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Supporting Information

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Supporting Information

Efficient Iridium-catalyzed C-H Functionalization/Silylation of Heteroarenes

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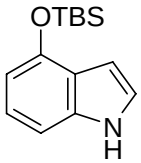
Experimental

General. All reactions were carried out under an argon atmosphere. Anhydrous solvents were freshly distilled from sodium benzophenone ketyl, except for CH_2Cl_2, which was distilled from CaH_2. Extracts were dried over anhydrous Na_2SO_4 and then filtered prior to removal of all volatiles under reduced pressure. Unless otherwise noted, commercially available materials were used without further purification. $[Ir(OMe)(COD)]_2$ was purchased from Alfa Aesar. Flash chromatography (FC) was performed using *E Merck* silica gel 60 (240–400 mesh). Thin layer chromatography was performed using precoated plates purchased from *E. Merck* (silica gel 60 PF254, 0.25 mm).

Nuclear magnetic resonance (NMR) spectra were recorded on a *Varian* 300 spectrometer at operating frequencies of 300 MHz (^{1}H NMR) or 75 MHz (^{13}C NMR). Chemical shifts (δ) are given in ppm relative to residual solvent (usually chloroform δ 7.27 for ^{1}H NMR or δ 77.23 for proton decoupled ^{13}C NMR), and coupling constants (*J)* in Hz. Multiplicity is tabulated as s for singlet, d for doublet, t for triplet, q for quadruplet, and m for multiplet, whereby the prefix app is applied in cases where the true multiplicity is unresolved, and *br* when the signal in question is broadened. The Michigan State University Mass Spectroscopy Facility provided high-resolution mass spectral analyses.



4-(*tert*-Butyldimethylsilyloxy)indole (5)[1]

To a solution of 4-hydroxyindole (40 mg, 0.3 mmol) in dry DMF (0.5 mL) was added imidazole (37 mg, 0.54 mmol) and TBSCl (52 mg, 0.32 mmol). The mixture was stirred at room temperature for 3 h. EtOAc (5 mL) and H_2O (5 mL) were added. The organic layer was separated and the aqueous layer was extracted with EtOAc (3 × 3 mL). The combined organic extracts were dried with Na_2SO_4 and concentrated under reduced pressure. The residue was purified by flash chromatography using silica gel to give 78

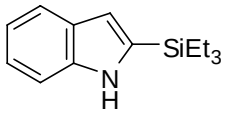
mg (100%) of 4-(*tert*-butyldimethylsilyloxy)indole.

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.09 (br, 1H), 7.10-7.01 (m, 3H), 6.60 (s, 1H), 6.54 (d, *J* = 6.0 Hz, 1H), 1.08 (s, 9H), 0.25 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 149.3, 138.0, 122.9 (2 × C), 121.9, 108.9, 104.9, 100.6, 26.0, 18.6, -4.1.

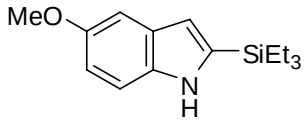
General procedure for the 2-silylation of heteroarenes with triethylsilane. A flamed-dried Schlenk tube was charged with heteroarene (0.2 mmol), $[Ir(OMe)(COD)]_2$ (6.6 mg, 0.01 mmol) and dtpy (5.4 mg, 0.02 mmol), then evacuated and flushed with argon three times. Under a positive flow of argon, 2-norbornene (56 mg, 0.6 mmol) and dry THF (1 mL) were added. After stirring for 5 min, triethylsilane (98 μL, 0.6 mmol) was added dropwise. The reaction mixture was heated at 80°C for 24 h or indicated time. The solvent was concentrated under reduced pressure. The residue was purified by flash chromatography using silica gel to give 2-(triethylsilyl)heteroarene.

For 1-tosyl-indole, the 3-silyation product was obtained using above general procedure.

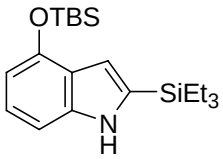
In case of furan (0.2 mmol) and thiophene (0.2 mmol), 2-norbornene (75 mg, 0.8mmol) and triethylsilane (130 μL, 0.6 mmol) were employed.



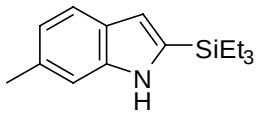
2-(Triethylsilyl)indole (2). 87% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.10 (br, 1H), 7.66-7.64 (m, 1H), 7.41 (dd, *J* = 8.1 Hz, 0.9 Hz, 1H), 7.22-7.16 (m, 1H), 7.13-7.07 (m, 1H), 6.76-6.75 (m, 1H), 1.07-1.01 (m, 9H), 0.89-0.81 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 138.8, 135.8, 128.9, 122.4, 120.7, 119.8, 112.6, 111.0, 7.7, 3.8. HRMS (ESI) for $C_{14}H_{22}NSi$ $[MH^+]$ Calc 232.1522, Found 232.1517.



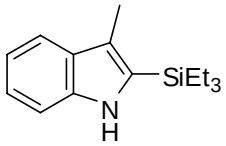
5-Methoxyl-2-triethylsilyl-indole (4). 90% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.05 (br, 1H), 7.30 (d, *J* = 9.0 Hz, 1H), 7.11 (s, 1H), 6.88 (dd, *J* = 8.4 Hz, 2.1 Hz, 1H), 6.69 (s, 1H), 3.87 (s, 3H), 1.07-1.02 (m, 9H), 0.89-0.81 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 154.3, 136.7, 134.2, 129.3, 113.0, 112.2, 111.7, 101.9, 56.1, 7.7, 3.8. HRMS (ESI) for $C_{15}H_{24}NOSi$ [MH^+] Calc 262.1627 Found 262.1620.



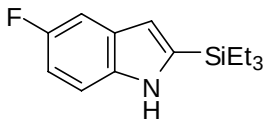
4-(*tert*-Butyldimethylsilyloxy)-2-(triethylsilyl)-indole (6). 82% yield; m.p 62-63 ^{o}C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.03 (br, 1H), 7.02 (s, 1H), 7.01 (d, *J* = 1.8 Hz, 1H), 6.74 (d, *J* = 2.4 Hz, 1H), 6.48 (dd, *J* = 4.8 Hz, 3.0 Hz, 1H), 1.06 (s, 9H), 1.05-1.00 (m, 9H), 0.87-0.79 (m, 6H), 0.24 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 149.0, 140.9, 134.1, 123.1, 122.8, 110.2, 108.5, 104.6, 26.1, 18.6, 7.7, 3.8, -4.0. HRMS (ESI) for $C_{20}H_{36}NOSi_2$ [MH^+] Calc 362.2335 Found 362.2338.



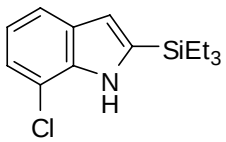
6-Methyl-2-triethylsilylindole (8). 86% yield**;** ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.97 (br, 1H), 7.52 (d, *J* = 7.5Hz, 1H), 7.20 (s, 1H), 6.93 (d, *J* = 8.1 Hz, 1H), 6.70 (s, 1H), 2.47 (s, 3H), 1.06-1.00 (m, 9H), 0.88-0.80 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 139.4, 135.0, 132.2, 126.8, 121.6, 120.3, 112.4, 110.9, 22.0, 7.7, 3.8. HRMS (ESI) for $C_{15}H_{24}NSi$ [MH^+] Calc 246.1678 Found 246.1682.



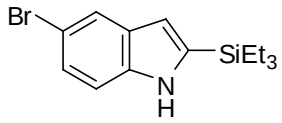
3-Methyl-2-triethylsilylindole (10). 55% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.87 (br, 1H), 7.62-7.58 (m, 1H), 7.38-7.35 (m, 1H), 7.22-7.16 (m, 1H), 7.14-7.09 (m, 1H), 2.42 (s, 3H), 1.06-1.01 (m, 9H), 0.95-0.87 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 138.5, 130.9, 129.7, 122.3, 121.1, 119.1, 118.8, 110.9, 10.8, 7.7, 3.9. HRMS (ESI) for $C_{15}H_{23}NSi$ [M^+] Calc 245.1600 Found 245.1606.



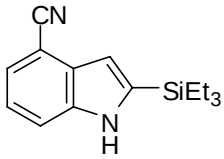
5-Fluoro-2-triethylsilylindole (12). 76% yield**;** ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.11 (br, 1H), 7.34-7.26 (m, 2H), 6.99-6.92 (m, 1H), 6.72-6.71 (m, 1H), 1.08-1.02 (m, 9H), 0.90-0.82 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.1 (*J* = 233.0 Hz), 138.2, 135.5, 129.2 (*J* = 9.7 Hz), 112.5 (*J* = 4.9 Hz), 111.4 (*J* = 9.7 Hz), 110.8 (*J* = 26.0 Hz), 105.2 (*J* = 22.4 Hz), 7.7, 3.7; HRMS (ESI) for $C_{14}H_{21}NFSi$ [MH^+] Calc 250.1427 Found 250.1425.



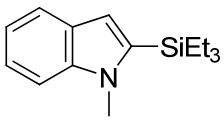
7-Chloro-2-triethylsilylindole (14). 64% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.21 (br, 1H), 7.54 (dd, *J* = 8.1 Hz, 0.9 Hz, 1H), 7.18 (dd, *J* = 7.8 Hz, 0.9 Hz, 1H), 7.03 (t, *J* = 7.5 Hz, 1H), 6.78 (d, *J* = 2.1 Hz, 1H), 1.08-1.02 (m, 9H), 0.91-0.83 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 137.0, 136.1, 130.4, 121.6, 120.6, 119.3, 116.5, 113.5, 7.7, 3.7; HRMS (ESI) for $C_{14}H_{21}NSiCl$ [MH^+] Calc 266.1132 Found 266.1133.



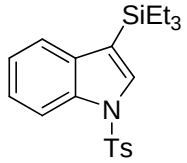
6-Bromo-2-triethylsilylindole (16). 52% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.12 (br, 1H), 7.75 (s, 1H), 7.26-7.25 (m, 2H), 6.66 (s, 1H), 1.02-0.96 (m, 9H), 0.88-0.80 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 137.6, 137.4, 130.7, 125.1, 123.1, 113.0, 112.3, 112.0, 7.7, 3.7; HRMS (ESI) for $C_{14}H_{21}NSiBr$ [MH^+] Calc 310.0627 Found 310.0624.



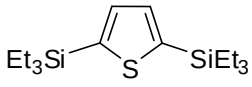
4-Nitro-2-triethylsilylindole (18). 41% yield; m.p 131-133 ^{o}C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.51 (br, 1H), 7.64 (dd, *J* = 8.4 Hz, 0.4 Hz, 1H), 7.46 (d, *J* = 7.5 Hz, 1H), 7.23-7.18 (m, 1H), 6.93-6.92 (m, 1H), 1.06-1.01 (m, 9H), 0.92-0.84 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 140.0, 138.6, 130.1, 125.3, 121.8, 119.3, 115.8, 111.0, 102.7, 7.6, 3.6. HRMS (ESI) for $C_{15}H_{21}N_2Si$ [MH^+] Calc 257.1474 Found 257.1470.



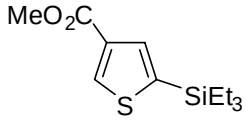
***N*-Methyl-2-triethylsilylindole (20).** 49% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.62 (d, *J* = 7.8 Hz, 1H), 7.34 (d, *J* = 8.1 Hz, 1H), 7.25-7.20 (m, 1H), 7.09 (t, *J* = 7.2 Hz, 1H), 6.73 (s, 1H), 3.84 (s, 3H), 1.04-0.99 (m, 9H), 0.95-0.88 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 140.4, 138.5, 128.7, 122.0, 120.7, 119.2, 113.1, 109.2, 33.2, 7.8, 4.2; HRMS (ESI) for $C_{15}H_{24}NSi$ [MH^+] Calc 246.1678 Found 246.1676.



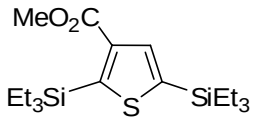
***N*-Tosyl-3-triethylsilylindole (22).** 70% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.97-7.94 (m, 1H), 7.76 (d, *J* = 7.8 Hz, 2H), 7.60-7.56 (m, 1H), 7.55 (s, 1H), 7.30-7.18 (m, 4H), 2.33 (s, 3H), 0.99-0.94 (m, 9H), 0.90-0.82 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 145.1, 136.1, 135.5, 135.3, 132.8, 130.1, 127.0, 124.4, 123.3, 122.7, 115.4, 113.7, 21.8, 7.7, 3.9. HRMS (ESI) for $C_{21}H_{28}NO_2SSi$ [MH^+] Calc 386.1610 Found 386.1607.



2,5-Bis(triethylsilyl)thiophene (24). 98% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.33 (s, 2H), 1.04-0.98 (m, 18H), 0.85-0.77 (m, 12H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 142.5, 135.6, 7.7, 4.8. HRMS (EI) for $C_{16}H_{32}SSi_2$ [M^+] Calc 312.1763 Found 312.1762.

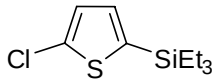


Methyl 5-(triethylsilyl)thiophene-3-carboxylate (26). 67% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.32 (d, *J* = 0.9Hz, 1H), 7.64 (d, *J* = 0.9Hz, 1H), 3.86 (s, 3H), 1.02-0.96 (m, 9H), 0.85-0.77 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 163.8, 138.6, 138.0, 135.6, 135.0, 52.0, 7.5, 4.4. HRMS (ESI) for $C_{12}H_{21}O_2SiS$ [MH^+] Calc 257.1032 Found 257.1035.

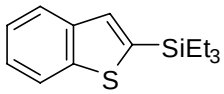


Methyl 2,5-bis(triethylsilyl)thiophene-3-carboxylate 33% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.77 (s, 1H), 3.86 (s, 3H), 1.02-0.94 (m, 24H), 0.81 (t, *J* = 7.5 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 164.6, 152.2, 142.7, 140.0, 138.5, 51.7, 7.9, 7.6, 4.6, 4.1.

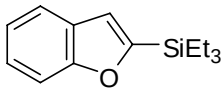
HRMS (ESI) for $C_{18}H_{38}O_2Si_2S$ [MH^+] Calc 371.1896 Found 371.1894.



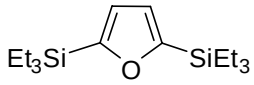
5-Chloro-2-triethylsilylthiophene (28). 91% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.00 (d, *J* = 3.6 Hz, 1H), 6.97 (d, *J* = 3.6 Hz, 1H), 1.02-0.96 (m, 9H), 0.81-0.73 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 137.0, 134.6, 134.3, 127.6, 7.5, 4.4. HRMS (EI) for $C_{10}H_{17}SSiCl$ [M^+] Calc 232.0509 Found 232.0508.



2-Triethylsilyl benzothiophene (30). 99% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.91-7.88 (m, 1H), 7.84-7.81 (m, 1H), 7.47 (s, 1H), 7.34-7.31 (m, 2H), 1.07-1.01 (m, 9H), 0.91-0.83 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 143.8, 141.2, 139.2, 131.8, 124.3, 124.1, 123.6, 122.4, 7.6, 4.5. HRMS (EI) for $C_{14}H_{20}SSi$ [M^+] Calc 248.1055 Found 248.1048.

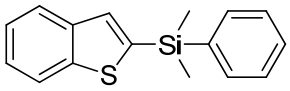


2-Triethylsilyl benzofuran (32). 83% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.60-7.57 (m, 1H), 7.53-7.50 (m, 1H), 7.30-7.16 (m, 2H), 6.99 (s, 1H), 1.07-1.01 (m, 9H), 0.88-0.81 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 161.6, 158.1, 128.0, 124.1, 122.2, 120.9, 117.2, 111.3, 7.3, 3.1. HRMS (EI) for $C_{14}H_{20}OSi$ [M^+] Calc 232.1283 Found 232.1283 .



2,5-Bis(triethylsilyl)furan (34).[2] 93% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.62 (s, 2H), 1.02-0.96 (m, 18H), 0.80-0.71 (m, 12H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 162.8, 120.1, 7.6,

3.6.

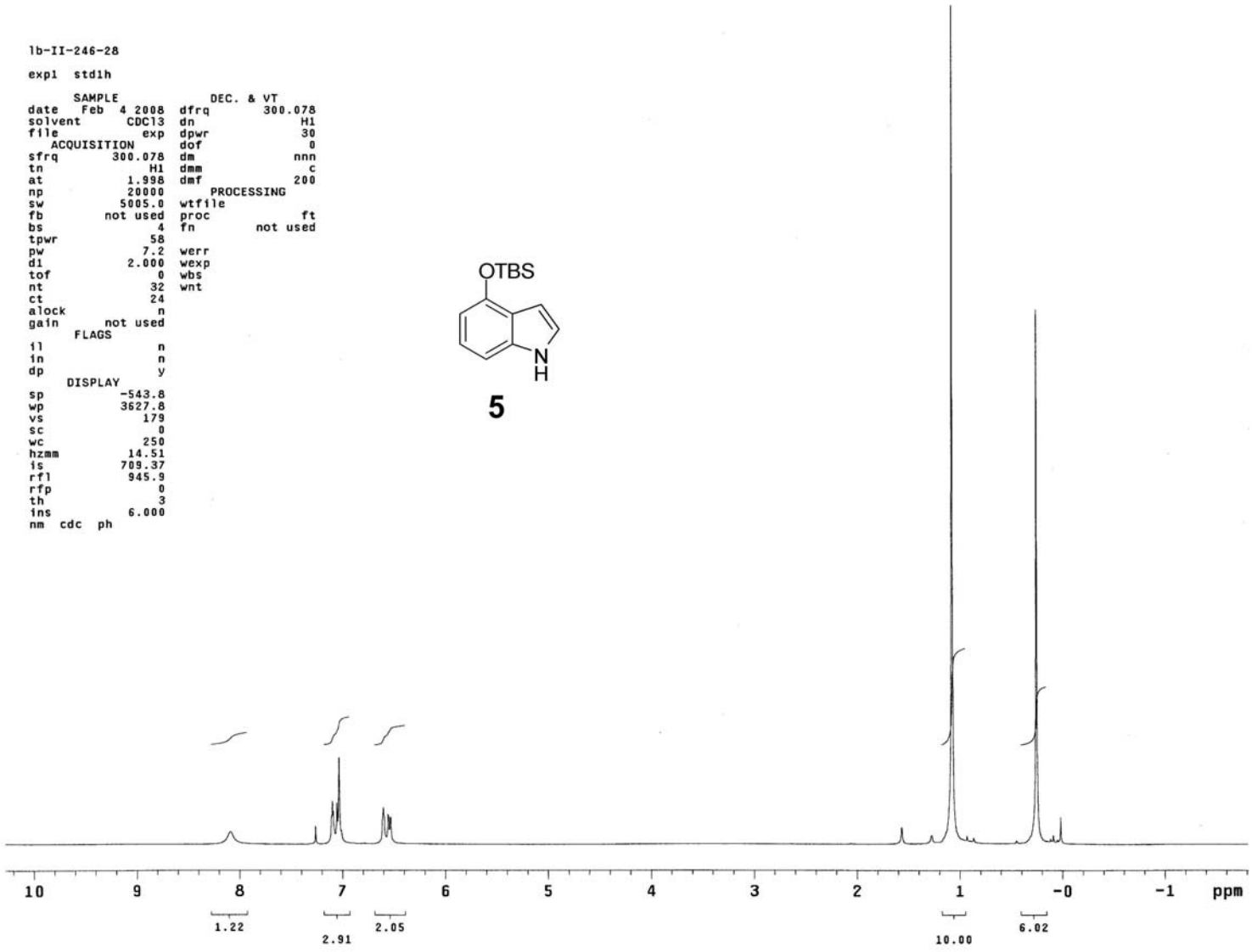


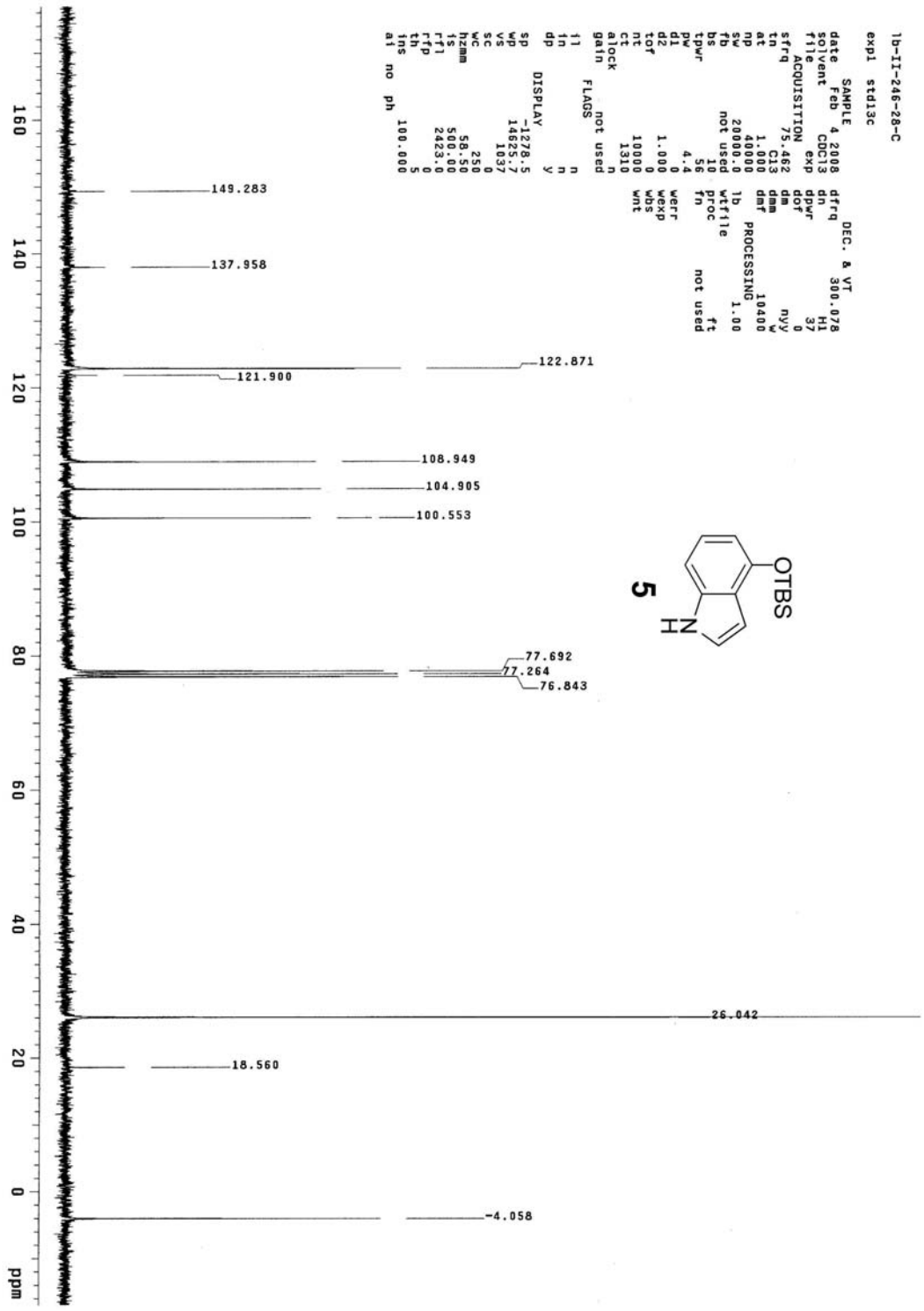
2-Dimethyl(phenyl)silyl benzothiophene (35). 92% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.90-7.80 (m, 2H), 7.64-7.61 (m, 2H), 7.50 (s, 1H), 7.42-7.31 (m, 5H); 0.67 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 144.1, 141.2, 140.3, 137.4, 134.2, 132.5, 129.8, 128.2, 124.5, 124.3, 123.8, 122.4, -1.4. HRMS (EI) for $C_{16}H_{16}SSi$ [M^+] Calc 268.0742 Found 268.0741.

