Supporting Information for the Communication Entitled "A reversible two-electron redox system involving a divalent lead species"

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Contents

General procedure. All experiments were performed under argon atmosphere in a glovebox or using standard Schlenk techniques. THF, toluene, hexane and pentane for reactions and benzene-d_6, THF-d_8 and toluene-d_8 for NMR mesurement were purified potassium mirror before used. ^{1}H NMR (500 MHz), ^{13}C NMR (101 MHz), ^{7}Li NMR (194 MHz) and ^{207}Pb NMR (105 MHz) were recorded on a Bruker DPX-400 Cryo, an AVANCE-500 or an AVANCE-500T.

Preparation of THF-stabilized plumbacyclopentadienylidene. A THF (3 mL) solution of 1,4-dilithio-1,3-butadiene **2**[S1] (138 mg including two diethyl ether molecules, 0.23 mmol) was added to a THF (3 mL) suspension of lead dichloride (70 mg, 0.25 mmol) and the resulting mixture was stirred at room temperature for 1.5 h. After evaporation of the solvent, an inorganic salt insoluble in pentane was removed by filtration followed by recrystallization from pentane to afford THF-stabilized plumbacyclopentadienylidene **1** (134 mg, 0.17 mmol, 74%).[S2]

Reduction of THF-stabilized plumbacyclopentadienylidene by lithium. Toluene (1.3 mL) was added to a mixture of plumbacyclopentadienylidene coordinated by two THF molecules **1** (172 mg, 0.22 mmol) and lithium (15 mg, 2.2 mmol) at room temperature, and the mixture was stirred for 9

hours. After removal of remaining lithium, the filtrate was concentrated and recrystallized from hexane and toluene (5 : 1) to afford dilithioplumbole **3** (127 mg including two THF molecules, 0.16 mmol, 73%) as green crystals. **3**: Mp: 190 ˚C (dec.). ^{1}H NMR (500 MHz, C_6D_6): δ 0.30 (s, 12H, Si*Me*$_2$^{t}Bu), 1.09–1.11 (m, 8H, *thf*), 1.23 (s, 18H, SiMe$_2$^{t}*Bu*), 3.34–3.36 (m, 8H, *thf*), 6.95–6.98 (m, 2H, *Ph*), 7.03–7.05 (m, 4H, *Ph*), 7.09–7.12 (m, 4H, *Ph*); ^{13}C NMR (101 MHz, C_6D_6): δ 3.56 (1˚, Si*Me*$_2$^{t}Bu, $J_{Pb–C}$ = 33 Hz), 17.04 (4˚, SiMe$_2$^{t}*Bu*), 25.15 (2˚, *thf*), 29.33 (1˚, SiMe$_2$^{t}*Bu*, $J_{Pb–C}$ = 21 Hz), 69.81 (2˚, *thf*), 124.86 (3˚, *Ph*), 126.75 (3˚, *Ph*), 132.09 (3˚, *Ph*), 158.71 (4˚, C_{ipso}, $J_{Pb–C}$ = 40 Hz), 159.38 (4˚, C_{β}, $J_{Pb–C}$ = 57 Hz), 213.47 (4˚, C_{α}, $J_{Pb–C}$ = 860 Hz); ^{7}Li NMR (194 MHz, C_6D_6): δ –3.5; ^{7}Li NMR (194 MHz, THF-C_6D_6): δ –2.3; ^{7}Li NMR (194 MHz, THF-d_8, 203 K): δ –5.0, –2.2; ^{29}Si NMR (99 MHz, C_6D_6): δ 5.6; ^{207}Pb NMR (105 MHz, C_6D_6): δ 2572.5. As the compound is highly sensitive to air and moisture, results for its elemental analysis were not reproducible or sufficient.

Oxidation of dilithioplumbole 3 by ferrocenium tetrafluoroborate. THF (3 mL) was added to a mixture of dilithioplumbole **3** (19 mg including two THF molecules, 0.024 mmol) and ferrocenium tetrafluoroborate (13 mg, 0.048 mmol) at room temperature. After stirring for a few miniutes, the ^{1}H and ^{13}C NMR spectra of the reaction mixture revealed the quantitative formation of THF-stabilized plumbacyclopentadienylidene **1**.

Oxidation of dilithioplumbole 3 by tris(pentafluorophenyl)borane. Benzene-d_6 (0.5 mL) was added to a mixture of dilithioplumbole **3** (25 mg including two THF molecules, 0.031 mmol) and tris(pentafluorophenyl)borane (31 mg, 0.061 mmol) at room temperature. After addition of a few drops of THF, the ^{1}H and ^{13}C NMR spectra of the reaction mixture revealed the quantitative formation of THF-stabilized plumbacyclopentadienylidene **1**.

