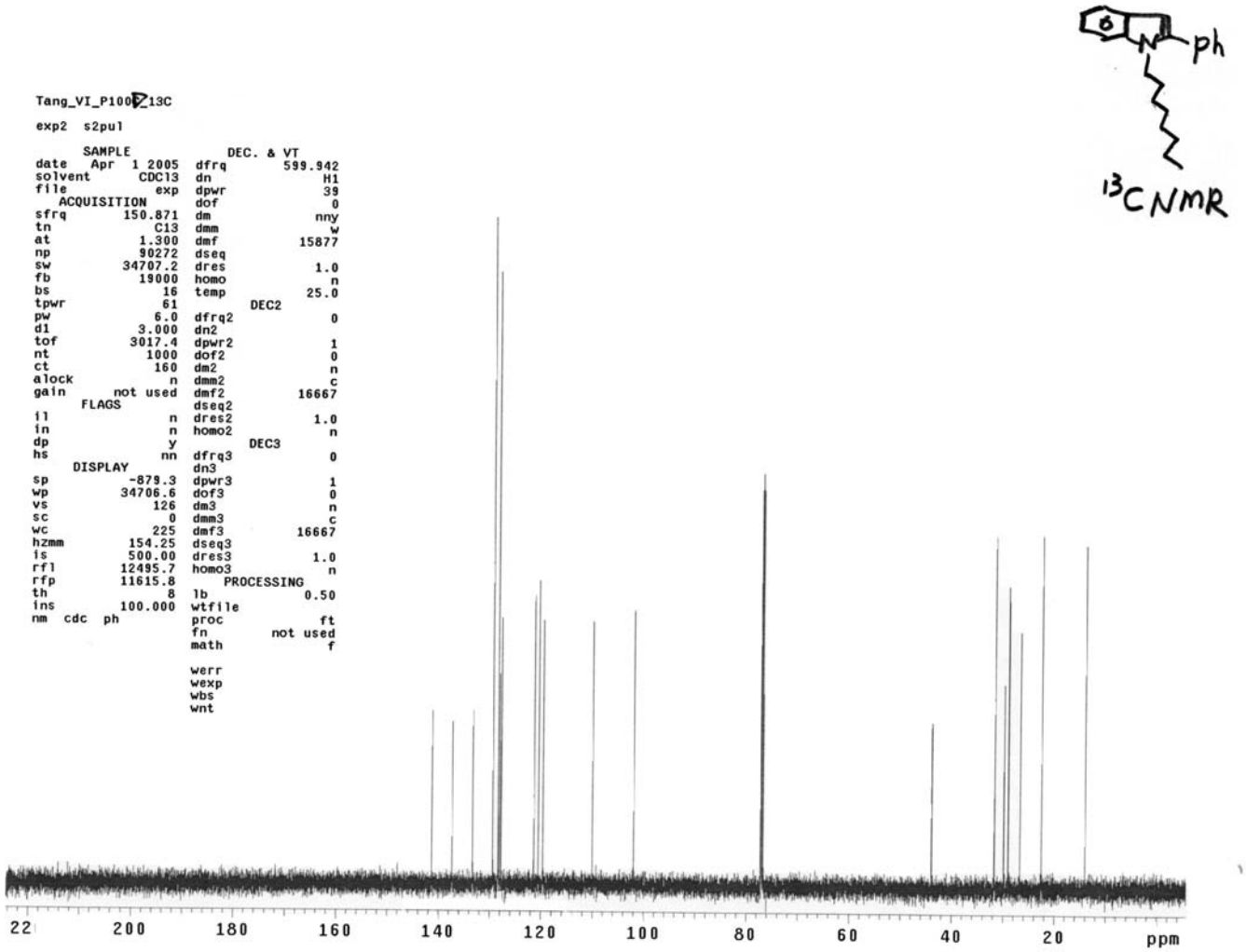
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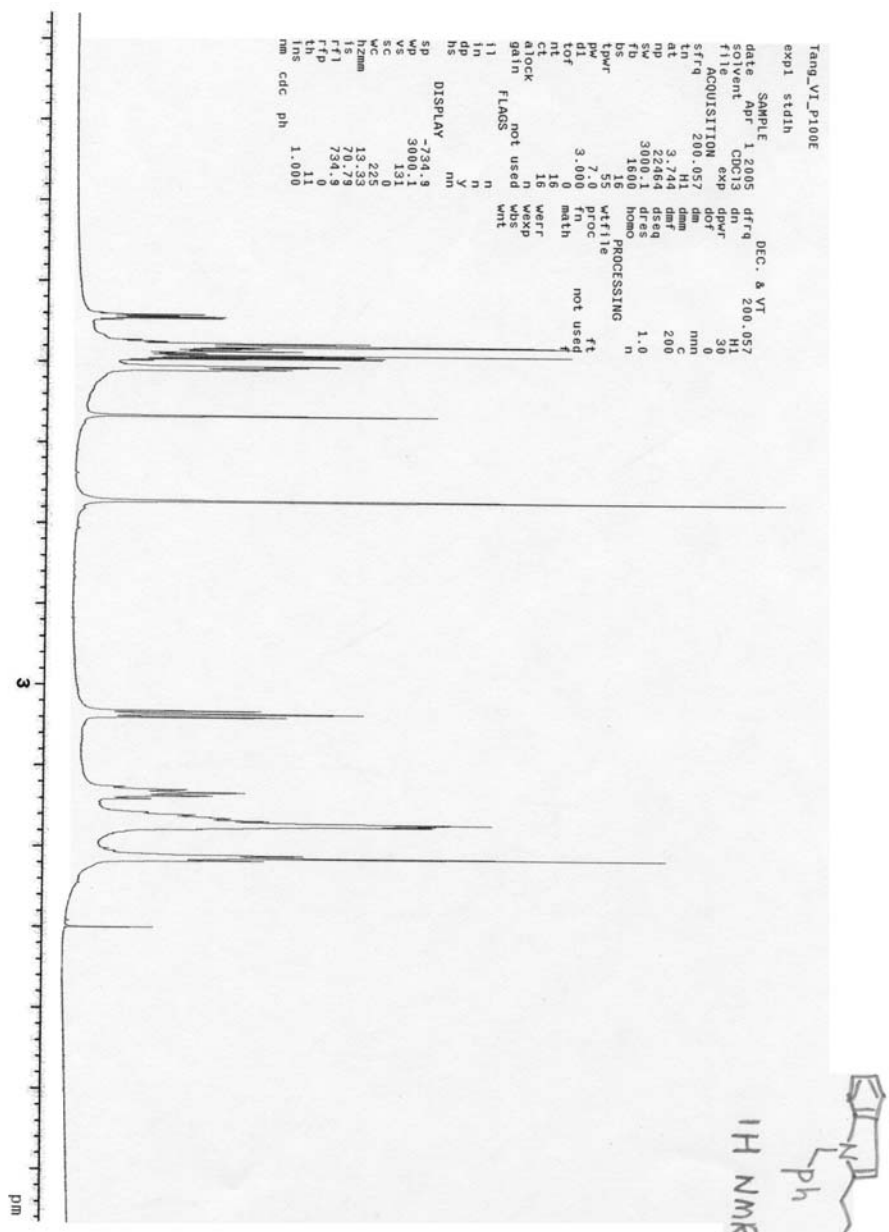
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