References

[1] G. Papageorgiou, J. E. T. Corrie, *J. Heterocyclic Chem.* **2005**, *42*, 1101.

[2] J-P. Bouillon, D. Saleur, C. Portella, *Synthesis*, **2000**, 843.


lb-II-246-28
exp1 std1h
SAMPLE
date Feb 4 2008
solvent CDCl3
file exp
ACQUISITION
sfrq 300.078
tn H1
at 1.998
np 20000
sw 5005.0
fb not used
bs 4
tpwr 58
pw 7.2
d1 2.000
tof 0
nt 32
ct 24
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -543.8
wp 3627.8
vs 179
sc 0
wc 250
hzmm 14.51
is 709.37
rfl 945.9
rfp 0
th 3
ins 6.000
nm cdc ph
DEC. & VT
dfrq 300.078
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
OTBS
N
H
5
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm
1.22
2.91
2.05
10.00
6.02

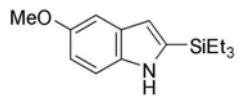


lb-II-246-28-C
exp1 std13c
SAMPLE
date Feb 4 2008
solvent CDCl3
file exp
ACQUISITION
sfrq 75.462
tn C13
at 1.000
np 40000
sw 20000.0
fb not used
bs 10
tpwr 56
pw 4.4
d1 0
d2 1.000
tof 0
nt 10000
ct 1310
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -1278.5
wp 14625.7
vs 1037
sc 0
wc 250
hzmm 58.50
is 500.00
rfl 2423.0
rfp 0
th 5
ins 100.000
ai no ph
DEC. & VT
dfrq 300.078
dn H1
dpwr 37
dof 0
dm nyy
dmm w
dmf 10400
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
149.283
137.958
122.871
121.900
108.949
104.905
100.553
77.692
77.264
76.843
26.042
18.560
-4.058
OTBS
N
H
5
160
140
120
100
80
60
40
20
0
ppm

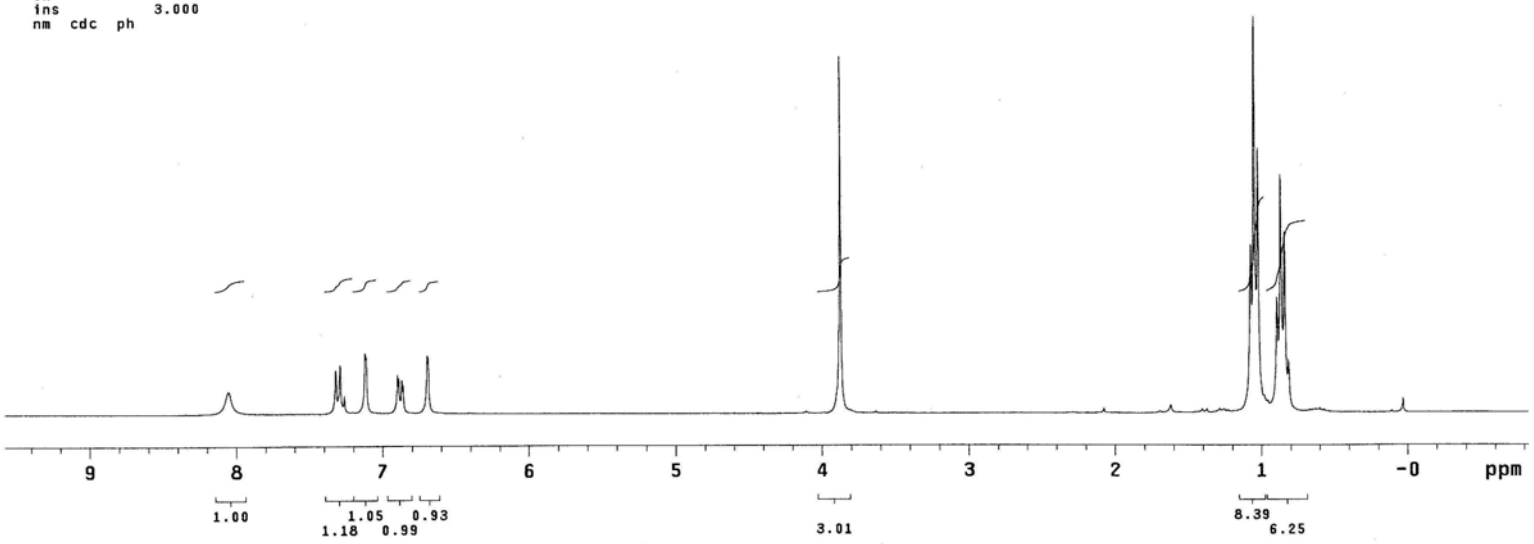
lb-II-220-20

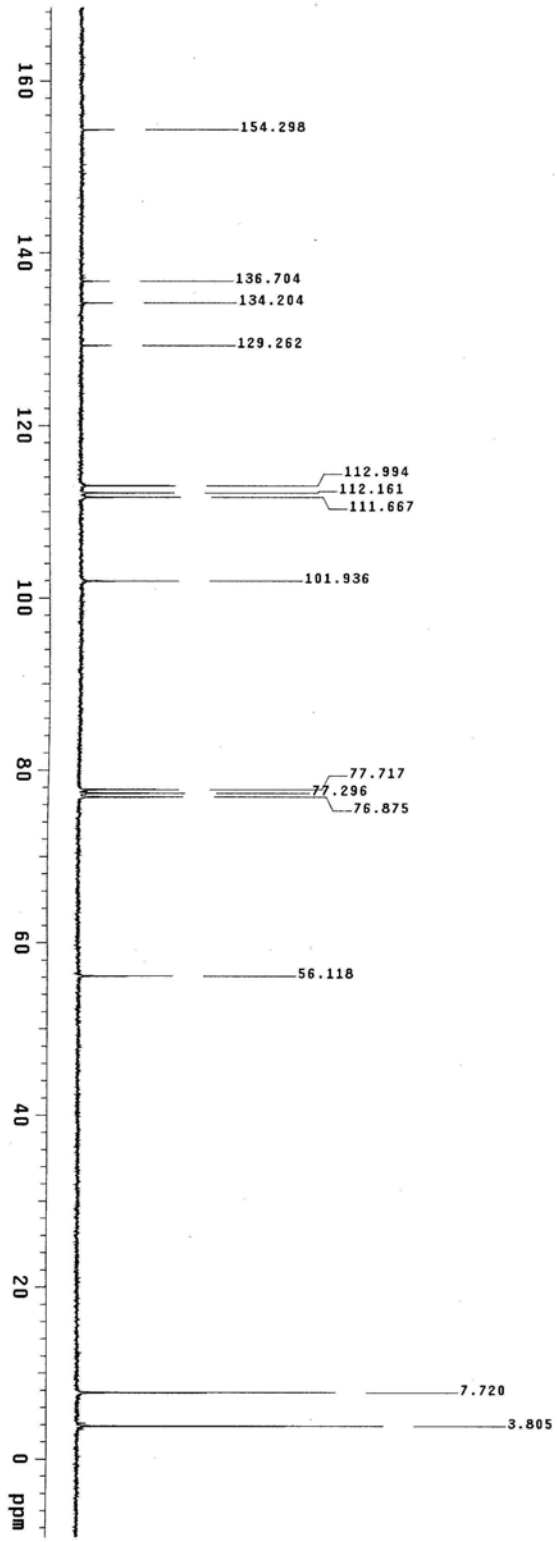
exp1 std1h

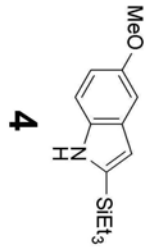
SAMPLE		DEC. & VT	
date	Jan 17 2008	dfrq	300.078
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.078	dm	nnn
tn	H1	dmm	c
at	1.998	dmf	200
np	20000	PROCESSING	
sw	5005.0	wtfile	
fb	not used	proc	ft
bs	4	fn	not used
tpwr	58		
pw	7.2	werr	
d1	2.000	wexp	
tof	0	wbs	
nt	32	wnt	
ct	16		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-248.7		
wp	3123.7		
vs	65		
sc	0		
wc	250		
hzmm	12.49		
is	500.00		
rfl	945.9		
rfp	0		
th	4		
ins	3.000		
nm cdc ph			



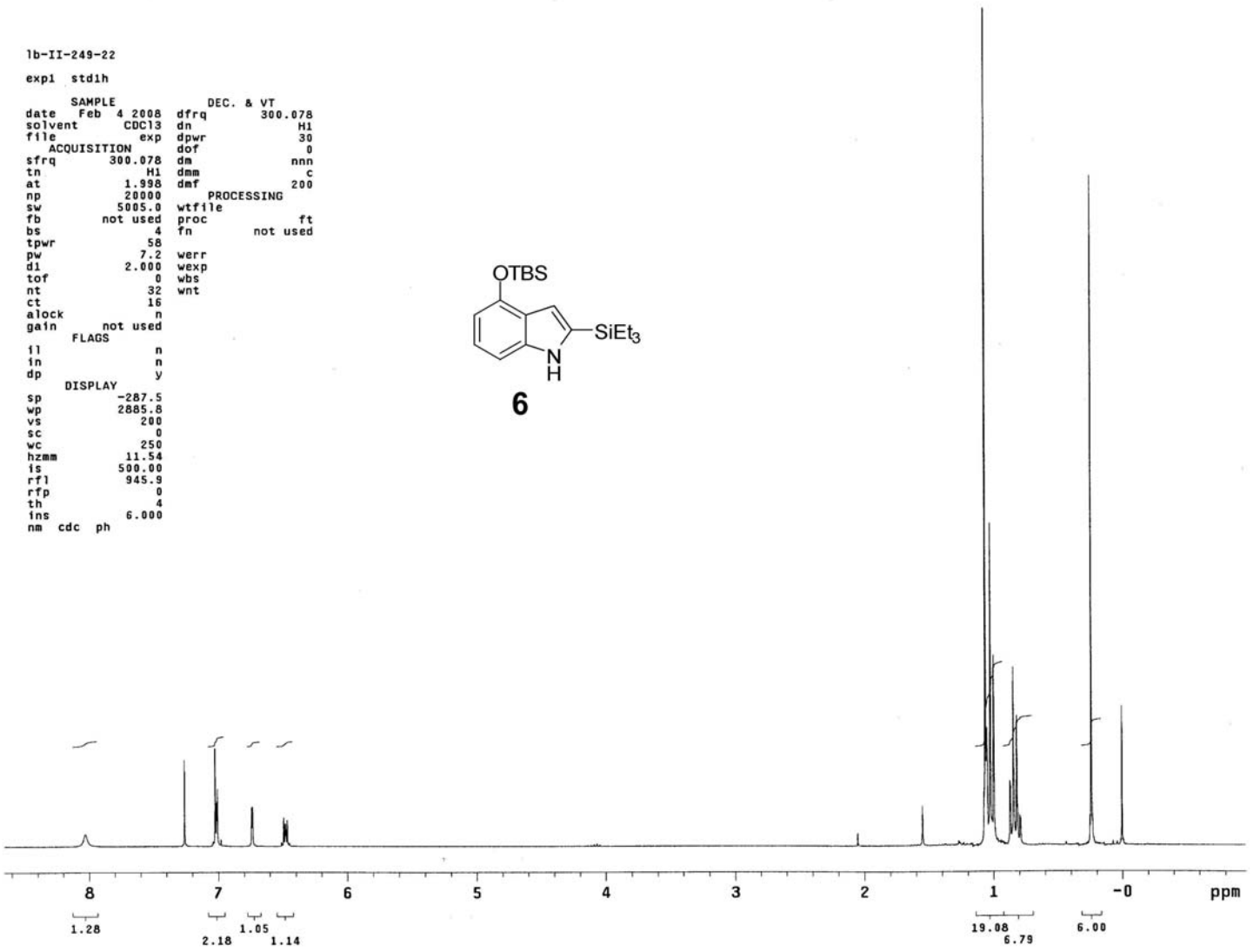
4

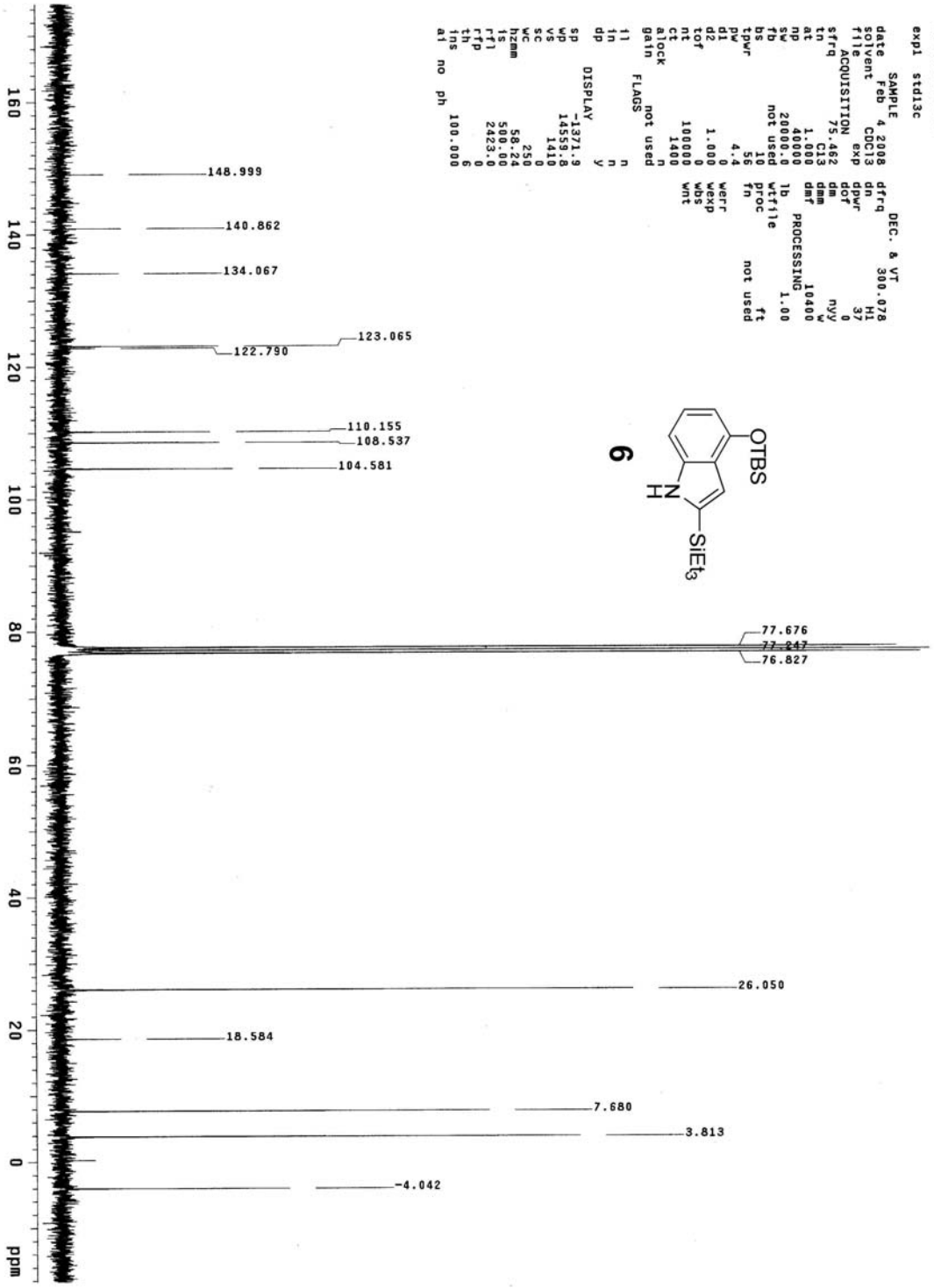


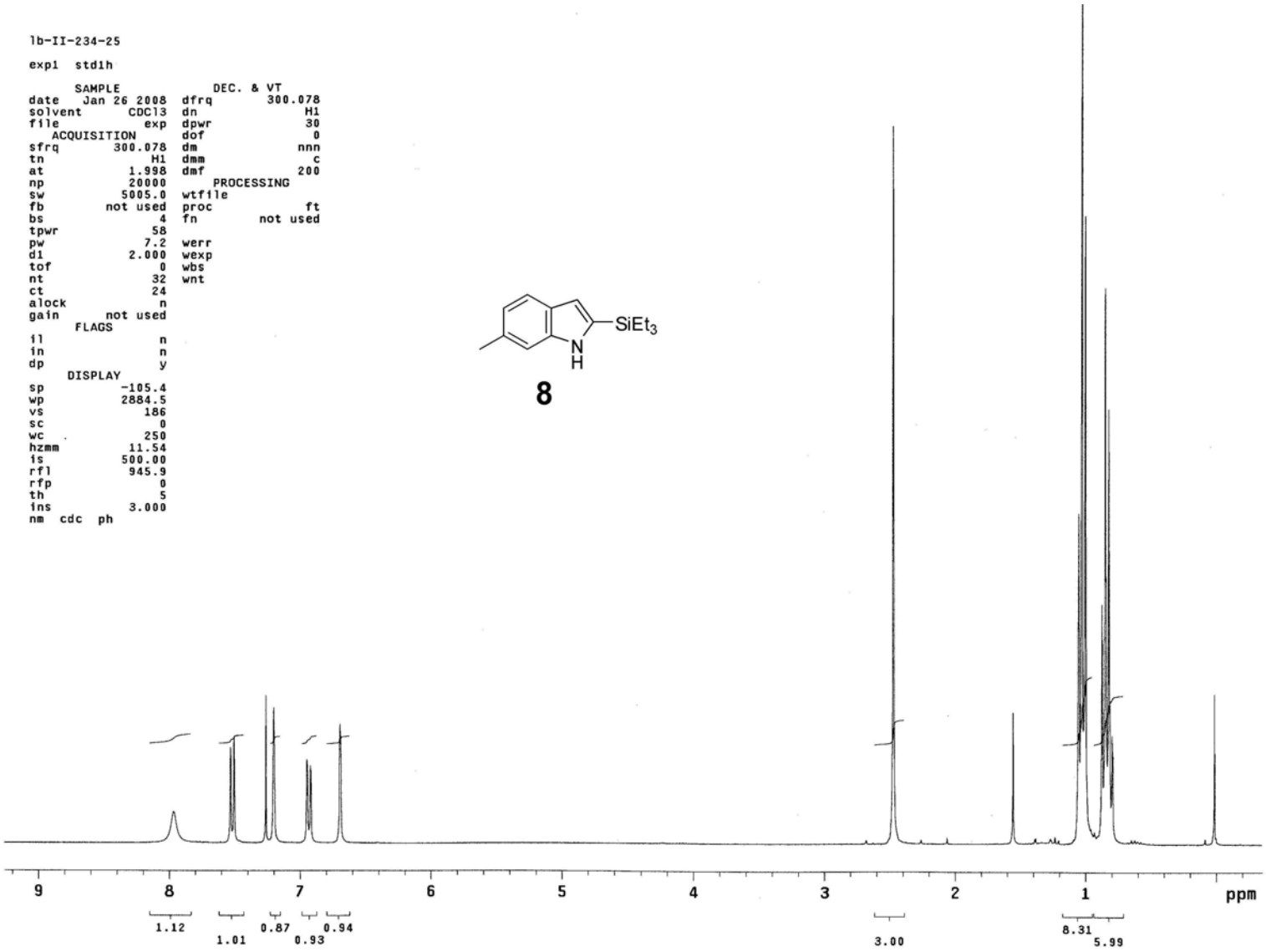

160
140
120
100
80
60
40
20
0
ppm
154.298
136.704
134.204
129.262
112.994
112.161
111.667
101.936
77.717
77.296
76.875
56.118
7.720
3.805