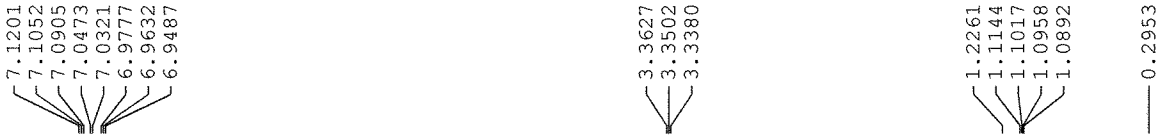
Reaction of dilithioplumbole 3 with 1,2-dibromoethane. A toluene solution of 1,2-dibromoethane (0.39 mol/L; 0.1 mL, 0.04 mmol) was added to a toluene (1 mL) solution of dilithioplumbole **3** (67 mg including two THF molecules, 0.084 mmol) at room temperature. After evaporation of volatile substances, an inorganic salt insoluble in hexane was removed by filtration. Concentration of the filtrate followed by recrystallization of the residue from THF and hexane (1 : 10) afforded 1,1'-dilithiobiplumbole **4** (48 mg including two THF and a hexane molecules, 0.031 mmol, 74%) as red crystals. **4**: Mp: 90 ˚C (dec.). ^{7}Li NMR (194 MHz, THF-C_6D_6): δ –0.3; ^{29}Si NMR (99 MHz, THF-C_6D_6): δ 4.0; ^{207}Pb NMR (105 MHz, THF-C_6D_6): δ 1390.6. The ^{1}H NMR signals are all broadening and therefore cannot be properly assigned, see the NMR chart shown below. Measurement of the ^{13}C NMR spectrum was not successful because the compound decomposed during the overnight accumulation. As the compound is highly sensitive to air and moisture, results for its elemental analysis were not reproducible or sufficient.

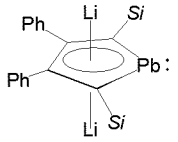
References

(S1) (a) M. Saito, M. Nakamura, T. Tajima and M. Yoshioka, *Angew. Chem., Int. Ed.*, 2007, **46**, 1504; (b) T. Kuwabara, J.-D. Guo, S. Nagase, M. Minoura, R. H. Herber and M. Saito, *Organometallics*, 2014, **33**, 2910.

(S2) M. Saito, T. Akiba, M. Kaneko, T. Kawamura, M. Abe, M. Hada and M. Minoura, *Chem. Eur. J.*, 2013, **19**, 16946.

TBDMS Ph dilithioplumbole 1H in C6D6





3

Si = Si^{t}BuMe$_2$

BRUKER

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INSTRUM	spect
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PULPROG	zg30
TD	65536
SOLVENT	C6D6
NS	8
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1719923 sec
RG	40.3
DW	48.400 usec
DE	6.50 usec
TE	300.2 K
D1	1.00000000 sec
TD0	1