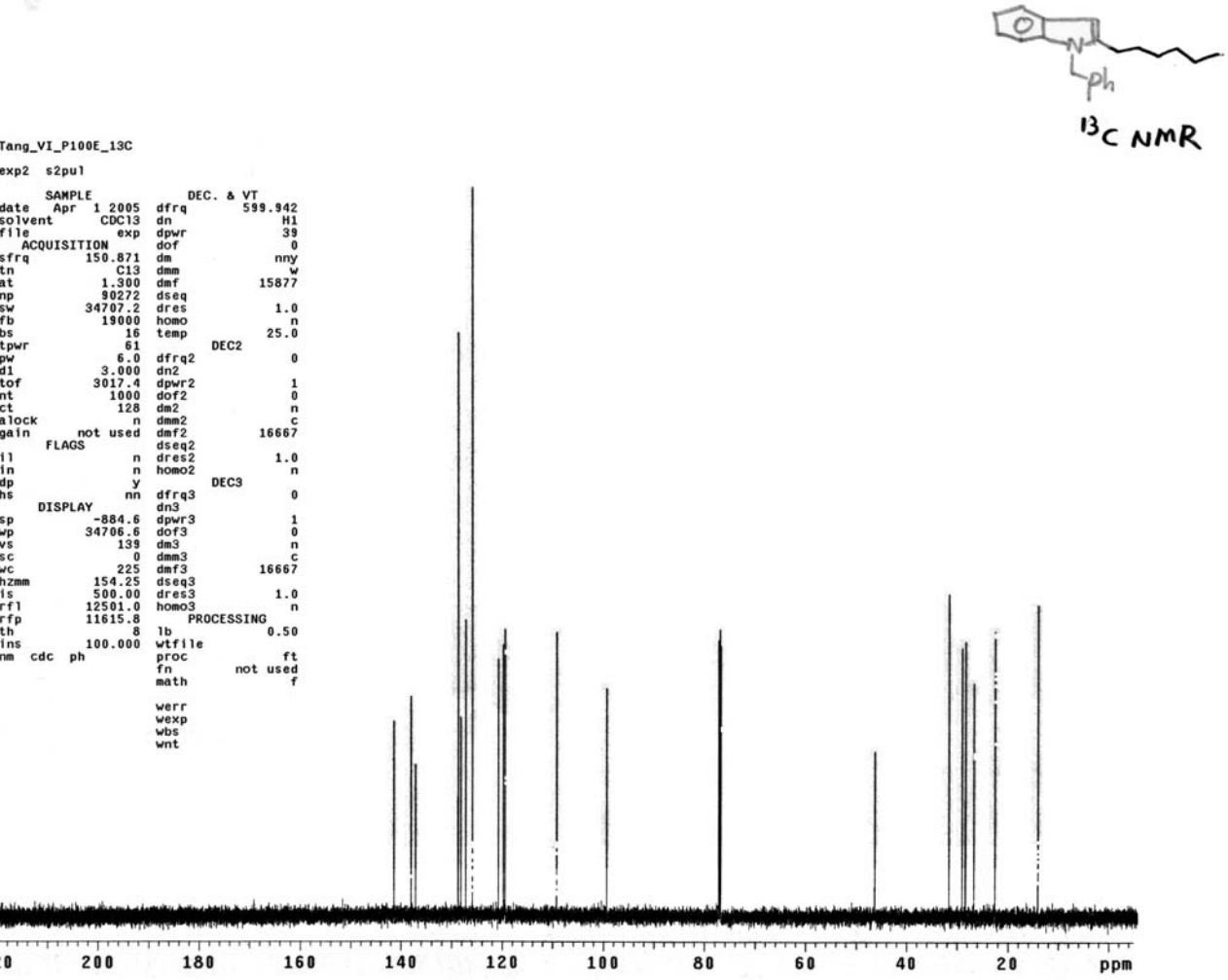
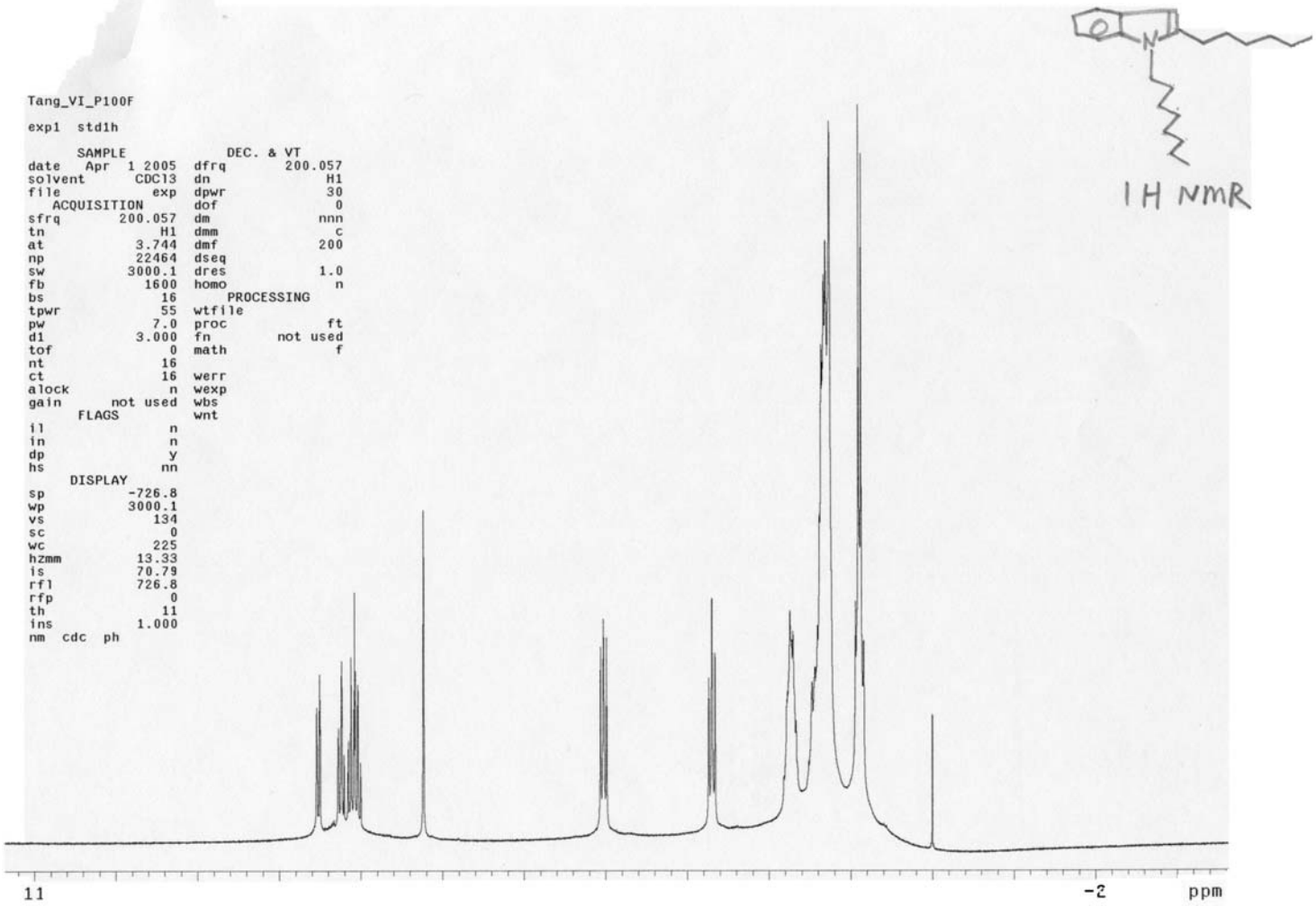
tof 0
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -734.9
wp 3000.1
vs 131
sc 0
wc 225
hzmm 13.33
is 70.79
rfl 734.9
rfp 0
th 11
ins 1.000
nm cdc ph
DEC. & VT
dfrq 200.057
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
3
ppm