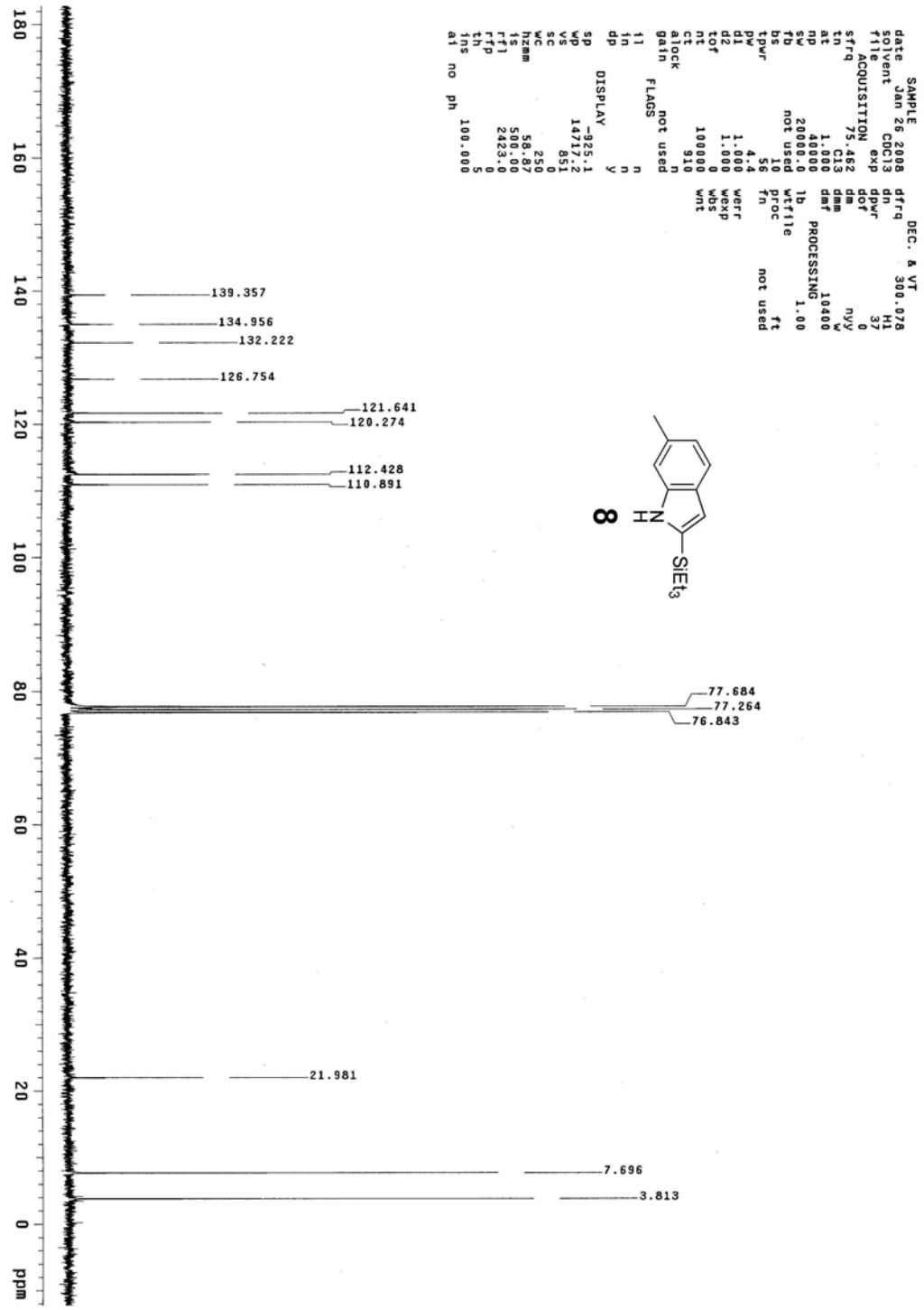

MeO
H
N
SiEt3

4


lb-II-249-22
expl stdlh
SAMPLE
date Feb 4 2008
solvent CDCl3
file exp
ACQUISITION
sfrq 300.078
tn H1
at 1.998
np 20000
sw 5005.0
fb not used
bs 4
tpwr 58
pw 7.2
d1 2.000
tof 0
nt 32
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -287.5
wp 2885.8
vs 200
sc 0
wc 250
hzmm 11.54
is 500.00
rfl 945.9
rfp 0
th 4
ins 6.000
nm cdc ph
DEC. & VT
dfrq 300.078
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
OTBS
SiEt3
N
H
6
8
7
6
5
4
3
2
1
-0
ppm
1.28
2.18
1.05
1.14
19.08
6.79
6.00


lb-II-249-22-C
exp1 std13c
SAMPLE
date Feb 4 2008
solvent CDCl3
file exp
ACQUISITION
sfrq 75.462
tn C13
at 1.000
np 40000
sw 20000.0
fb not used
bs 56
tpwr 4.4
pw 0
d1 1.000
d2 0
tof 100000
nt 1400
ct n
alock
gain
FLAGS
fl n
in n
dp y
DISPLAY
sp -1371.9
wp 14559.8
vs 1410
sc 0
wc 250
hzmm 58.24
is 500.00
rfl 2423.0
rfp 0
th 6
ins
al no ph
100.000
DEC. & VT
dfrq 300.078
dn H1
dpwr 37
dof 0
nyy
w
10400
PROCESSING
lb 1.00
wtfile
proc
fn
not used
ft
werr
wexp
wbs
wnt
OTBS
N
H
SiEt3
6
148.999
140.862
134.067
123.065
122.790
110.155
108.537
104.581
77.676
77.247
76.827
26.050
18.584
7.680
3.813
-4.042
ppm

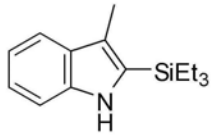

lb-II-234-25
exp1 std1h
SAMPLE
date Jan 26 2008
solvent CDCl3
file exp
ACQUISITION
sfrq 300.078
tn H1
at 1.998
np 20000
sw 5005.0
fb not used
bs 4
tpwr 58
pw 7.2
d1 2.000
tof 0
nt 32
ct 24
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -105.4
wp 2884.5
vs 186
sc 0
wc 250
hzmm 11.54
is 500.00
rfl 945.9
rfp 0
th 5
ins 3.000
nm cdc ph
DEC. & VT
dfrq 300.078
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
SiEt3
N
H
8
9
8
7
6
5
4
3
2
1
ppm
1.12
1.01
0.87
0.93
0.94
3.00
8.31
5.99


lb-II-234-25-C
expl stdl3c
SAMPLE
date Jan 26 2008
solvent CDCl3
file exp
ACQUISITION
sfrq 75.462
tn C13
at 1.000
np 40000
sw 20000.0
fb not used
bs 10
tpwr 56
pw 4.4
d1 1.000
d2 1.000
tof 0
nt 100000
ct 910
alock wnt
gain n
FLAGS
il n
in n
dp y
DISPLAY
sp -925.1
wp 14717.2
vs 851
sc 0
wc 250
hzmm 58.87
is 500.00
rfl 2423.0
rfp 0
th 5
ins
at 100.000 no ph
DEC. & VT
dfrq 300.078
dn H1
dpwr 37
dof 0
dm nyy
dmm w
dmf 10400
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
139.357
134.956
132.222
126.754
121.641
120.274
112.428
110.891
77.684
77.264
76.843
21.981
7.696
3.813
8
SiEt3
180
160
140
120
100
80
60
40
20
0
ppm

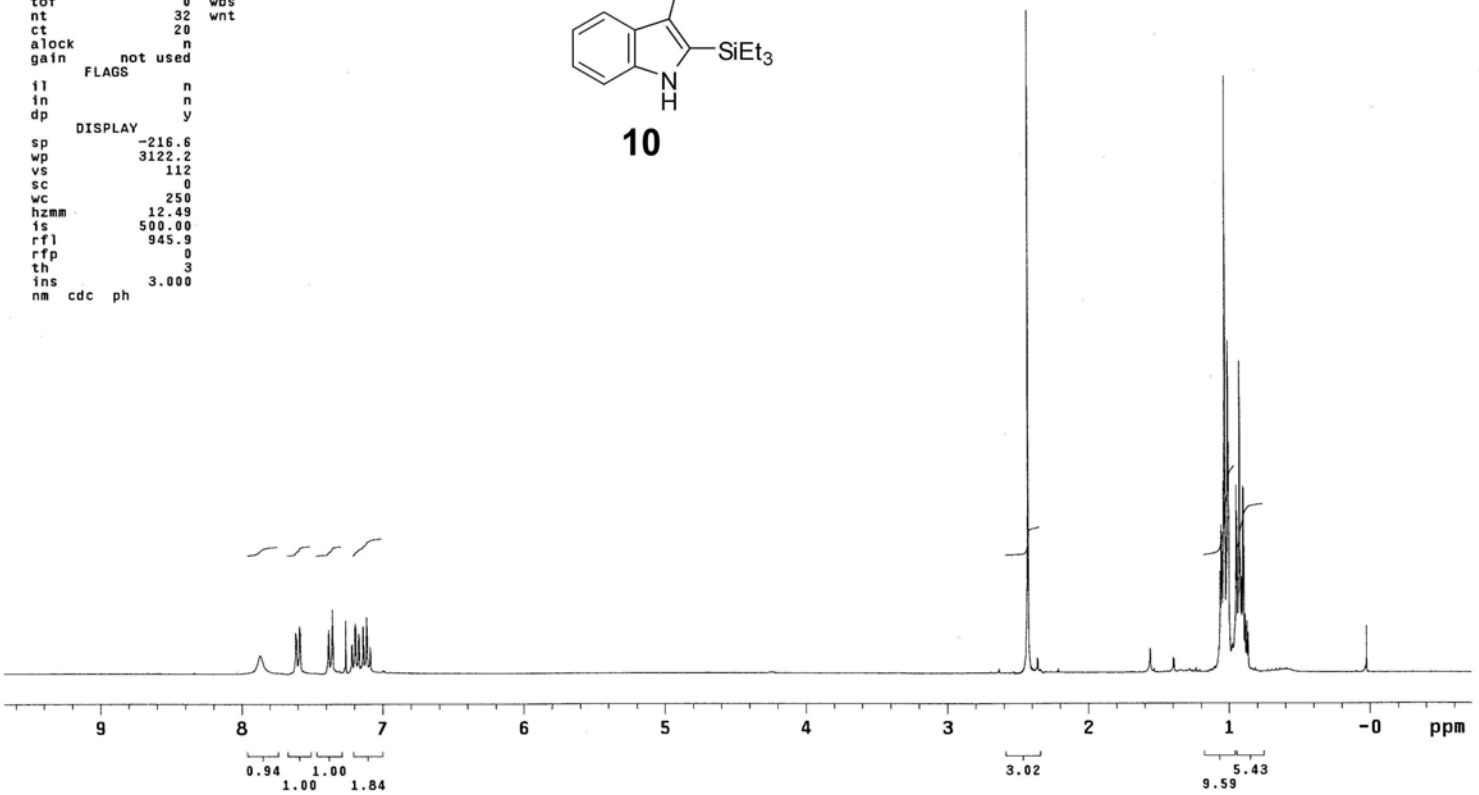
lb-II-208-28

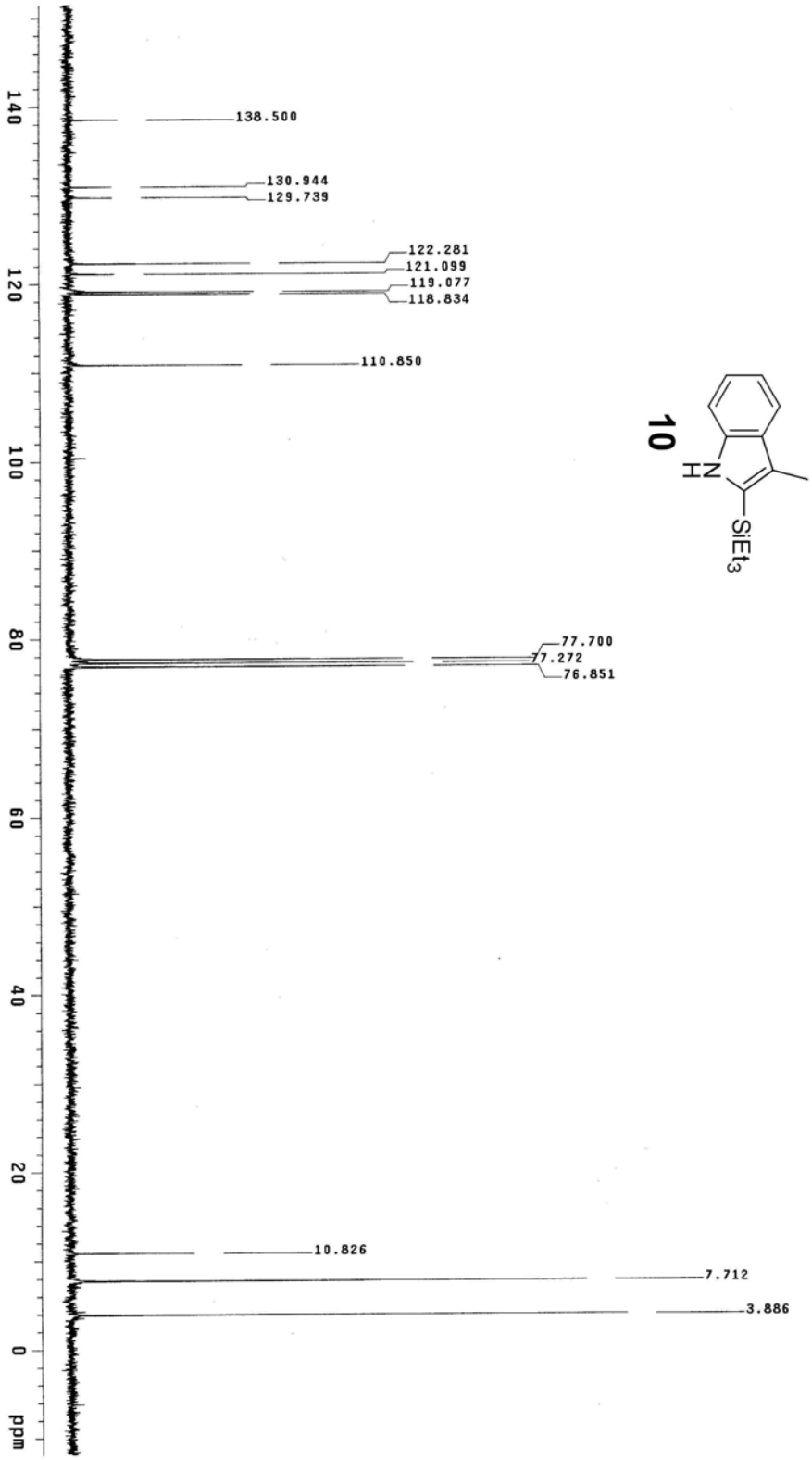
exp1 stdlh

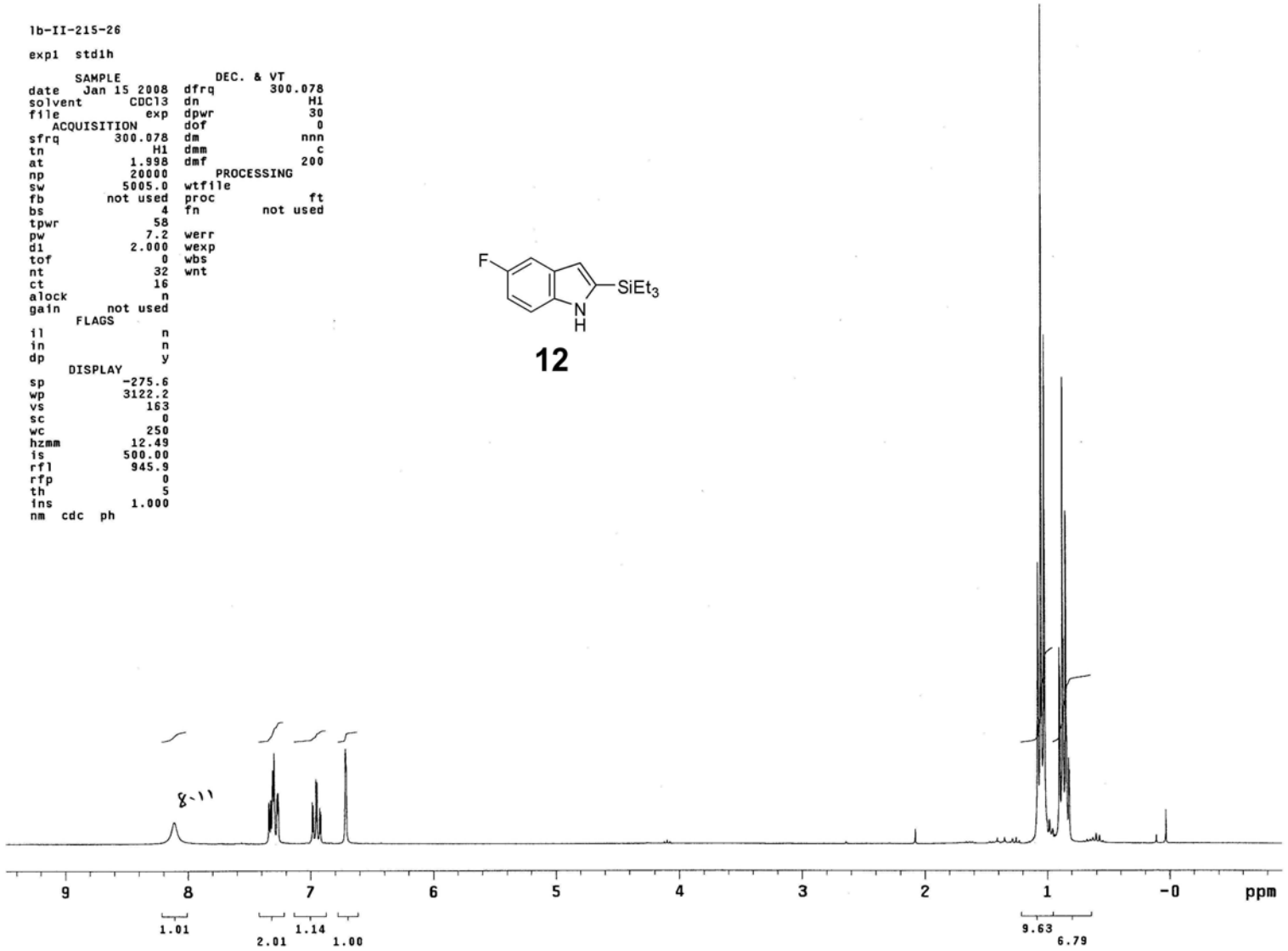
SAMPLE		DEC. & VT	
date	Jan 12 2008	dfrq	300.078
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.078	dm	nnn
tn	H1	dmm	c
at	1.998	dmf	200
np	20000	PROCESSING	
sw	5005.0	wtfile	
fb	not used	proc	ft
bs	4	fn	not used
tpwr	58		
pw	7.2	werr	
d1	2.000	wexp	
tof	0	wbs	
nt	32	wnt	
ct	20		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-216.6		
wp	3122.2		
vs	112		
sc	0		
wc	250		
hzmm	12.49		
is	500.00		
rfl	945.9		
rfp	0		
th	3		
ins	3.000		
nm cdc ph			