N
Ph

1H NMR

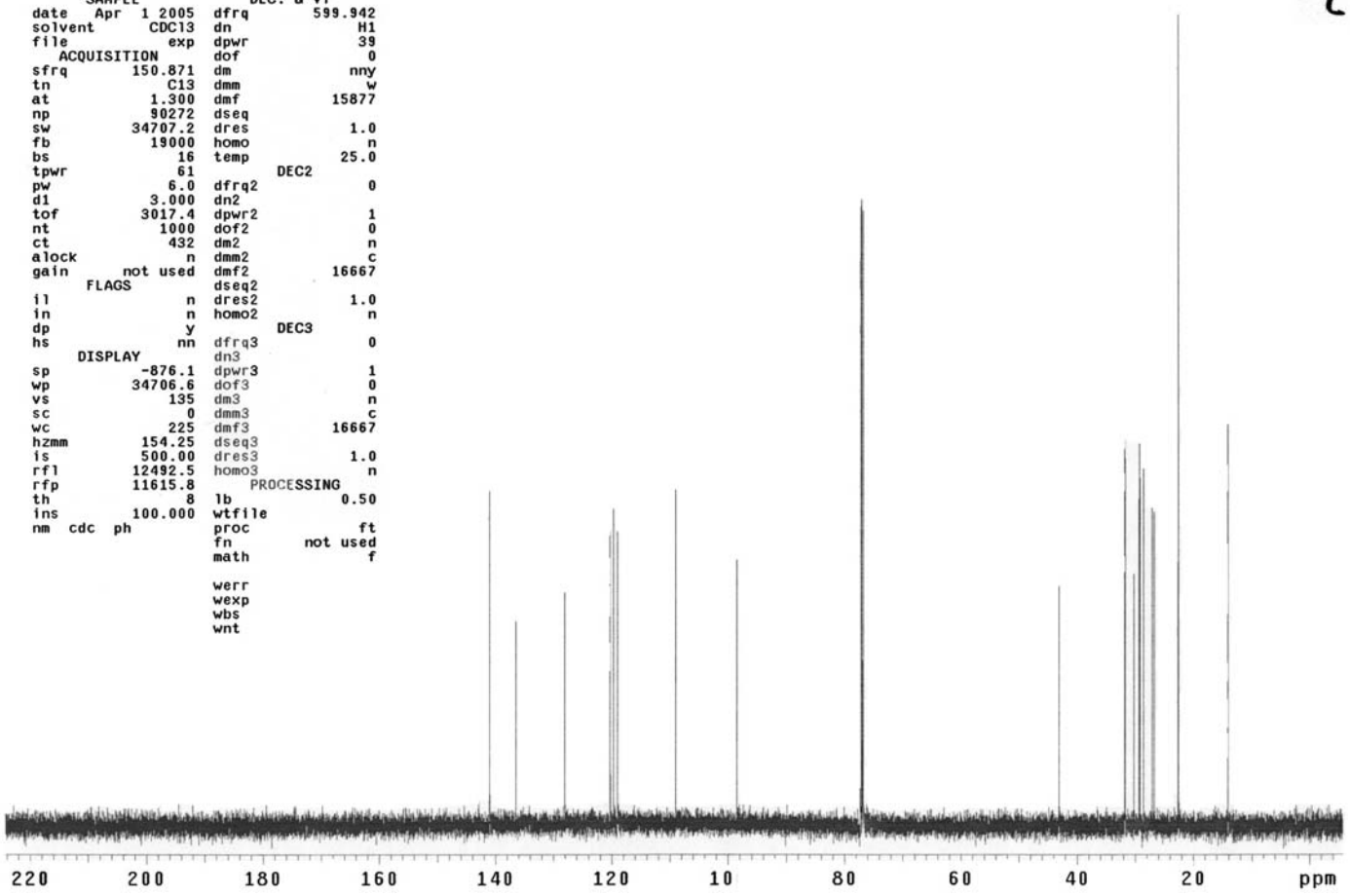

Ph
13C NMR
Tang_VI_P100E_13C
exp2 s2pul
SAMPLE
date Apr 1 2005
solvent CDCl3
file exp
ACQUISITION
sfrq 150.871
tn C13
at 1.300
np 90272
sw 34707.2
fb 19000
bs 16
tpwr 61
pw 6.0
d1 3.000
tof 3017.4
nt 1000
ct 128
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -884.6
wp 34706.6
vs 139
sc 0
wc 225
hzmm 154.25
is 500.00
rfl 12501.0
rfp 11615.8
th 8
ins 100.000
nm cdc ph
DEC. & VT
dfrq 599.942
dn H1
dpwr 39
dof 0
dm nny
dmm w
dmf 15877
dseq
dres 1.0
homo n
temp 25.0
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 16667
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 16667
dseq3
dres3 1.0
homo3 n
PROCESSING
lb 0.50
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
220
200
180
160
140
120
100
80
60
40
20
ppm

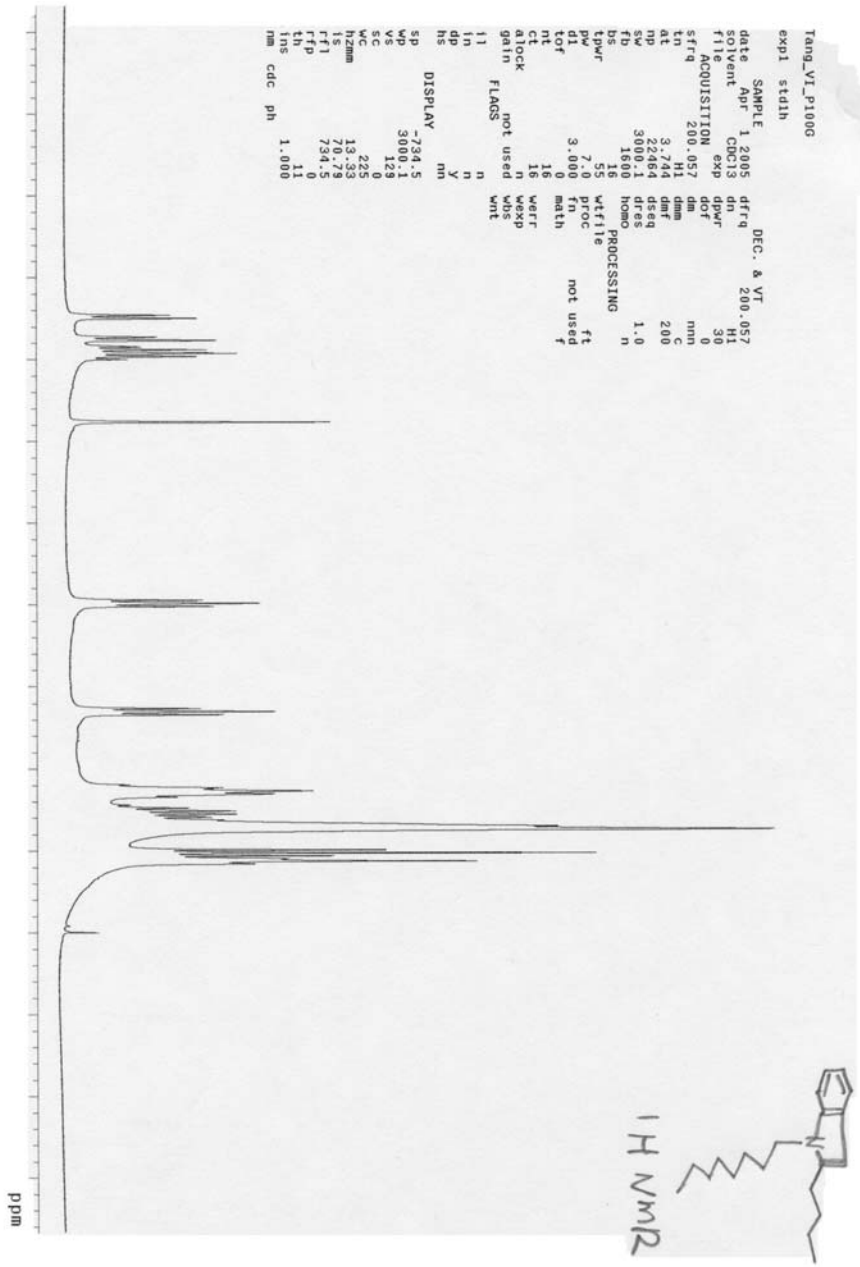

Tang_VI_P100F
exp1 std1h
SAMPLE
date Apr 1 2005
solvent CDCl3
file exp
ACQUISITION
sfrq 200.057
tn H1
at 3.744
np 22464
sw 3000.1
fb 1600
bs 16
tpwr 55
pw 7.0
d1 3.000
tof 0
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -726.8
wp 3000.1
vs 134
sc 0
wc 225
hzmm 13.33
is 70.79
rfl 726.8
rfp 0
th 11
ins 1.000
nm cdc ph
DEC. & VT
dfrq 200.057
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
1H NMR
11
-2
ppm