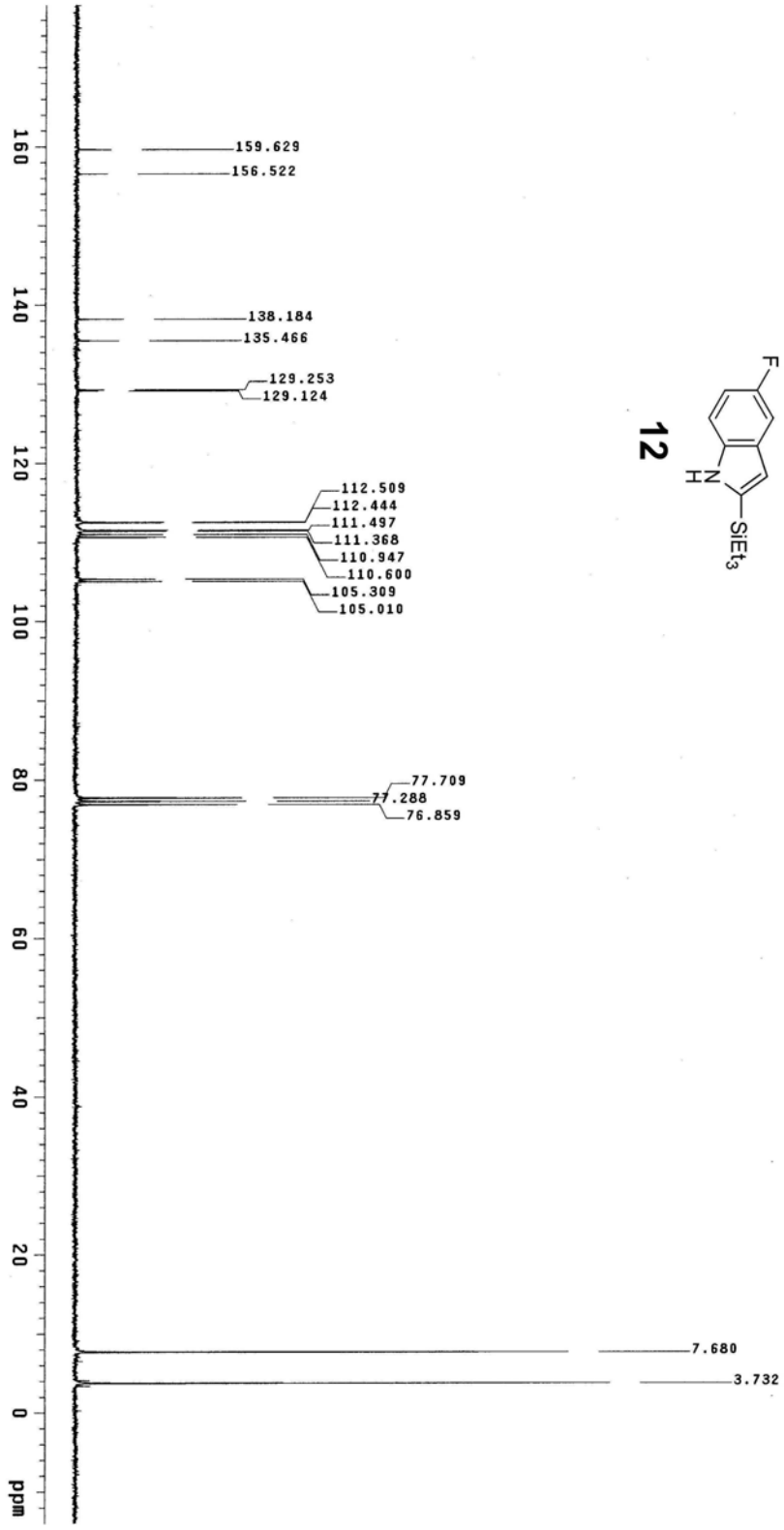


10



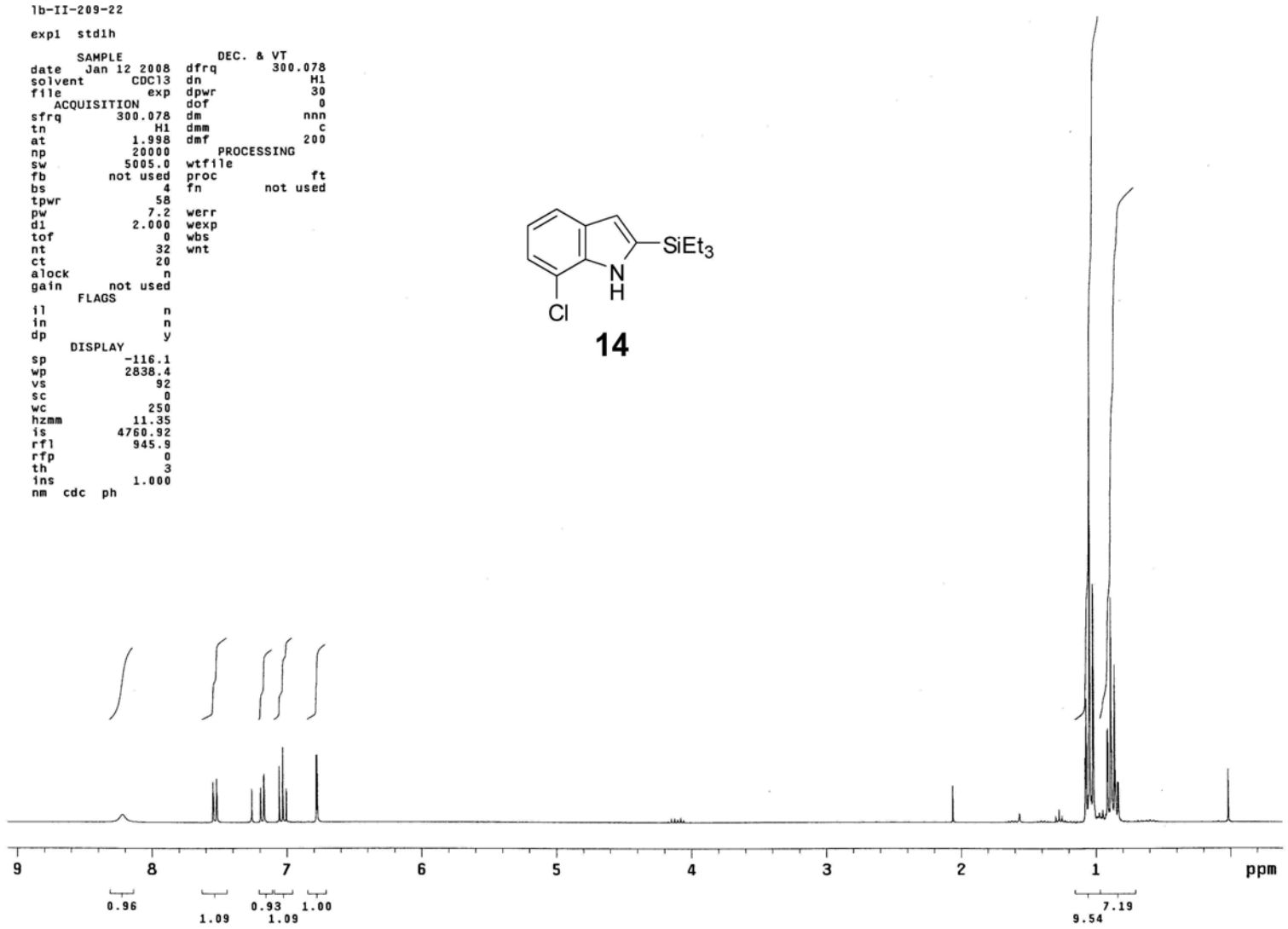

138.500
130.944
129.739
122.281
121.099
119.077
118.834
110.850
77.700
77.272
76.851
10.826
7.712
3.886
ppm
140
120
100
80
60
40
20
0
10
N
H
SiEt3

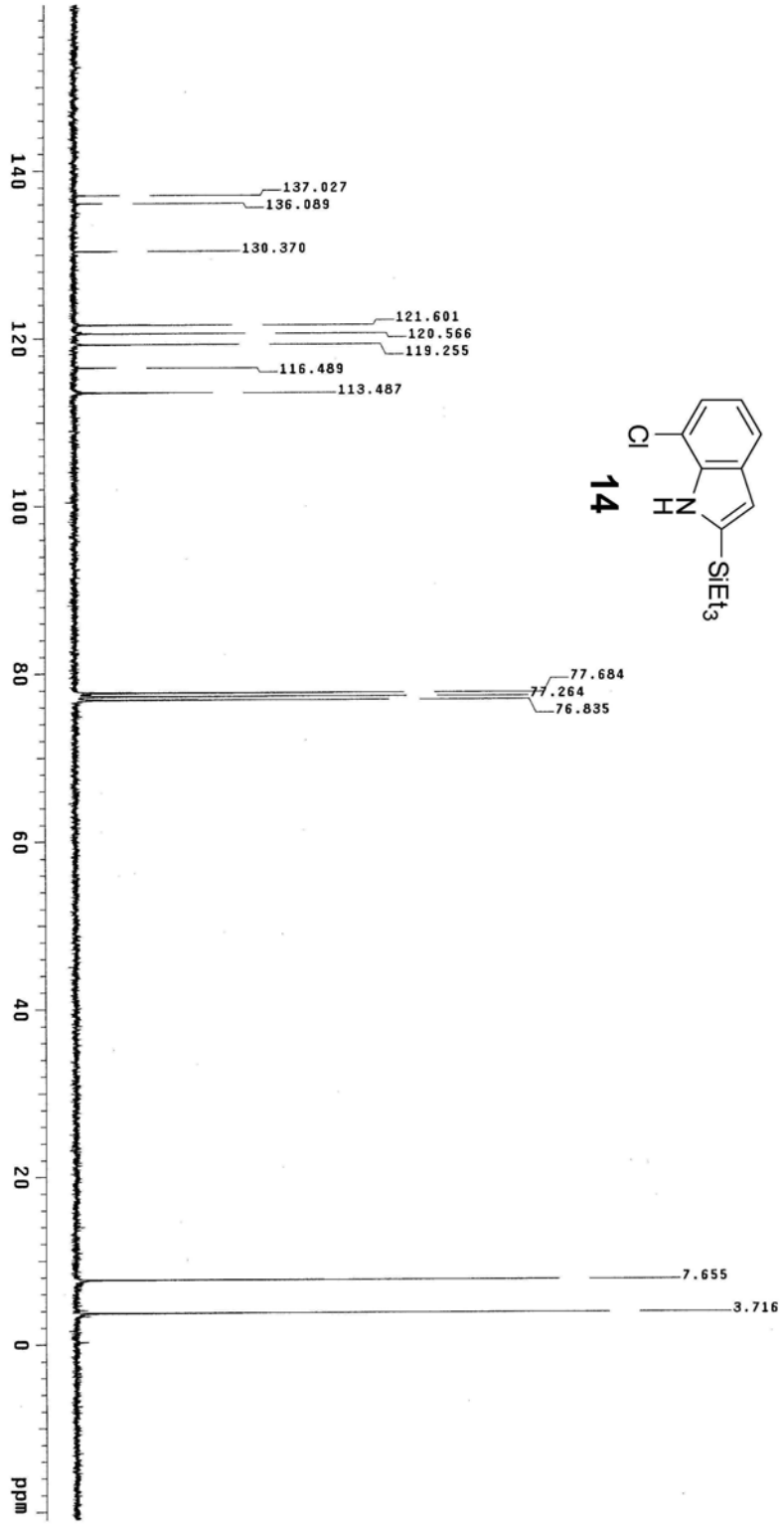

lb-II-215-26
exp1 stdlh
SAMPLE
date Jan 15 2008
solvent CDCl3
file exp
ACQUISITION
sfrq 300.078
tn H1
at 1.998
np 20000
sw 5005.0
fb not used
bs 4
tpwr 58
pw 7.2
d1 2.000
tof 0
nt 32
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -275.6
wp 3122.2
vs 163
sc 0
wc 250
hzmm 12.49
is 500.00
rfl 945.9
rfp 0
th 5
ins 1.000
nm cdc ph
DEC. & VT
dfrq 300.078
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
F
SiEt3
N
H
12
9
8
7
6
5
4
3
2
1
-0
ppm
1.01
2.01
1.14
1.00
9.63
6.79

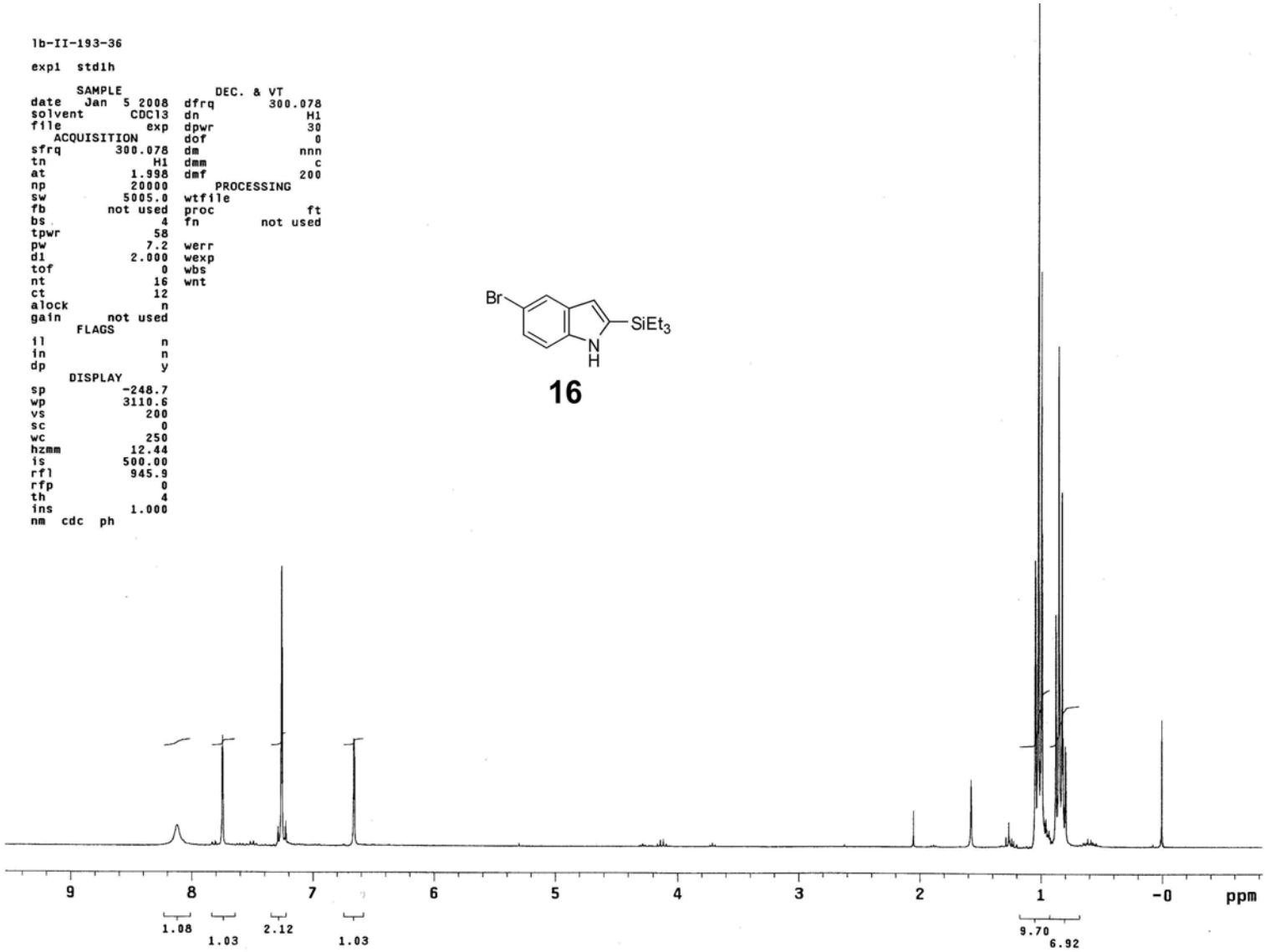

159.629
156.522
138.184
135.466
129.253
129.124
112.509
112.444
111.497
111.368
110.947
110.600
105.309
105.010
77.709
77.288
76.859
7.680
3.732
160
140
120
100
80
60
40
20
0
ppm

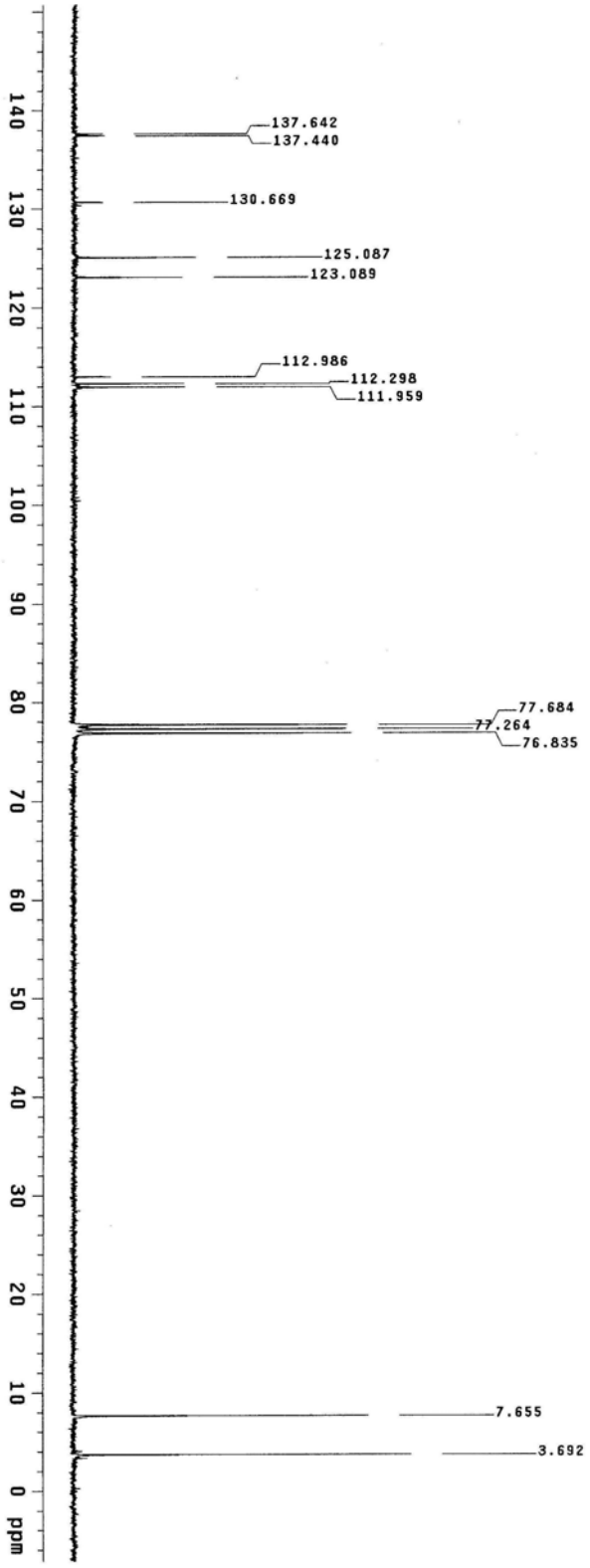
12

F
N
H
SiEt3

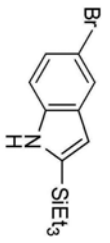

lb-II-209-22
exp1 std1h
SAMPLE
date Jan 12 2008
solvent CDCl3
file exp
ACQUISITION
sfrq 300.078
tn H1
at 1.998
np 20000
sw 5005.0
fb not used
bs 4
tpwr 58
pw 7.2
d1 2.000
tof 0
nt 32
ct 20
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -116.1
wp 2838.4
vs 92
sc 0
wc 250
hzmm 11.35
is 4760.92
rfl 945.9
rfp 0
th 3
ins 1.000
nm cdc ph
DEC. & VT
dfrq 300.078
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
SiEt3
N
H
Cl
14
9
8
7
6
5
4
3
2
1
ppm
0.96
1.09
0.93
1.09
1.00
9.54
7.19


137.027
136.089
130.370
121.601
120.566
119.255
116.489
113.487
77.684
77.264
76.835
7.655
3.716
140
120
100
80
60
40
20
0
ppm
14
Cl
N
H
SiEt3


lb-II-193-36
expl stdlh
SAMPLE
date Jan 5 2008
solvent CDCl3
file exp
ACQUISITION
sfrq 300.078
tn H1
at 1.998
np 20000
sw 5005.0
fb not used
bs 4
tpwr 58
pw 7.2
d1 2.000
tof 0
nt 16
ct 12
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -248.7
wp 3110.6
vs 200
sc 0
wc 250
hzmm 12.44
is 500.00
rfl 945.9
rfp 0
th 4
ins 1.000
nm cdc ph
DEC. & VT
dfrq 300.078
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
Br
SiEt3
N
H
16
9
8
7
6
5
4
3
2
1
-0
ppm
1.08
1.03
2.12
1.03
9.70
6.92


137.642
137.440
130.669
125.087
123.089
112.986
112.298
111.959
77.684
77.264
76.835
7.655
3.692
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm

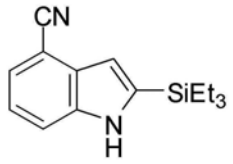
16


Br
N
H
SiEt3

lb-II-240-31

exp1 stdlh

SAMPLE		DEC. & VT	
date	Jan 31 2008	dfrq	300.078
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.078	dm	nnn
tn	H1	dmm	c
at	1.998	dmf	200
np	20000	PROCESSING	
sw	5005.0	wtfile	
fb	not used	proc	ft
bs	4	fn	not used
tpwr	58		
pw	7.2	werr	
d1	2.000	wexp	
tof	0	wbs	
nt	32	wnt	
ct	24		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-187.0		
wp	3134.1		
vs	114		
sc	0		
wc	250		
hzmm	12.54		
is	500.00		
rfl	945.9		
rfp	0		
th	6		
ins	1.000		
nm cdc ph			



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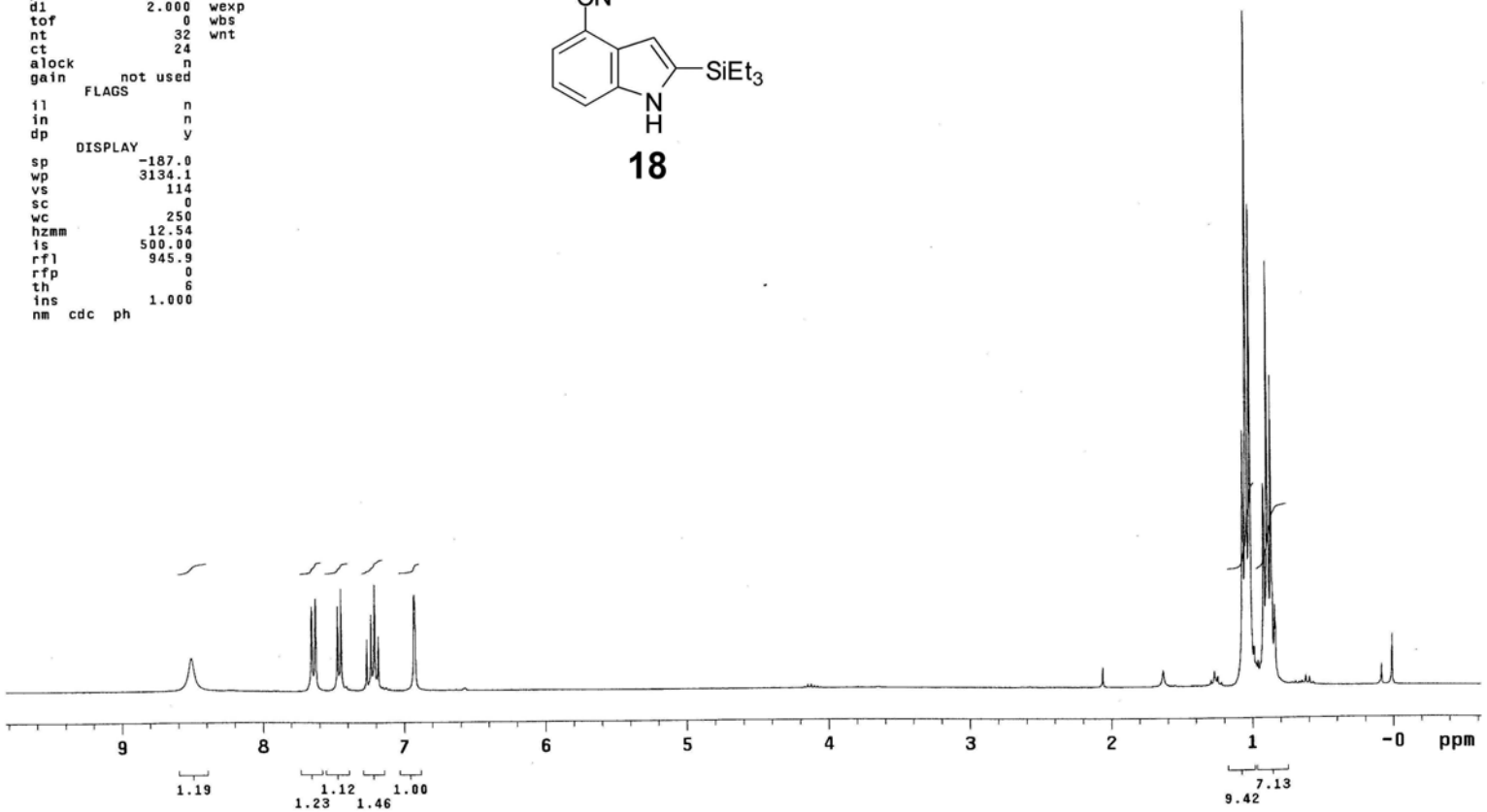
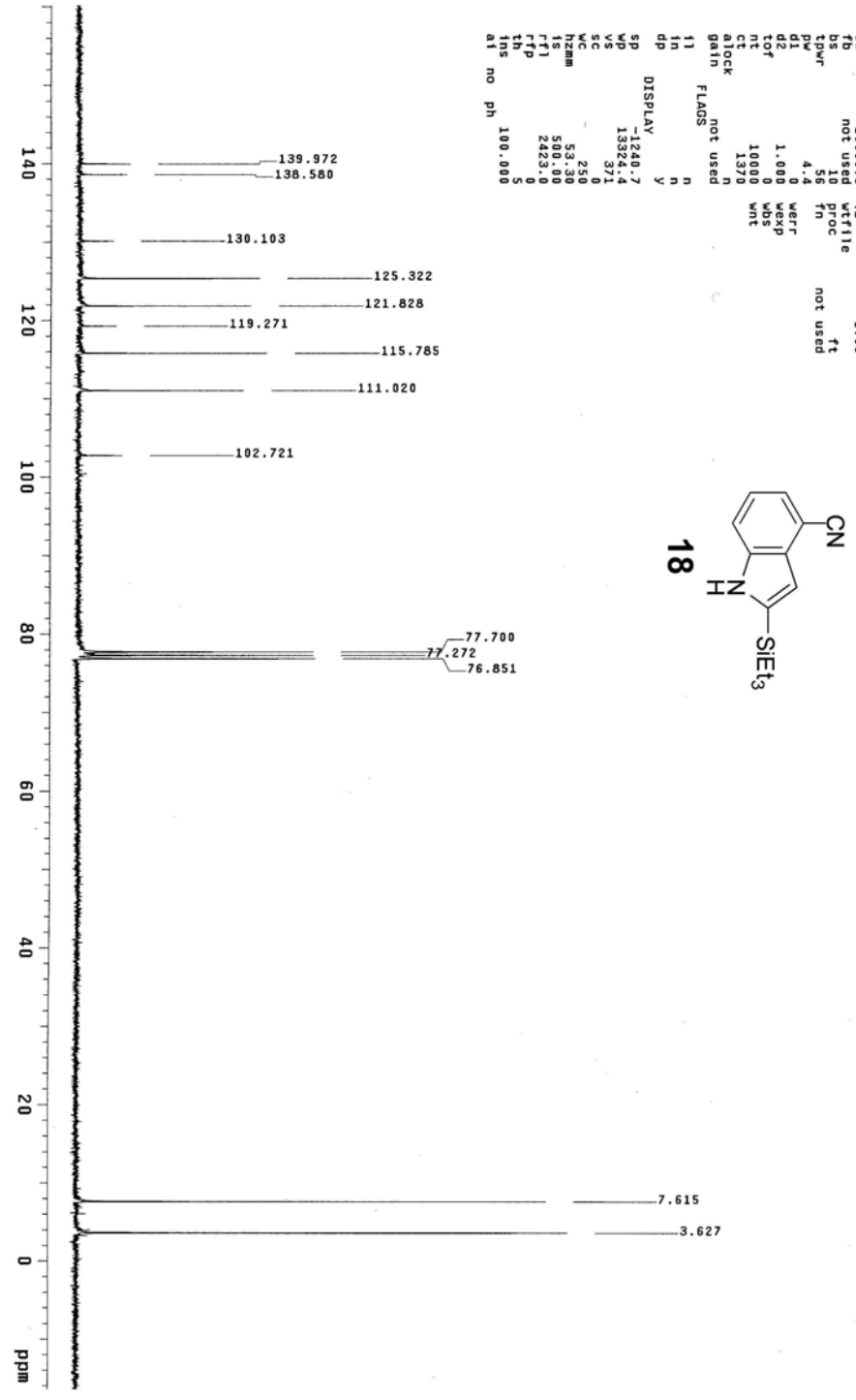
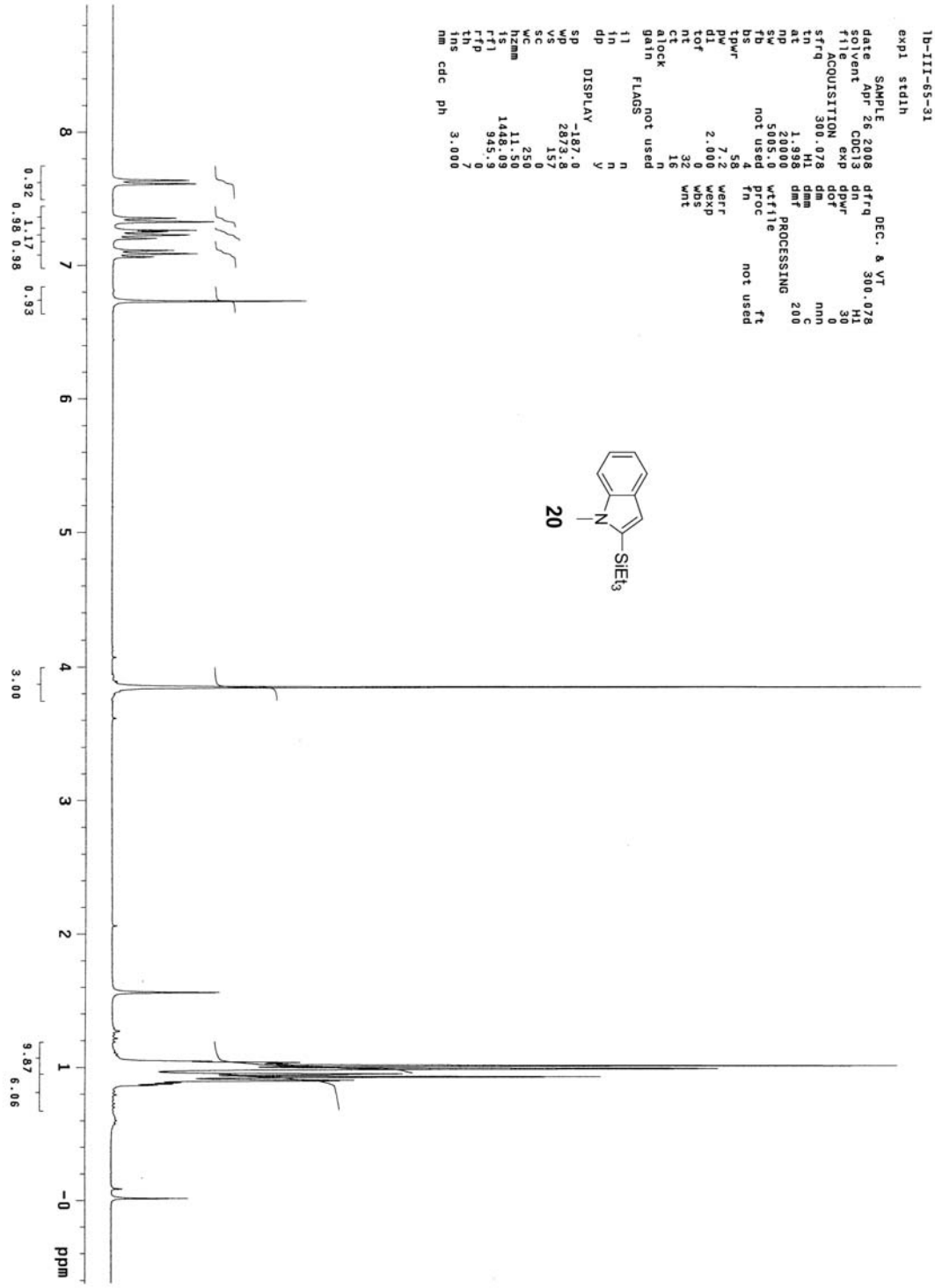
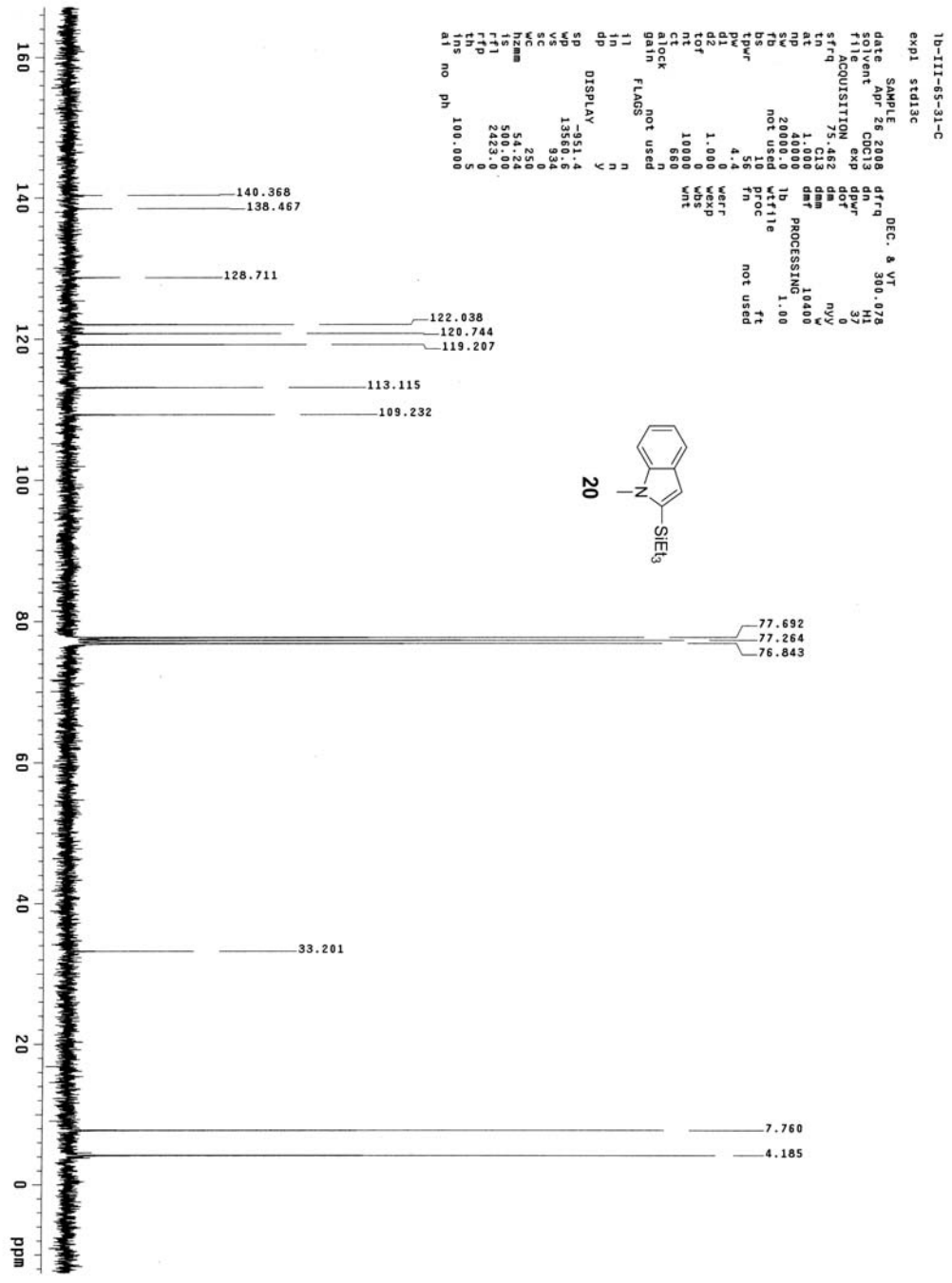
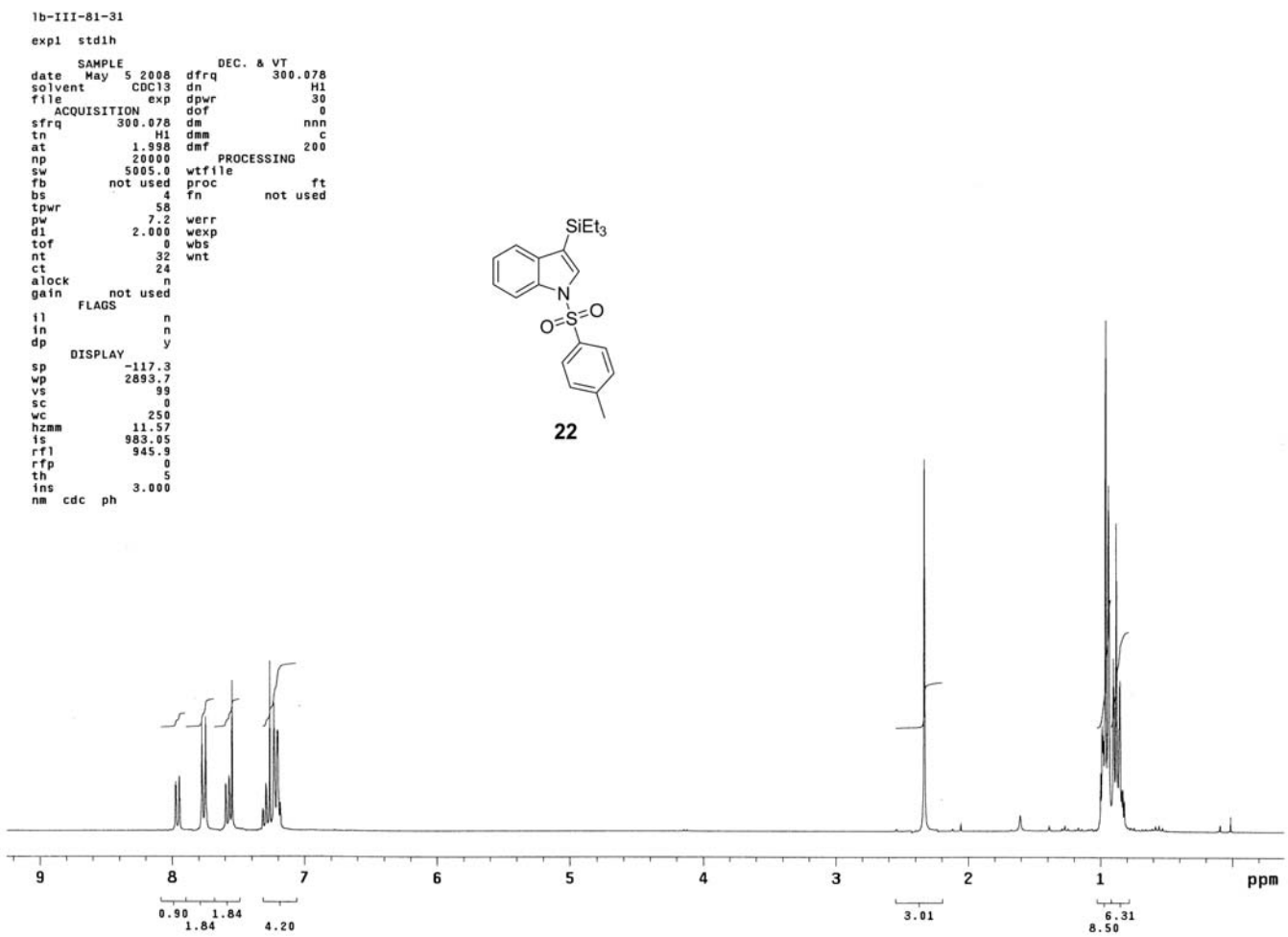
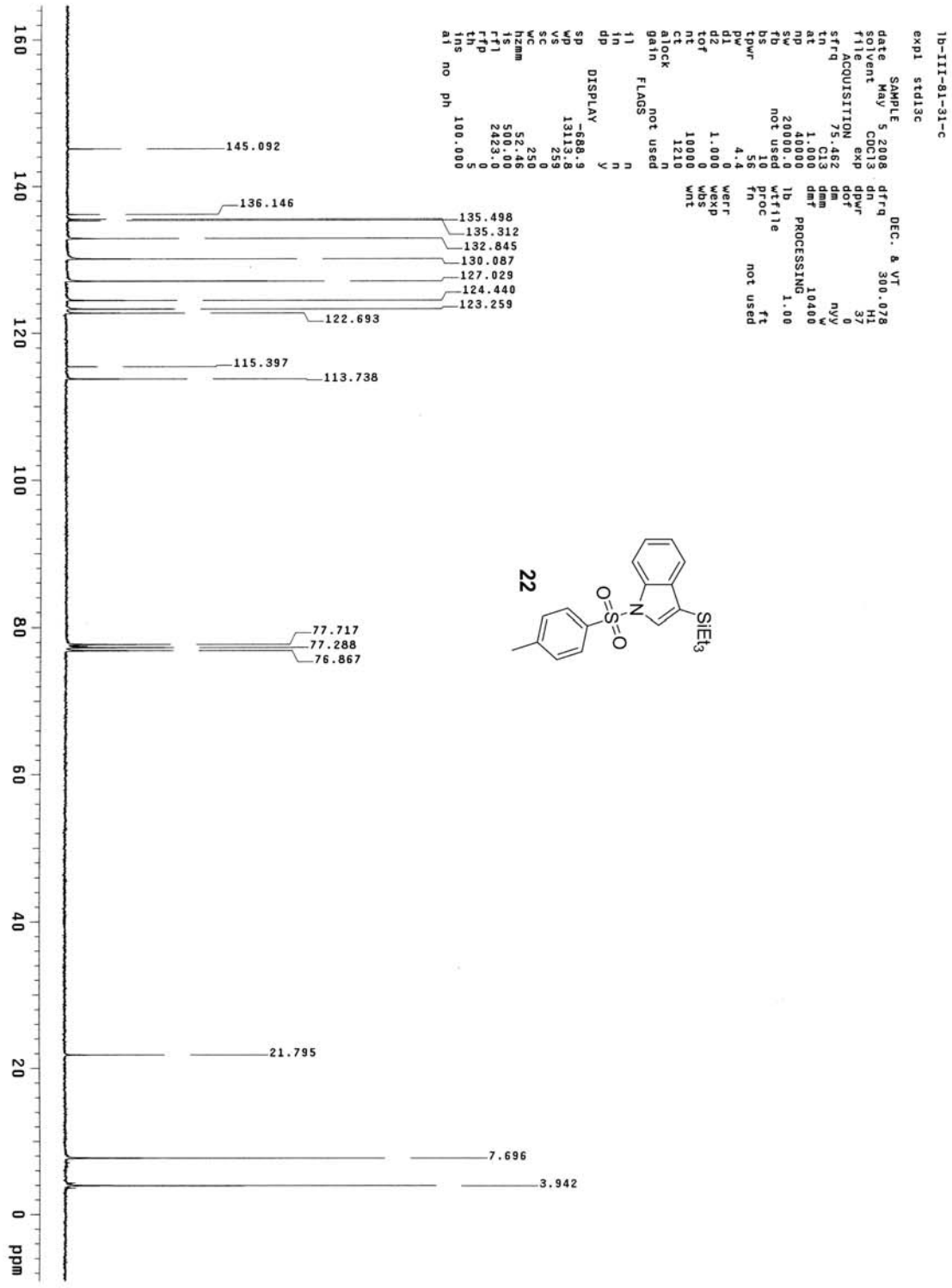



1b-I-240-31-C
exp1 std13c
SAMPLE
date Jan 31 2008
solvent CDCl3
file exp
ACQUISITION
sfrq 75.462
tn C13
at 1.000
np 40000
sw 20000.0
fb not used
bs
tpwr 56
pw 4.4
d1 4.0
d2 1.000
tof 0
nt 10000
ct 1370
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -1240.7
wp 13324.4
vs 371
sc 0
wc 250
hzmm 53.30
fs 500.00
rfl 2423.0
rfp 0
th 5
ins
ai no ph 100.000
dfrq DEC. & VT
dn 300.078
H1
dpwr 37
dof 0
nyy
w
dmm 10400
dmf
lb PROCESSING 1.00
wtfile
proc ft
fn not used
CN
N
H
SiEt3
18
139.972
138.580
130.103
125.322
121.828
119.271
115.785
111.020
102.721
77.700
77.272
76.851
7.615
3.627
140
120
100
80
60
40
20
0
ppm


lb-III-65-31
expl stdlh
SAMPLE DEC. & VT
date Apr 26 2008 dfrq 300.078
solvent CDCl3 dn H1
file exp dpwr 30
ACQUISITION dof 0
sfrq 300.078 dm nnn
tn H1 dmm c
at 1.998 dmf 200
np 20000 PROCESSING
sw 5005.0 wtfile
fb not used proc ft
bs 4 fn not used
tpwr 58
pw 7.2 werr
d1 2.000 wexp
tof 0 wbs
nt 32 wnt
ct 16
alock n
gain n
FLAGS
il n
in n
dp y
DISPLAY
sp -187.0
wp 2873.8
vs 157
sc 0
wc 250
hzmm 11.50
is 1448.09
rfl 945.9
rfp 0
th 7
ins 3.000
nm cdc ph
20
N
SiEt3
8
7
6
5
4
3
2
1
-0
ppm
0.92
1.17
0.93
0.98
0.98
3.00
9.87
6.06