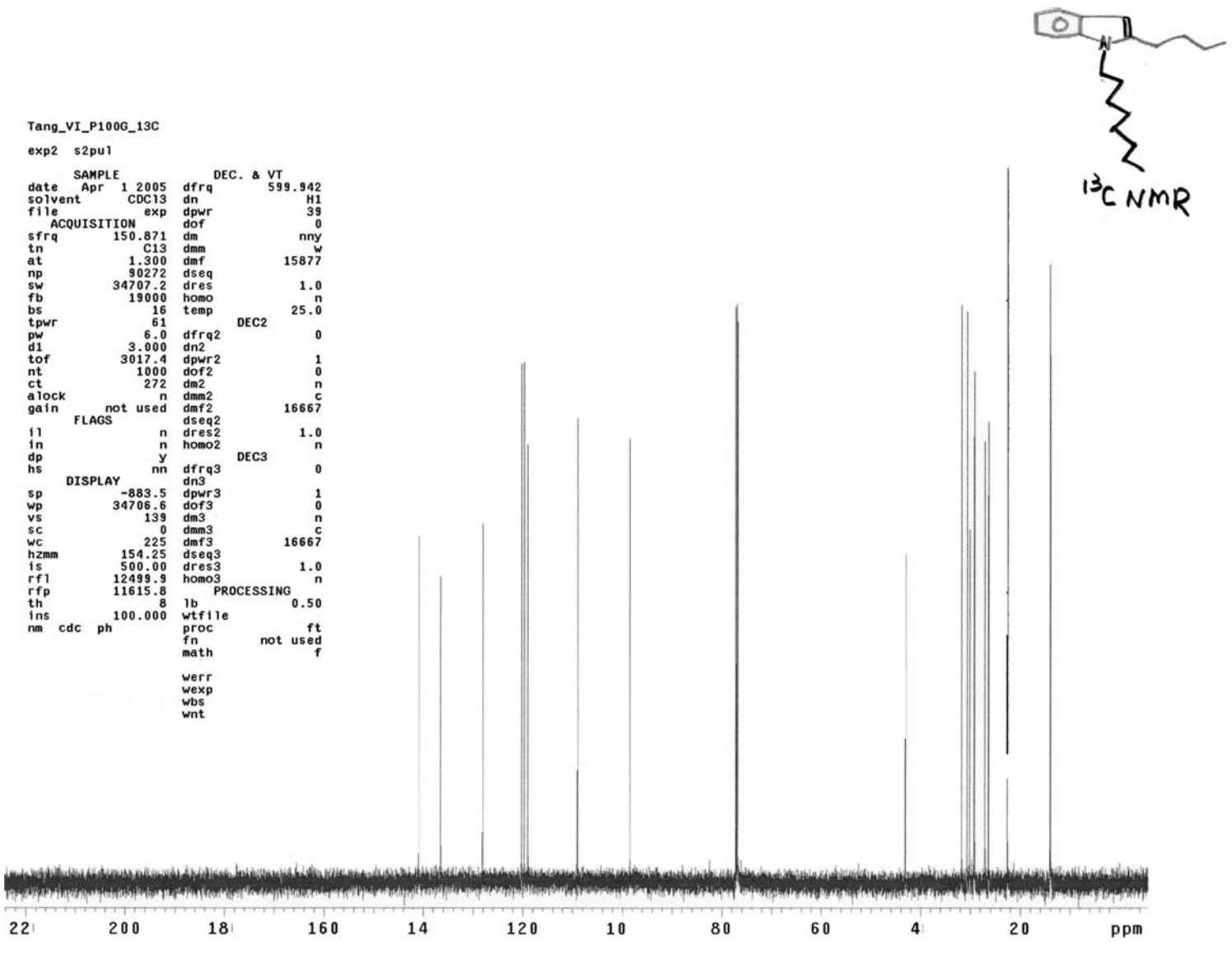
Tang_VI_P100F_13C

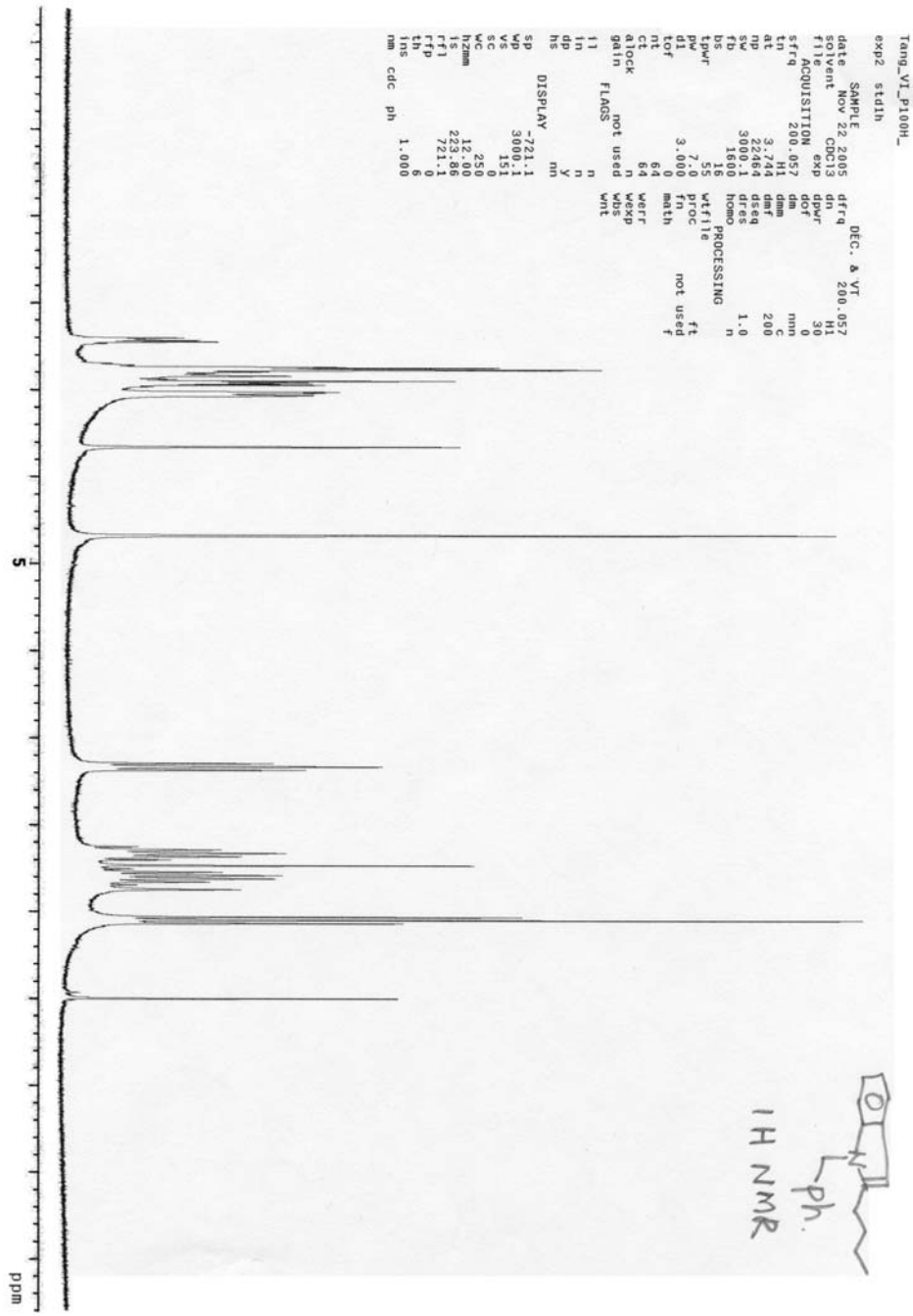
exp2 s2pul

SAMPLE		DEC. & VT	
date	Apr 1 2005	dfrq	599.942
solvent	CDCl3	dn	H1
file	exp	dpwr	39
ACQUISITION		dof	0
sfrq	150.871	dm	nny
tn	C13	dmm	w
at	1.300	dmf	15877
np	90272	dseq	
sw	34707.2	dres	1.0
fb	19000	homo	n
bs	16	temp	25.0
tpwr	61	DEC2	
pw	6.0	dfrq2	0
d1	3.000	dn2	
tof	3017.4	dpwr2	1
nt	1000	dof2	0
ct	432	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	16667
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-876.1	dpwr3	1
wp	34706.6	dof3	0
vs	135	dm3	n
sc	0	dmm3	c
wc	225	dmf3	16667
hzmm	154.25	dseq3	
is	500.00	dres3	1.0