140.368
138.467
128.711
122.038
120.744
119.207
113.115
109.232
77.692
77.264
76.843
33.201
7.760
4.185
ppm
20
SiEt3

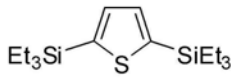

1b-III-81-31
expl stdlh
SAMPLE
date May 5 2008
solvent CDCl3
file exp
ACQUISITION
sfrq 300.078
tn H1
at 1.998
np 20000
sw 5005.0
fb not used
bs 4
tpwr 58
pw 7.2
d1 2.000
tof 0
nt 32
ct 24
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -117.3
wp 2893.7
vs 99
sc 0
wc 250
hzmm 11.57
is 983.05
rfl 945.8
rfp 0
th 5
ins 3.000
nm cdc ph
DEC. & VT
dfrq 300.078
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
SiEt3
N
O=S=O
22
9
8
7
6
5
4
3
2
1
ppm
0.90
1.84
1.84
4.20
3.01
6.31
8.50


lb-III-81-31-c
expl std13c
SAMPLE
date May 5 2008
solvent CDCl3
file exp
ACQUISITION
sfrq 75.462
tn C13
at 1.000
np 40000
sw 20000.0
fb not used
bs 10
tpwr 56
pw 4.4
d1 0
d2 1.000
tof 0
nt 10000
ct 1210
alock n
gain n
FLAGS
fl n
in n
dp y
DISPLAY
sp -688.9
wp 13313.8
vs 259
sc 0
wc 250
hzmm 52.46
is 500.00
rfl 2423.0
rfp 0
th 5
ins
ai no ph
100.000
dfrq DEC. & VT
dn 300.078
dpwr H1
dof 37
dm 0
dmm nyy
dmf w
PROCESSING 10400
lb 1.00
wtfile
proc ft
fn not used
wnt
wbs
wexp
werr
145.092
136.146
135.498
135.312
132.845
130.087
127.029
124.440
123.259
122.693
115.397
113.738
77.717
77.288
76.867
21.795
7.696
3.942
22
SiEt3
160
140
120
100
80
60
40
20
0
ppm

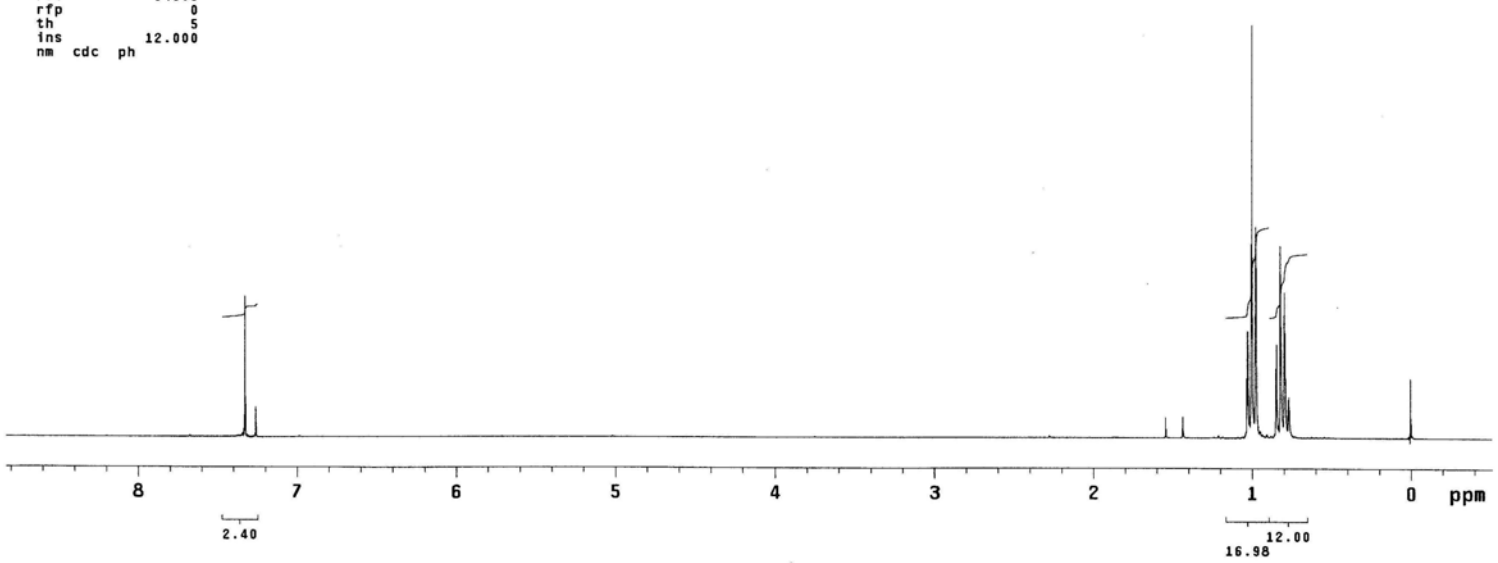
lb-II-19-30

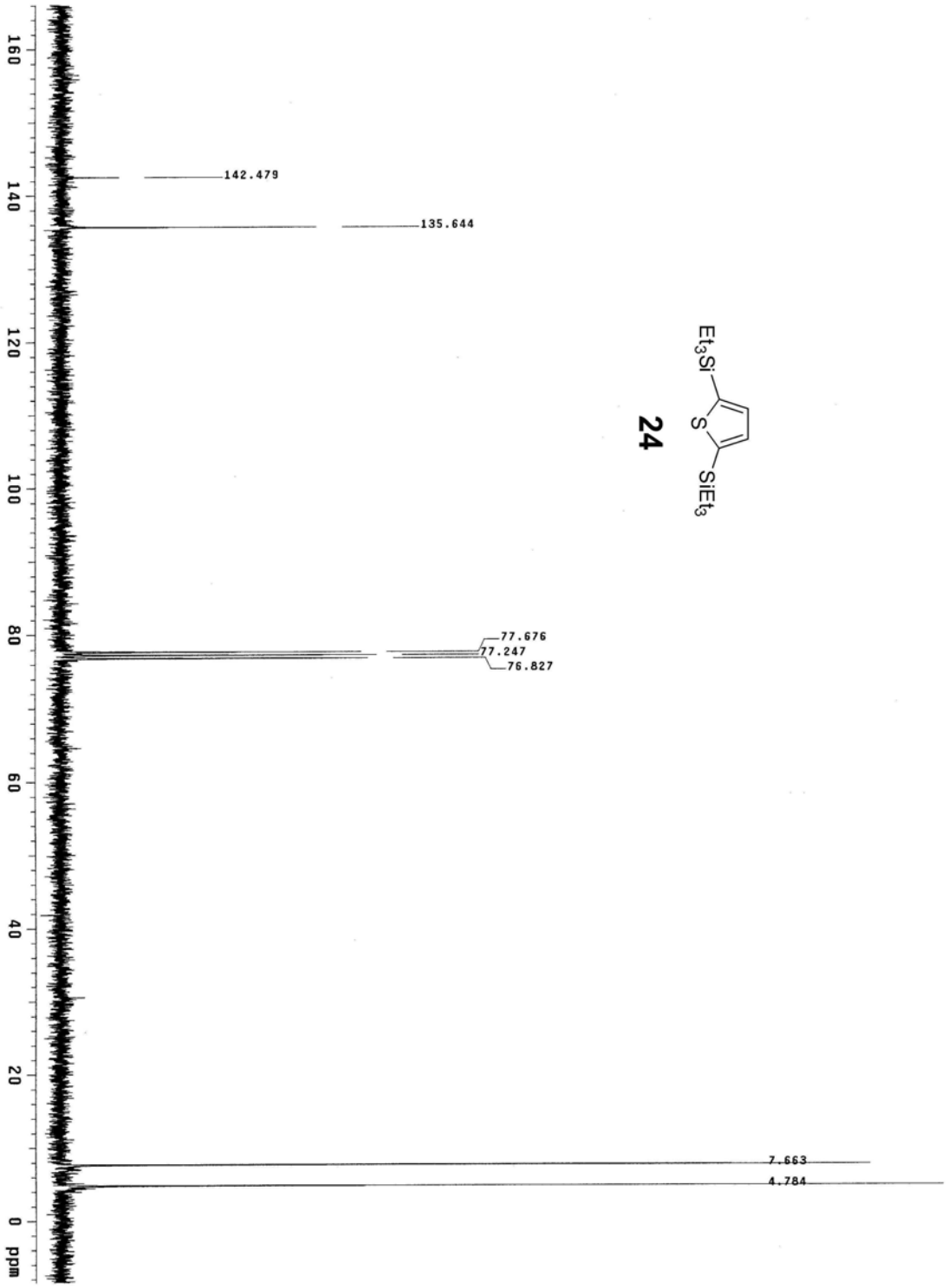
expl std1h

SAMPLE		DEC. & VT	
date	Jan 7 2008	dfrq	300.078
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.078	dm	nnn
tn	H1	dmm	c
at	1.998	dmf	200
np	20000	PROCESSING	
sw	5005.0	wtfile	
fb	not used	proc	ft
bs	4	fn	not used
tpwr	58		
pw	7.2	werr	
d1	2.000	wexp	
tof	0	wbs	
nt	32	wnt	
ct	24		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-153.4		
wp	2803.0		
vs	69		
sc	0		
wc	250		
hzmm	11.21		
is	500.00		
rfl	945.9		
rfp	0		
th	5		
ins	12.000		
nm cdc ph			



24



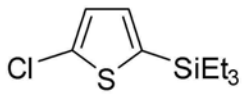

142.479
135.644
77.676
77.247
76.827
7.663
4.784
160
140
120
100
80
60
40
20
0 ppm
Et3Si
S
SiEt3

24

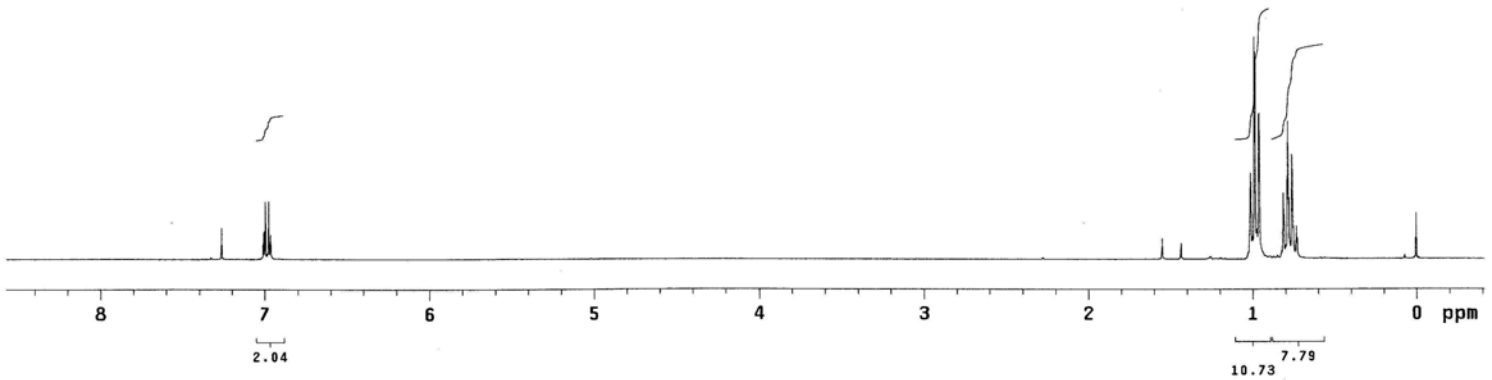
lb-II-227-26

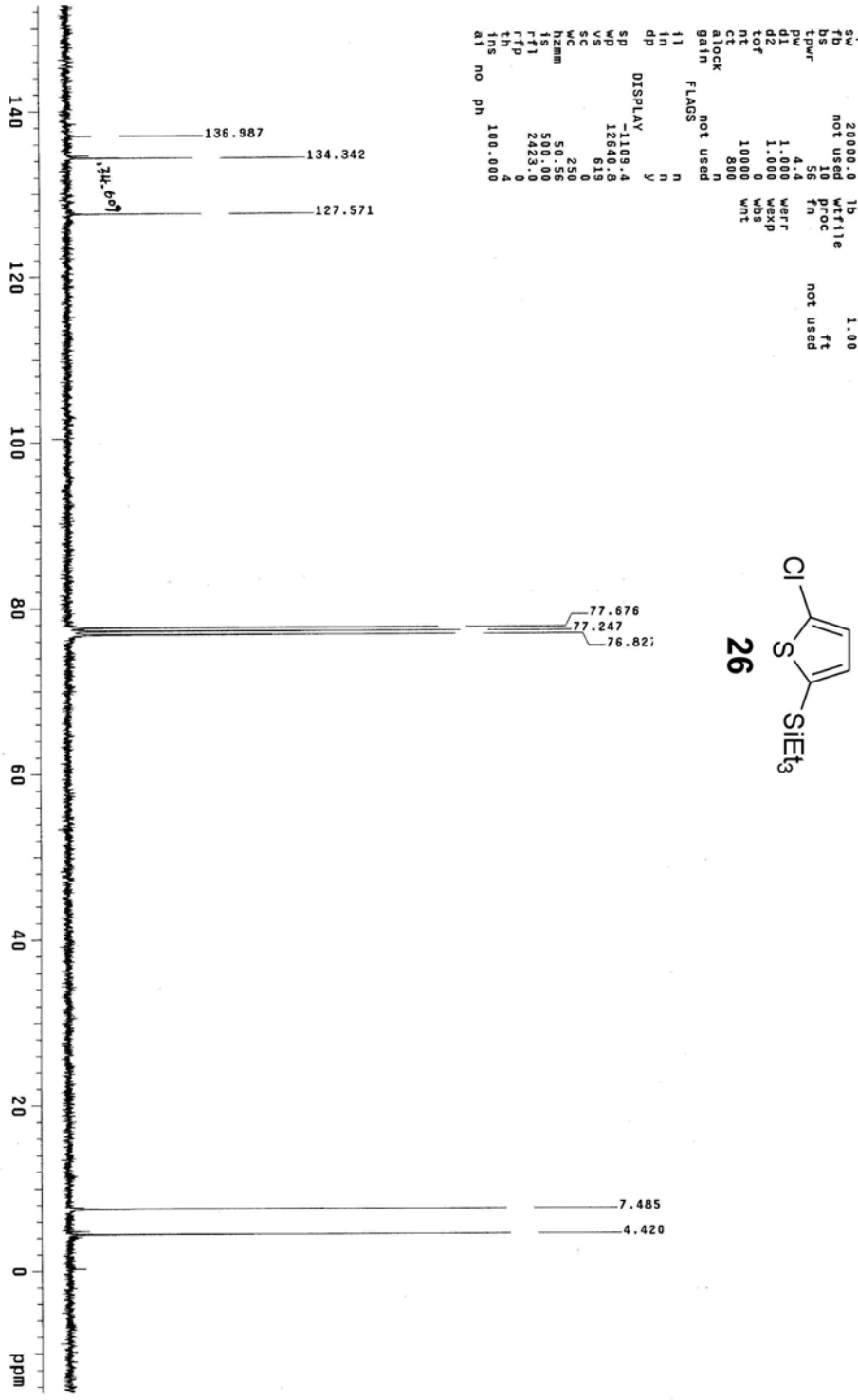
exp1 std1h

SAMPLE		DEC. & VT	
date	Jan 22 2008	dfrq	300.078
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.078	dm	nnn
tn	H1	dmm	c
at	1.998	dmf	200
np	20000	PROCESSING	
sw	5005.0	wtfile	
fb	not used	proc	ft
bs	4	fn	not used
tpwr	58		
pw	7.2	werr	
d1	2.000	wexp	
tof	0	wbs	
nt	32	wnt	
ct	32		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-123.8		
wp	2696.3		
vs	37		
sc	0		
wc	250		
hzmm	10.79		
is	500.00		
rfl	945.9		
rfp	0		
th	6		
ins	2.000		
nm	cdc ph		

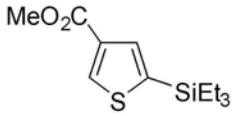


26

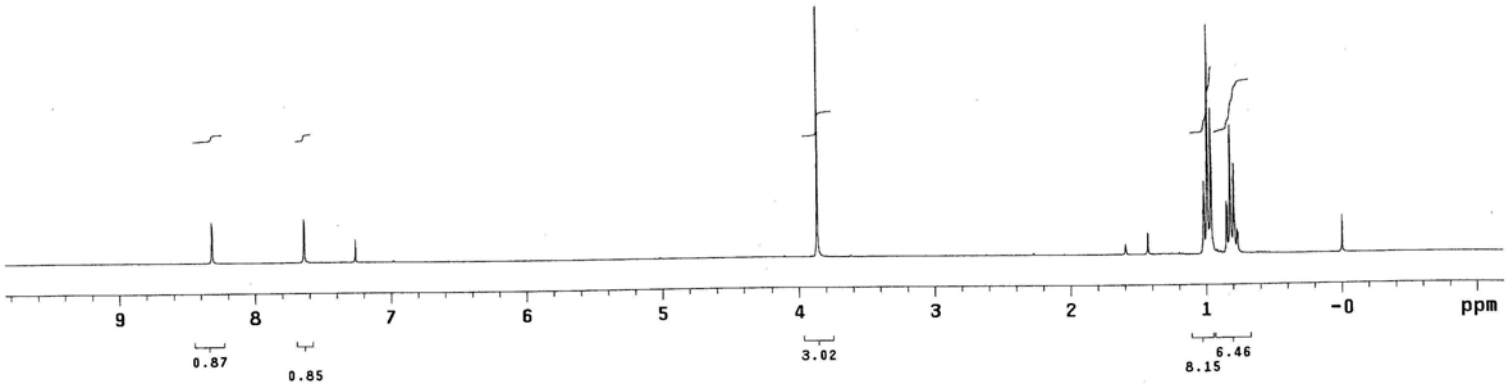


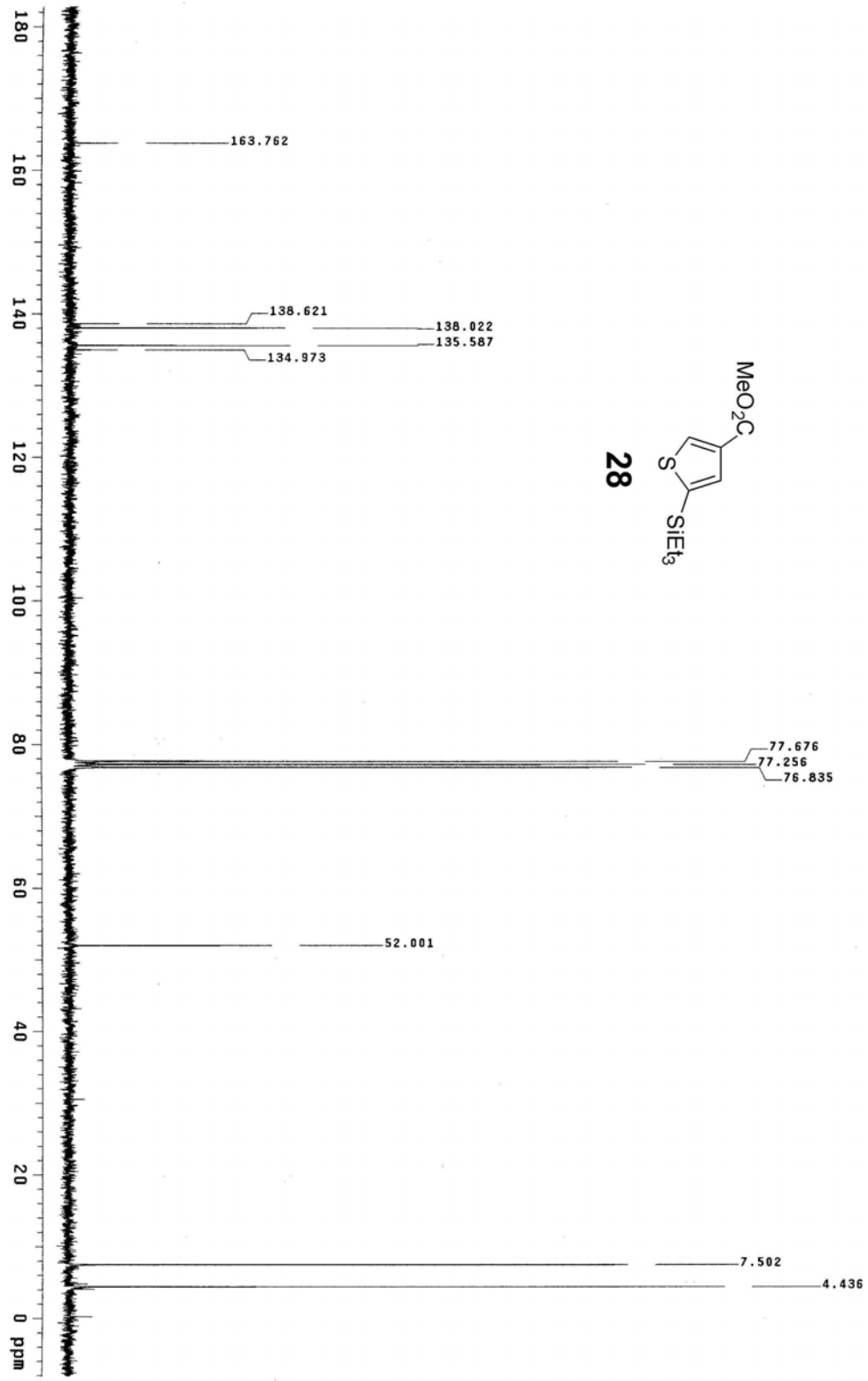

lb-II-227-26-c
expl std13c
SAMPLE
date Jan 22 2008
solvent CDCl3
file ACQUISITION
sfrq 75.462
tn C13
at 1.000
np 40000
sw 20000.0
fb not used
bs
tpwr 56
pw 4.4
d1 1.000
d2 1.000
tof 0
nt 10000
ct 800
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -1109.4
wp 12640.8
vs 619
sc 0
wc 250
hzmm 50.56
fs 500.00
rf1 2423.0
rfp 0
th 4
ins
al no ph
100.000
DEC. & VT
dfrq 300.078
dn H1
dof 37
dpwr 0
nyy
dm y
dmf 10400
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
136.987
134.342
127.571
77.676
77.247
76.827
7.485
4.420
Cl
S
SiEt3
26
140
120
100
80
60
40
20
0
ppm

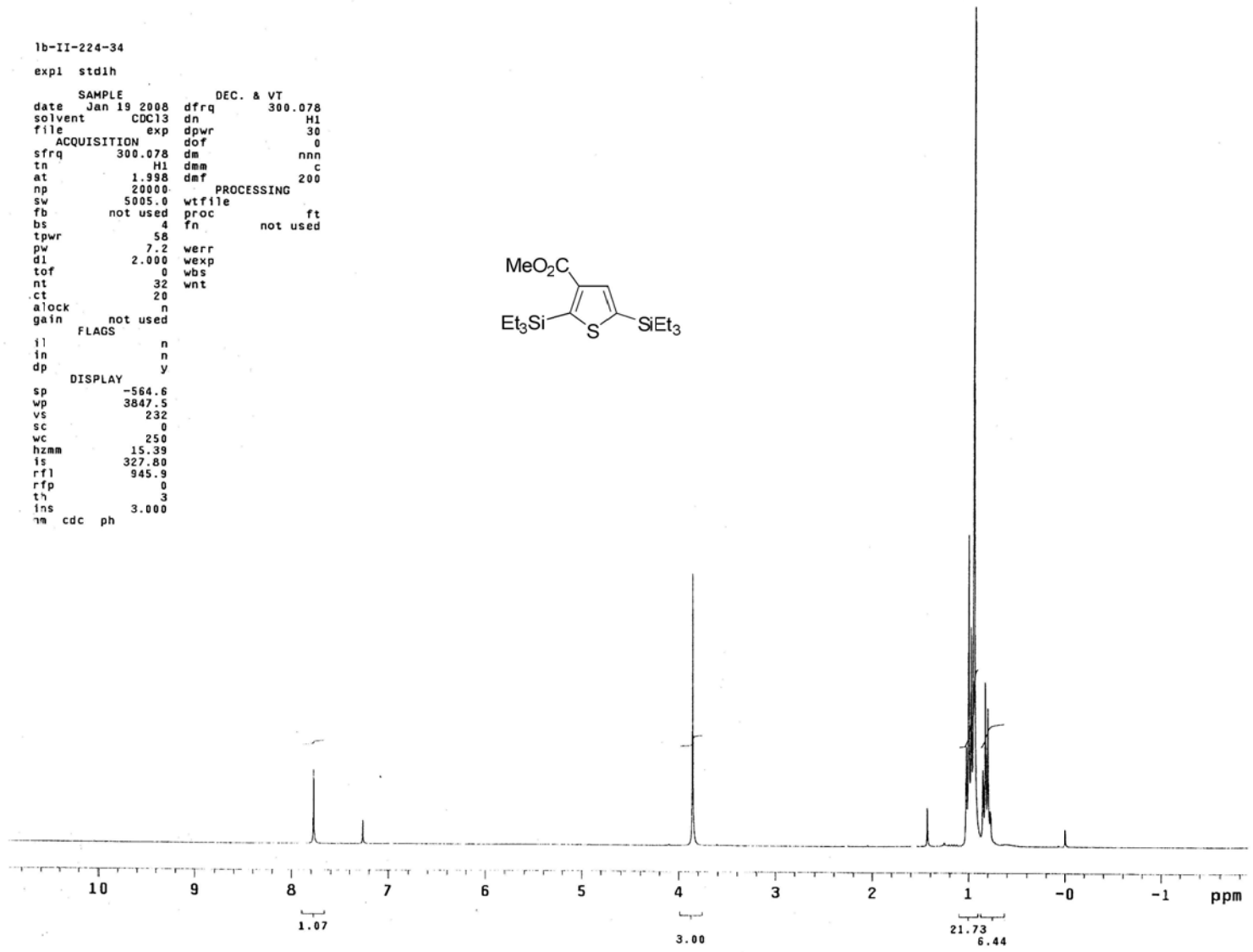
```
lb-II-224-33

exp1  std1h

      SAMPLE
date   Jan 19 2008
solvent        CDCl3
file             exp
   ACQUISITION
sfrq          300.078
tn                 H1
at              1.998
np              20000
sw             5005.0
fb          not used
bs                 4
tpwr              58
pw               7.2
d1             2.000
tof                0
nt                32
ct                24
alock              n
gain        not used
      FLAGS
il                 n
in                 n
dp                 y
     DISPLAY
sp             -358.4
wp             3311.6
vs                 41
sc                  0
wc                250
hzmm            13.25
is             500.00
rfl             945.9
rfp                 0
th                  8
ins             3.000
nm  cdc  ph

      DEC. & VT
dfrq          300.078
dn                 H1
dpwr               30
dof                 0
dm                nnn
dmm                 c
dmf               200
    PROCESSING
wtfile
proc               ft
fn          not used
werr
wexp
wbs
wnt
```

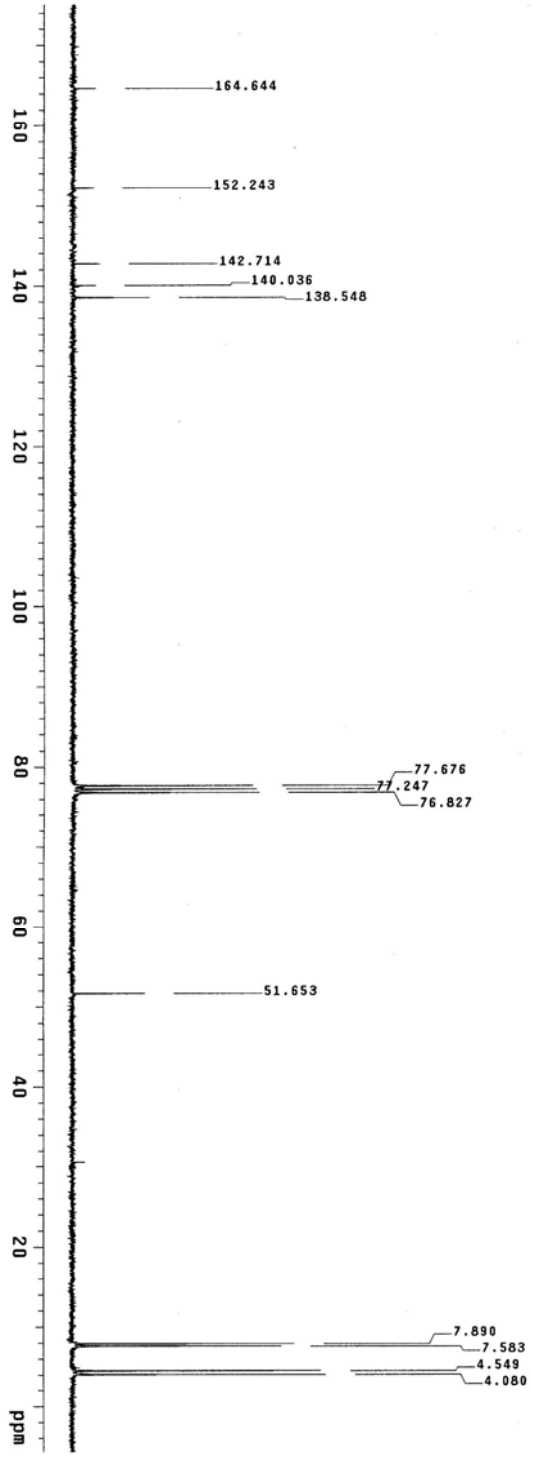


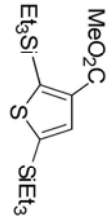
28




180
160
140
120
100
80
60
40
20
0 ppm
163.762
138.621
138.022
135.587
134.973
77.676
77.256
76.835
52.001
7.502
4.436
28
MeO2C
S
SiEt3


lb-II-224-34
expl stdlh
SAMPLE
date Jan 19 2008
solvent CDCl3
file exp
ACQUISITION
sfrq 300.078
tn H1
at 1.998
np 20000
sw 5005.0
fb not used
bs 4
tpwr 58
pw 7.2
d1 2.000
tof 0
nt 32
ct 20
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -564.6
wp 3847.5
vs 232
sc 0
wc 250
hzmm 15.39
is 327.80
rfl 945.9
rfp 0
th 3
ins 3.000
nm cdc ph
DEC. & VT
dfrq 300.078
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
MeO2C
Et3Si
SiEt3
S
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm
1.07
3.00
21.73
6.44

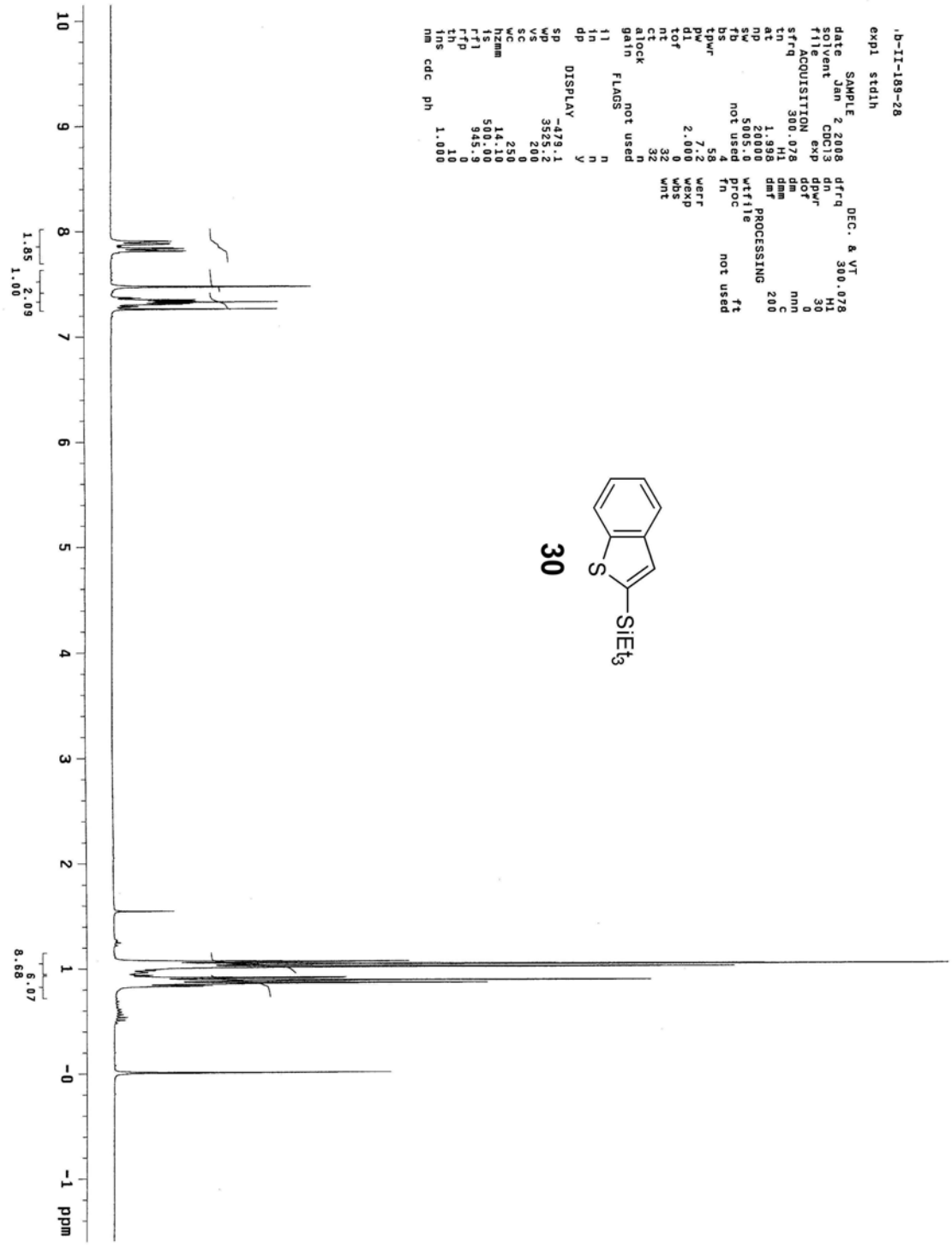


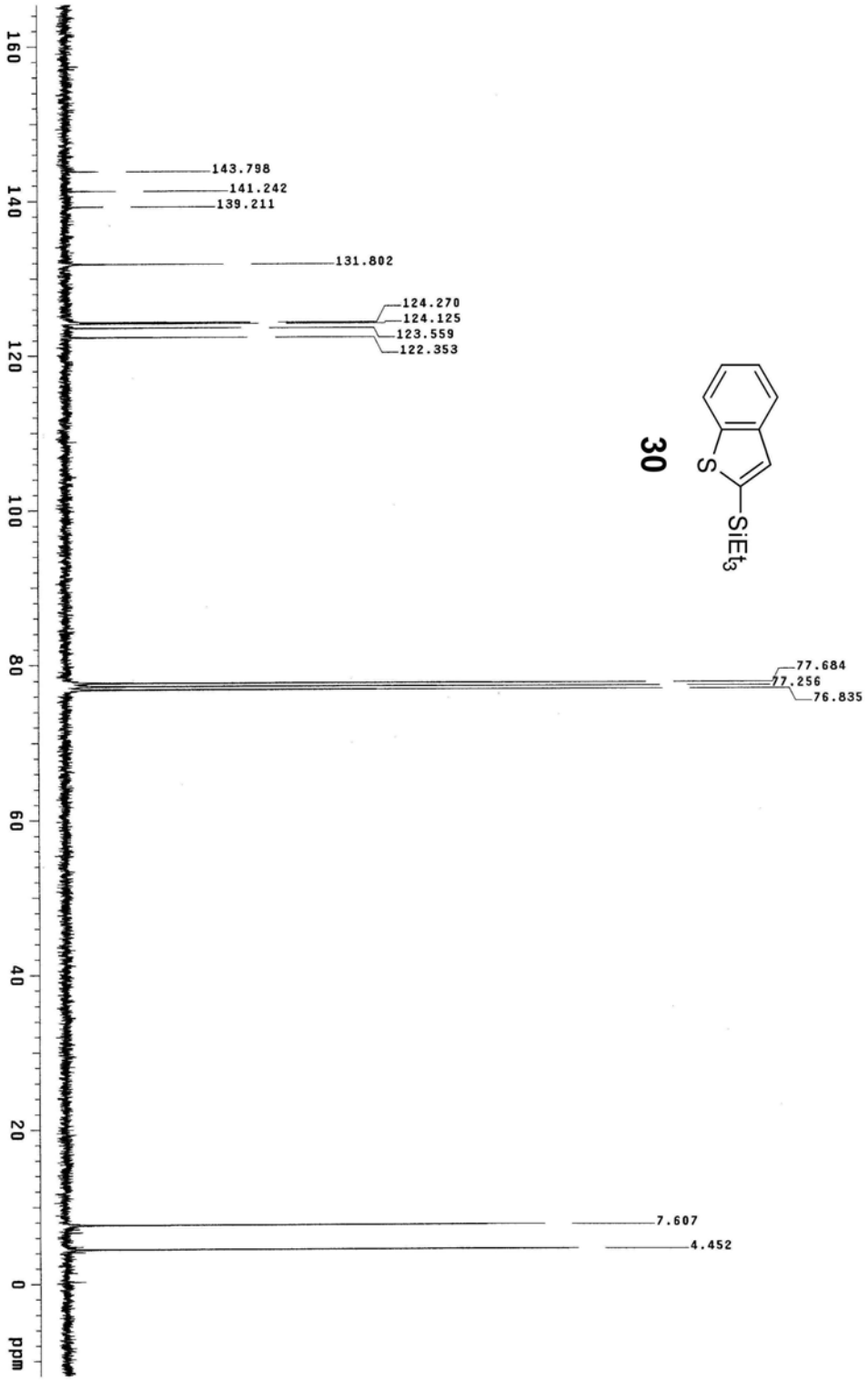


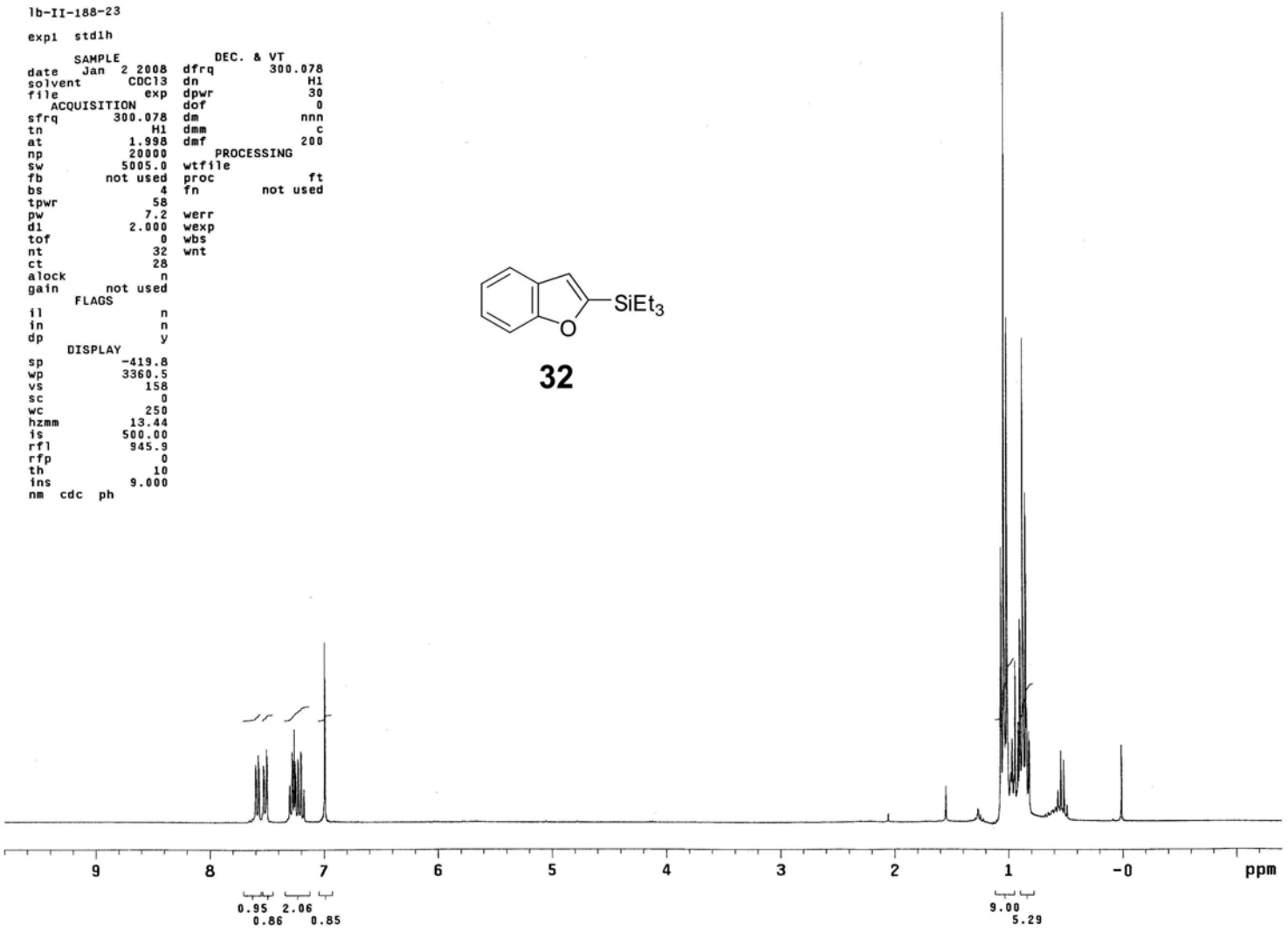
1b-II-224-34-c

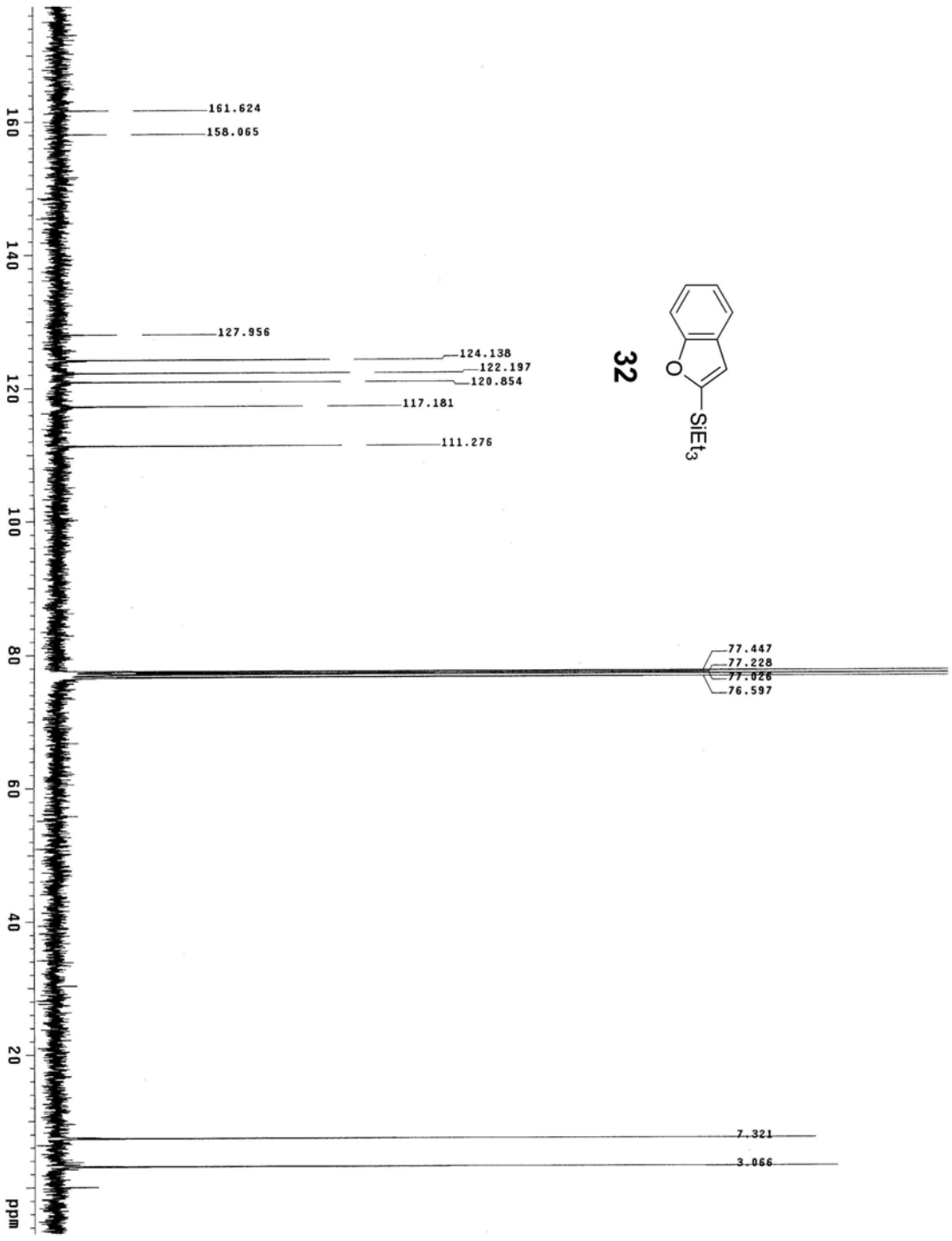
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "pele300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.000 sec
Width 20000.0 Hz
500 repetitions
OBSERVE C13, 75.4541519 MHz
DECOUPLE H1, 300.0775554 MHz
Power 37 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 9 hr, 8 min, 38 sec