rfl	12492.5	homo3	n
rfp	11615.8	PROCESSING	
th	8	lb	0.50
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



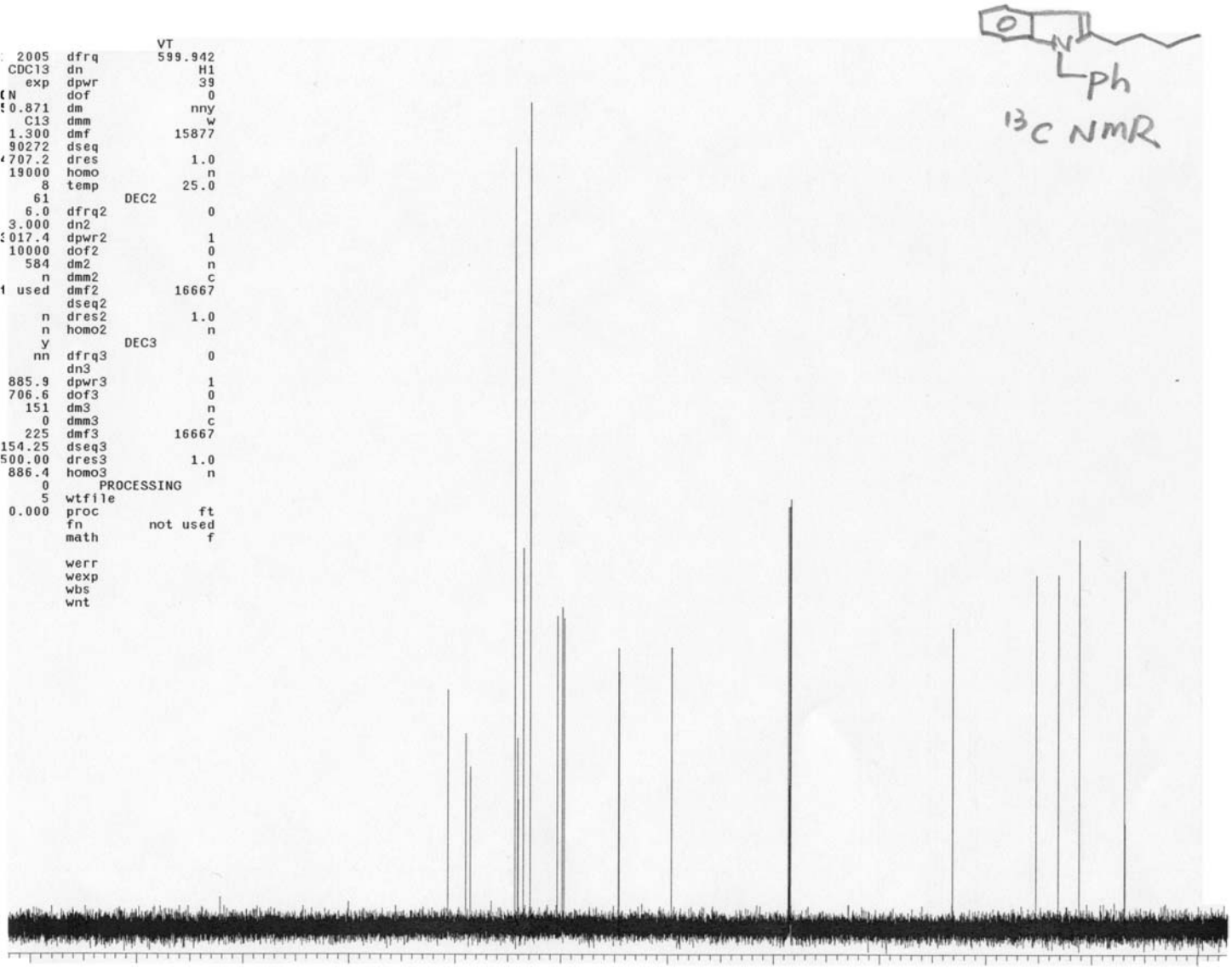

Tang_VI_P10DG
expl stdih
SAMPLE DEC. & VT
date Apr 1 2005 dfrq 200.057
solvent CDCl3 dn H1
file exp dpwr 30
ACQUISITION dof 0
sfrq 200.057 dm nnn
tn H1 dmm c
at 3.744 dmf 200
np 22464 dseq
sw 3000.1 dres 1.0
fb 1600 homo n
bs 16
tpwr 55 wtfile PROCESSING
pw 7.0 proc ft
d1 3.000 fn not used
tof 0 math f
nt 16
ct 16 werr
alock n wexp
gain not used wbs
FLAGS wnt
il n
in y
dp nn
hs
DISPLAY
sp -734.5
wp 3000.1
vs 129
sc 0
wc 223
hzmm 13.33
is 70.79
rfl 734.5
rfp 0
th 11
ins 1.000
nm cdc ph
ppm
1H NMR


Tang_VI_P100G_13C
exp2 s2pul
SAMPLE
date Apr 1 2005
solvent CDCl3
file exp
ACQUISITION
sfrq 150.871
tn C13
at 1.300
np 90272
sw 34707.2
fb 19000
bs 16
tpwr 61
pw 6.0
d1 3.000
tof 3017.4
nt 1000
ct 272
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -883.5
wp 34706.6
vs 139
sc 0
wc 225
hzmm 154.25
is 500.00
rfl 12499.9
rfp 11615.8
th 8
ins 100.000
nm cdc ph
DEC. & VT
dfrq 599.942
dn H1
dpwr 39
dof 0
dm nny
dmm w
dmf 15877
dseq
dres 1.0
homo n
temp 25.0
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 16667
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 16667
dseq3
dres3 1.0
homo3 n
PROCESSING
lb 0.50
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
13C NMR
220
200
180
160
140
120
100
80
60
40
20
ppm


Tang_VI_P100H_
exp2 std1h
date SAMPLE Nov 22 2005
solvent CDCl3
file /export/acquisition/data/
sfrq 200.057
tn H1
at 3.744
np 22464
sw 3000.1
fb 1600
bs 16
tpwr 55
pw 7.0
dl 3.000
tof 0
nt 64
ct 64
alock n
gain n
FLAGS not used
il n
in n
dp y
hs nn
DISPLAY
sp -721.1
wp 3000.1
vp 151
sc 0
wc 250
hzmm 12.00
is 223.86
rfl 721.1
rfp 0
th 6
ins 1.000
nm cdc ph
DEC. & VT
dfrq 200.057
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
math
not used f
wexp
wbs
wnt
Ph.
1H NMR
5
ppm

ng_VI_P1

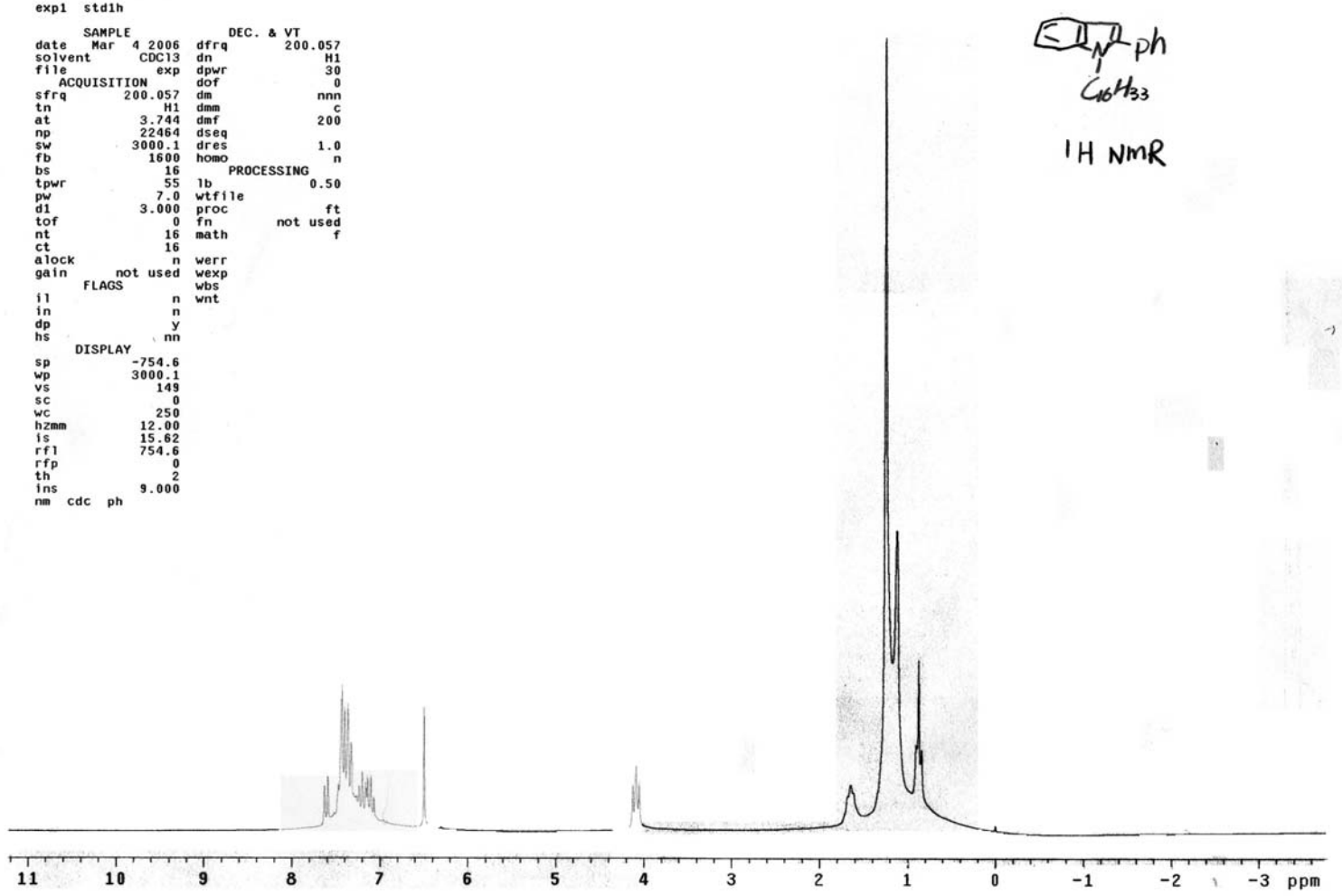
SAMPLE			VT
date	Nov 2 2005	dfrq	599.942
solvent	CDCl3	dn	H1
file	exp	dpwr	39
ACQUISITIO	N	dof	0
sfrq	15 0.871	dm	nny
tn	C13	dmm	w
at	1.300	dmf	15877
np	90272	dseq	
sw	3 707.2	dres	1.0
fb	19000	homo	n
bs	8	temp	25.0
tpwr	61	DEC2	
pw	6.0	dfrq2	0
d1	3.000	dn2	
tof	017.4	dpwr2	1
nt	10000	dof2	0
ct	584	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	16667
F1.AGS		dseq2	
	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
		dn3	
sp	885.9	dpwr3	1
wp	706.6	dof3	0
vs	151	dm3	n
sc	0	dmm3	c
wc	225	dmf3	16667
hzmm	154.25	dseq3	
is	500.00	dres3	1.0
rfl	886.4	homo3	n
rfp	0	PROCESSING	
th	5	wtfile	
ins	0.000	proc	ft
nm		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	