ib-II-189-28
expl stdih
SAMPLE
date Jan 2 2008
solvent CDCl3
file exp
ACQUISITION
sfrq 300.078
tn H1
at 1.998
np 20000
sw 5005.0
fb not used
bs 4
tpwr 58
pw 7.2
d1 2.000
tof 0
nt 32
ct 32
alock n
gain n
FLAGS
fl not used
in n
dp y
DISPLAY
sp -479.1
wp 3525.2
vs 200
sc 0
wc 250
hzmm 14.10
ins 500.09
rfl 945.0
rfp 0
th 10
ins 1.000
nm cdc ph
DEC. & VT
dfrq 300.078
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
S
SiEt3
30
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm
1.85
2.09
1.00
6.07
8.68


160
140
120
100
80
60
40
20
0
ppm
143.798
141.242
139.211
131.802
124.270
124.125
123.559
122.353
77.684
77.256
76.835
7.607
4.452
30
S
SiEt3


lb-II-188-23
exp1 stdlh
SAMPLE
date Jan 2 2008
solvent CDCl3
file exp
ACQUISITION
sfrq 300.078
tn H1
at 1.998
np 20000
sw 5005.0
fb not used
bs 4
tpwr 58
pw 7.2
d1 2.000
tof 0
nt 32
ct 28
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -419.8
wp 3360.5
vs 158
sc 0
wc 250
hzmm 13.44
is 500.00
rfl 945.9
rfp 0
th 10
ins 9.000
nm cdc ph
DEC. & VT
dfrq 300.078
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
SiEt3
O
32
9
8
7
6
5
4
3
2
1
-0
ppm
0.95 2.06 0.85
0.86
9.00
5.29


161.624
158.065
127.956
124.138
122.197
120.854
117.181
111.276
77.447
77.228
77.026
76.597
7.321
3.066
ppm
160
140
120
100
80
60
40
20
32
O
SiEt3

STANDARD 1H OBSERVE

expl std1h

SAMPLE		DEC. & VT	
date	Jan 11 2008	dfrq	300.078
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.078	dm	nnn
tn	H1	dmm	c
at	1.998	dmf	200
np	20000	PROCESSING	
sw	5005.0	wtfile	
fb	not used	proc	ft
bs	4	fn	not used
tpwr	58		
pw	7.2	werr	
d1	2.000	wexp	
tof	0	wbs	
nt	32	wnt	
ct	20		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-137.8		
wp	2534.7		
vs	82		
sc	0		
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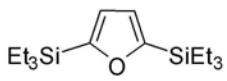
nm cdc ph			



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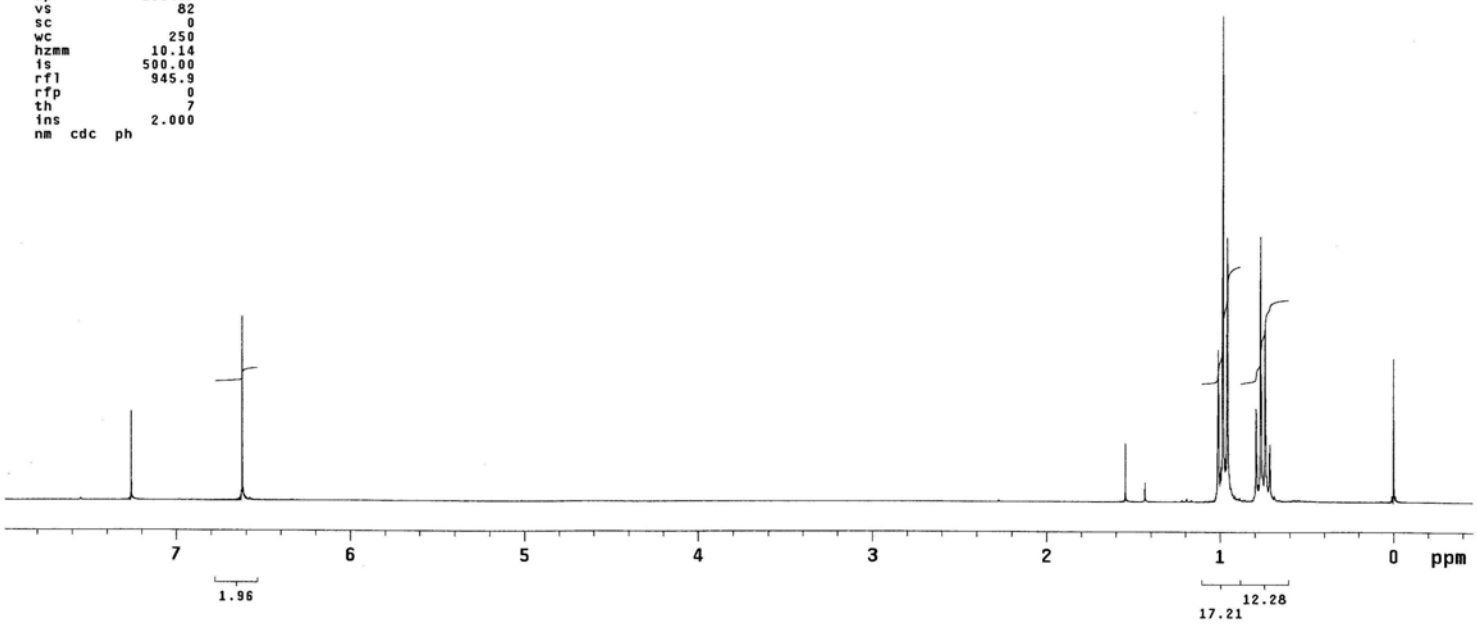

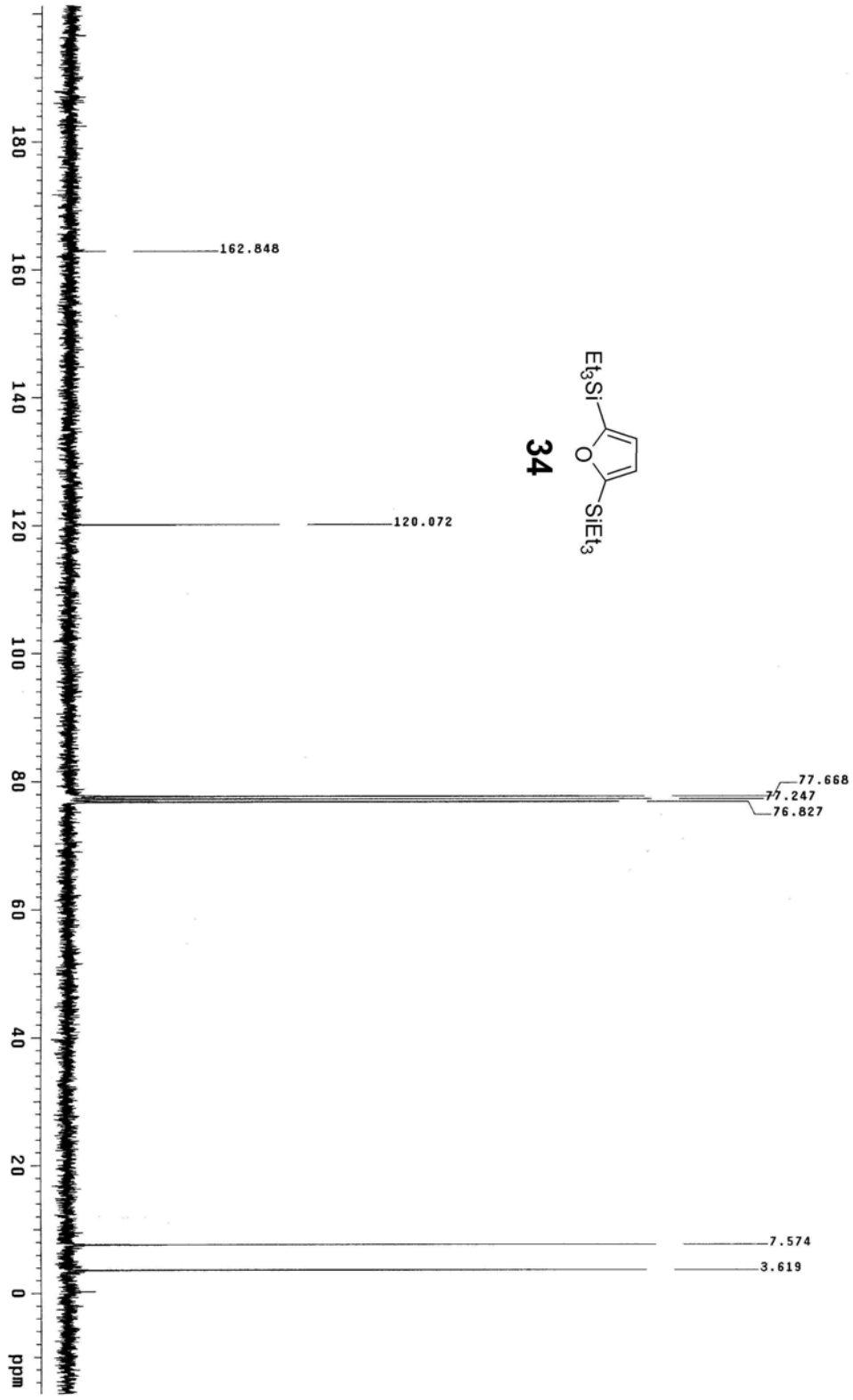
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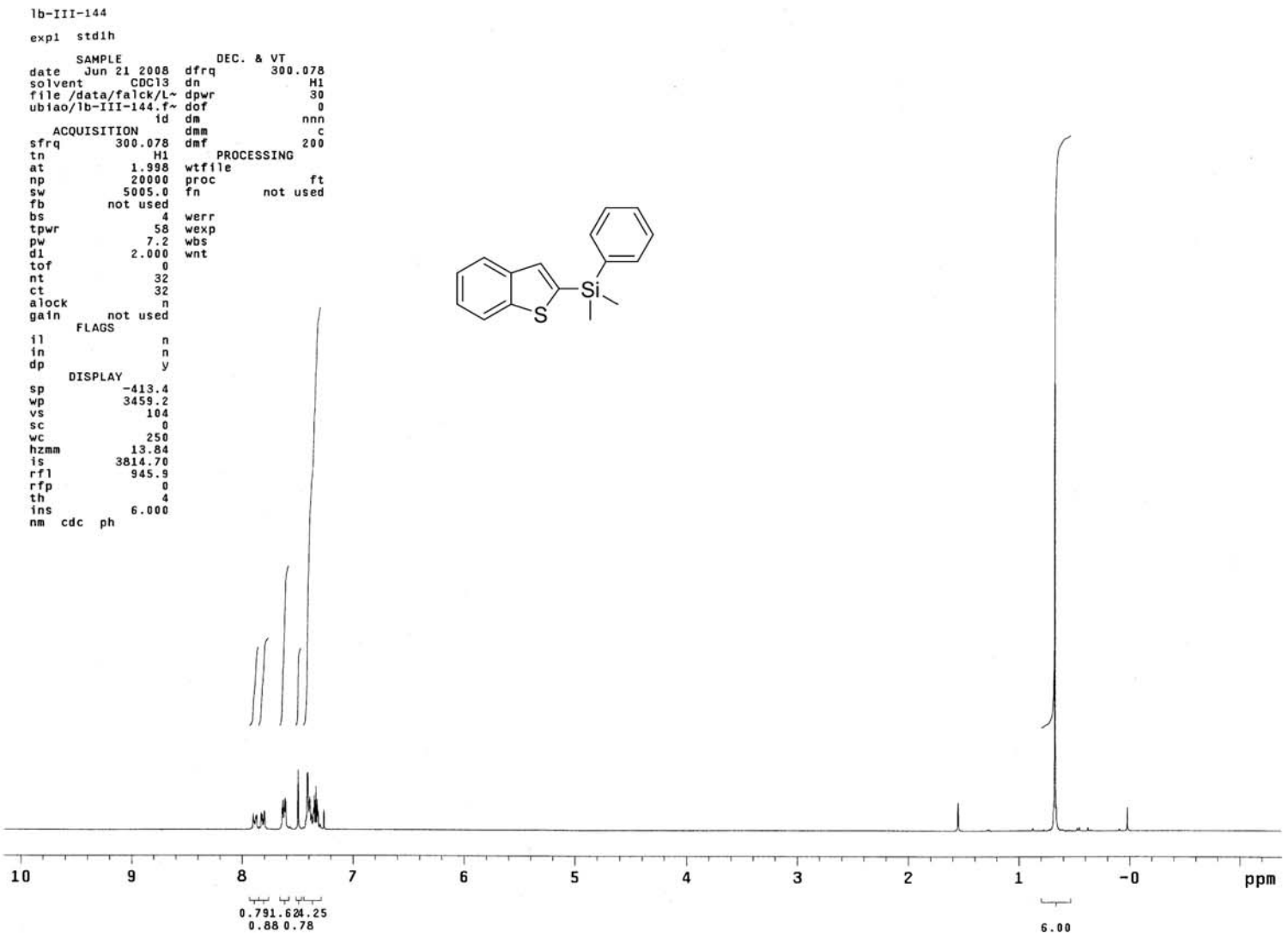
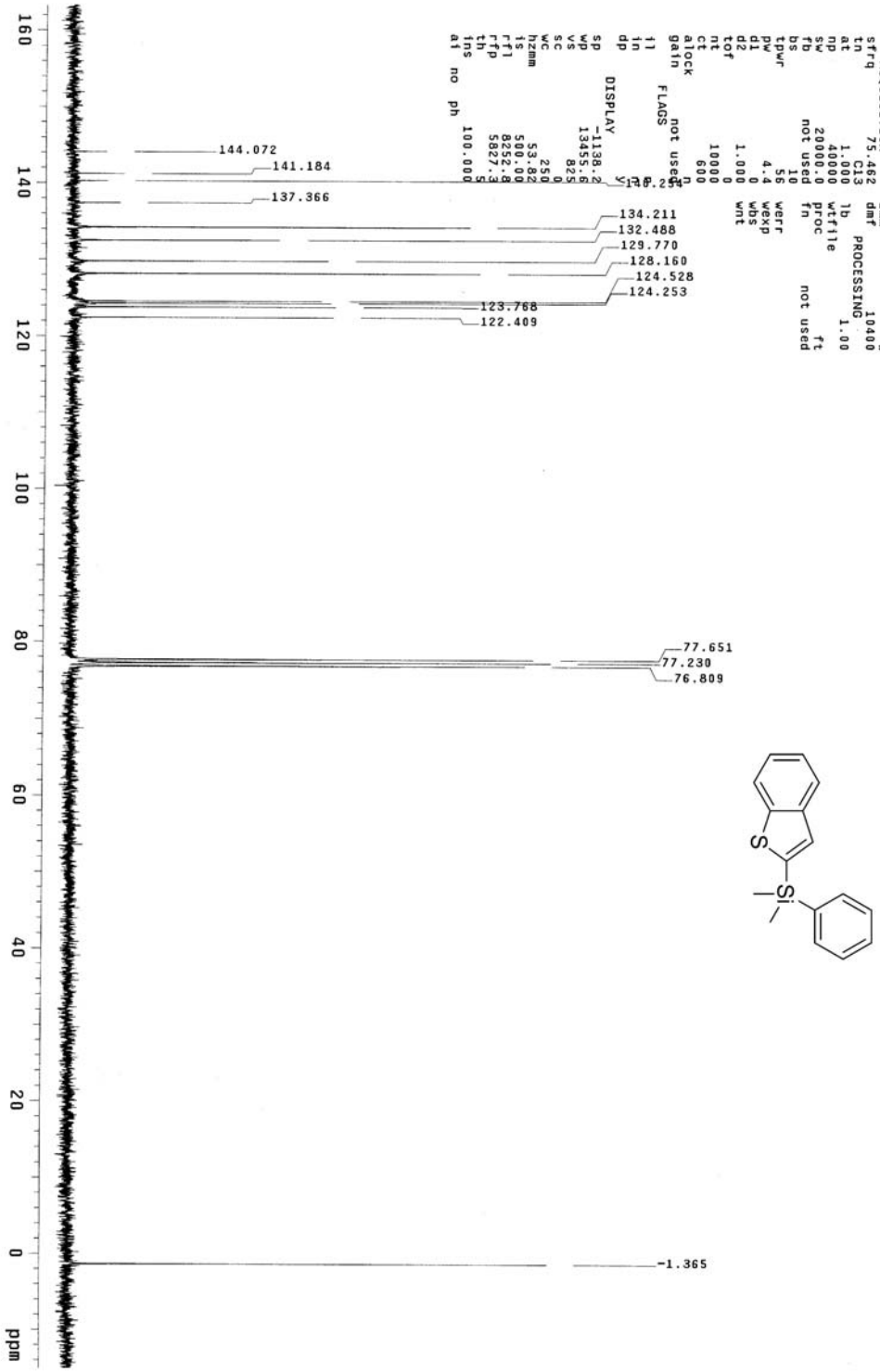
120.072
77.668
77.247
76.827
7.574
3.619
ppm
180
160
140
120
100
80
60
40
20
0
Et3Si
O
SiEt3
34


lb-III-144
expl std1h
SAMPLE
date Jun 21 2008
solvent CDCl3
file /data/falck/L~
ubiao/lb-III-144.f~
id
ACQUISITION
sfrq 300.078
tn H1
at 1.998
np 20000
sw 5005.0
fb not used
bs 4
tpwr 58
pw 7.2
d1 2.000
tof 0
nt 32
ct 32
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -413.4
wp 3459.2
vs 104
sc 0
wc 250
hzmm 13.84
is 3814.70
rfl 945.9
rfp 0
th 4
ins 6.000
nm cdc ph
DEC. & VT
dfrq 300.078
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
Si
S
10
9
8
7
6
5
4
3
2
1
-0
ppm
0.79 1.62 4.25
0.88 0.78
6.00


lb-III-144-C
exp1 std13c
SAMPLE
date Jun 21 2008
solvent CDCl3
file /data/falck/L~ dpwr
ubiao/lb-III-144-C~ dof
.fid dm
ACQUISITION nyy
sfrq 75.462 dmf 10400
tn C13 PROCESSING w
at 1.000 lb 1.00
np 40000 wtfile
sw 20000.0 proc ft
fb not used fn not used
bs 10
tpwr 56 werr
pw 4.4 wexp
d1 0 wbs
d2 1.000 wnt
tof 0
nt 10000
ct 600
alock n
gain not used
FLAGS
il n
in y
dp y
DISPLAY
sp -1138.2
wp 13455.6
vs 825
sc 0
wc 250
hzmm 53.82
is 500.00
rfl 8252.8
rfp 5827.3
th 5
ins 100.000
ai no ph
DEC. & VT
dfrq 300.078
dn H1
dof 37
dm 0
nyy
w
1.00
1.00
ft
not used
144.072
141.184
137.366
134.211
132.488
129.770
128.160
124.528
124.253
123.768
122.409
77.651
77.230
76.809
-1.365
S
Si
160
140
120
100
80
60
40
20
0
ppm