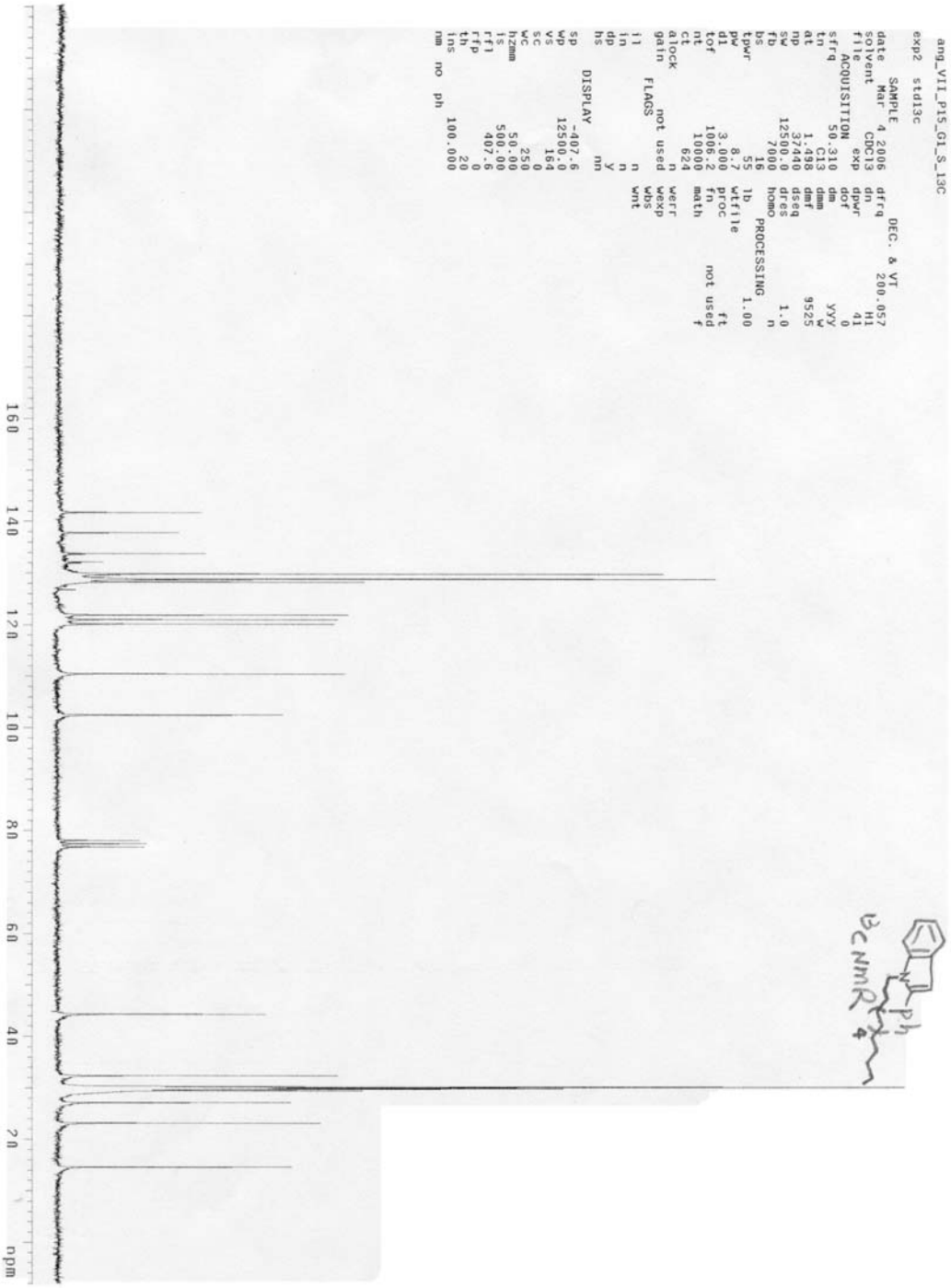


Tang_VII_P15_G1_s2

exp1 stdlh

SAMPLE		DEC. & VT	
date	Mar 4 2006	dfrq	200.057
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	200.057	dm	nnn
tn	H1	dmm	c
at	3.744	dmf	200
np	22464	dseq	
sw	3000.1	dres	1.0
fb	1600	homo	n
bs	16	PROCESSING	
tpwr	55	lb	0.50
pw	7.0	wtfile	
d1	3.000	proc	ft
tof	0	fn	not used
nt	16	math	f
ct	16		
alock	n	werr	
gain	not used	wexp	
FLAGS		wbs	
il	n	wnt	
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-754.6		
wp	3000.1		
vs	149		
sc	0		
wc	250		
hzmm	12.00		
is	15.62		
rfl	754.6		
rfp	0		
th	2		
ins	9.000		
nm cdc ph			




ang_VII_P15_G1_S_13C
exp2 std13c
SAMPLE
date Mar 4 2006
solvent CDCl3
file
ACQUISITION
sfrq
tn C13
at 1.498
np 37440
sw 12500.0
fb 70.0
bs 16
tpwr 55
pw 8.7
d1 3.000
tof 1006.2
nt 10000
ct 624
alock n
gain
FLAGS
not used
il n
in n
dp y
hs nn
DISPLAY
sp -407.6
wp 12500.0
vs 164
sc 0
wc 250
hzmm 50.00
500.00
rfl 407.6
rfp 0
th 20
ins
nm no ph 100.000
DEC. & VT
dfrq 200.057
dn H1
dpwr 41
dof 0
dm yyy
dmm w
dmf 9525
dseq
dres 1.0
homo n
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
160
140
120
100
80
60
40
20
ppm