======== CHANNEL f1 ========

NUC1	1H
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PL1	2.60 dB
PL1W	15.32226563 W
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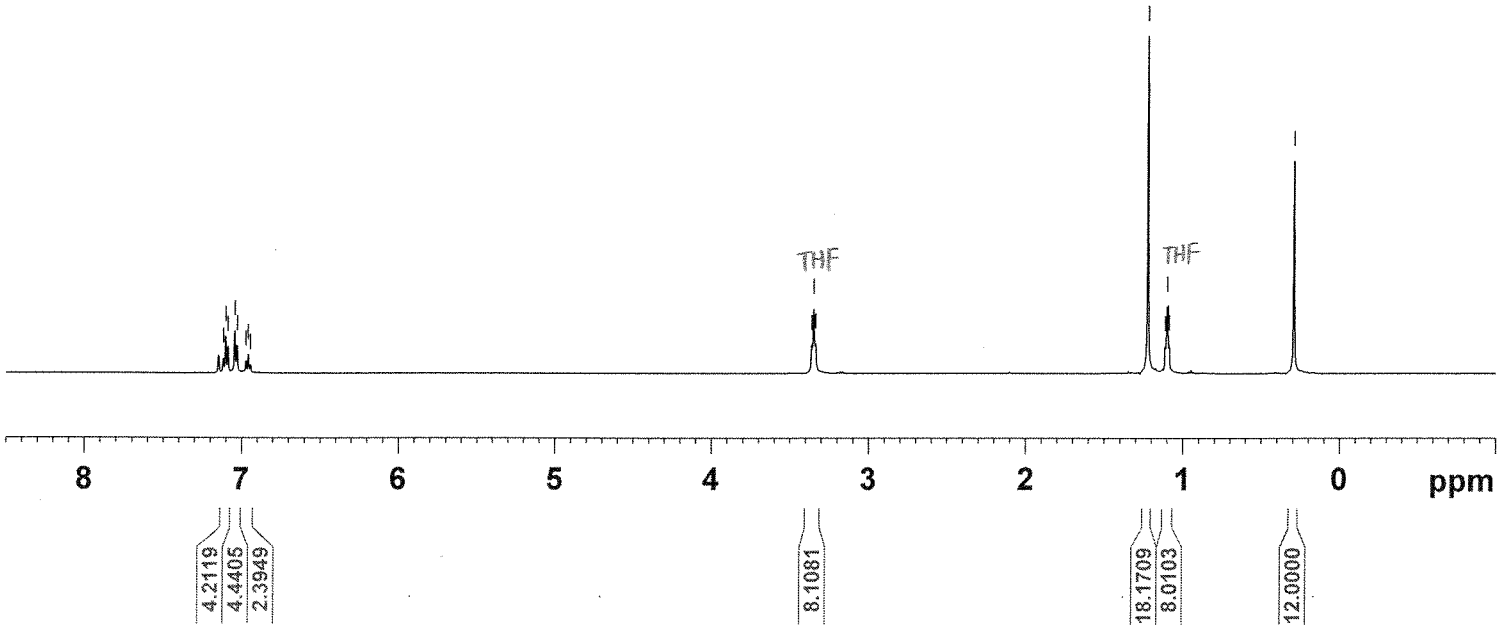
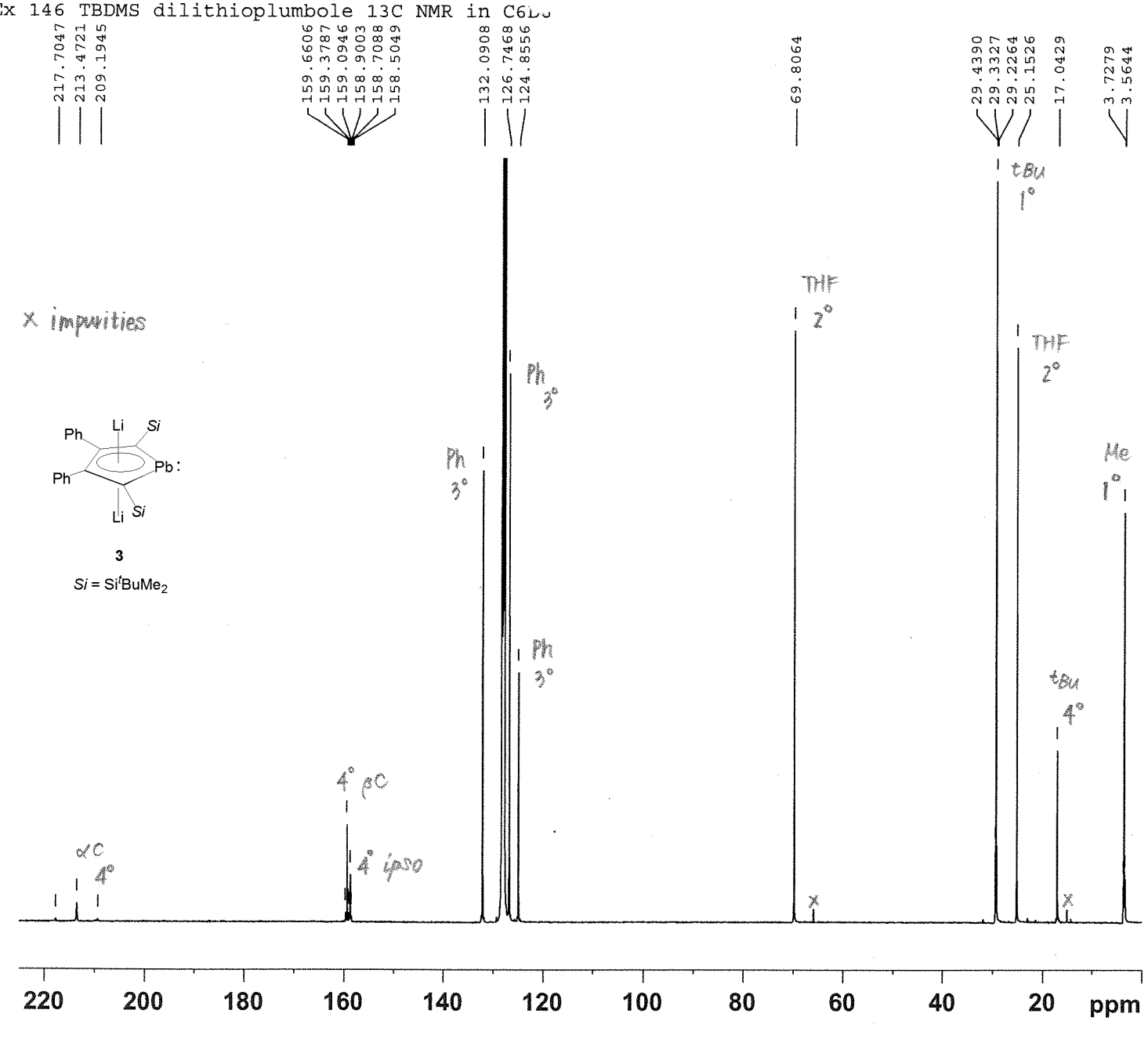


Ex 146 TBDMS dilithioplumbole 13C NMR in C6D6



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PROCNO	1

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PULPROG	zgpg30
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SWH	29761.904 Hz
FIDRES	0.454131 Hz
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D11	0.03000000 sec
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======== CHANNEL f1 ========

NUC1	13C
P1	12.00 usec
PLW1	15.50000000 W
SFO1	100.6248425 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PLW2	5.19999981 W
PLW12	0.14444000 W
PLW13	0.11700000 W
SFO2	400.1316005 MHz

F2 - Processing parameters

SI	32768
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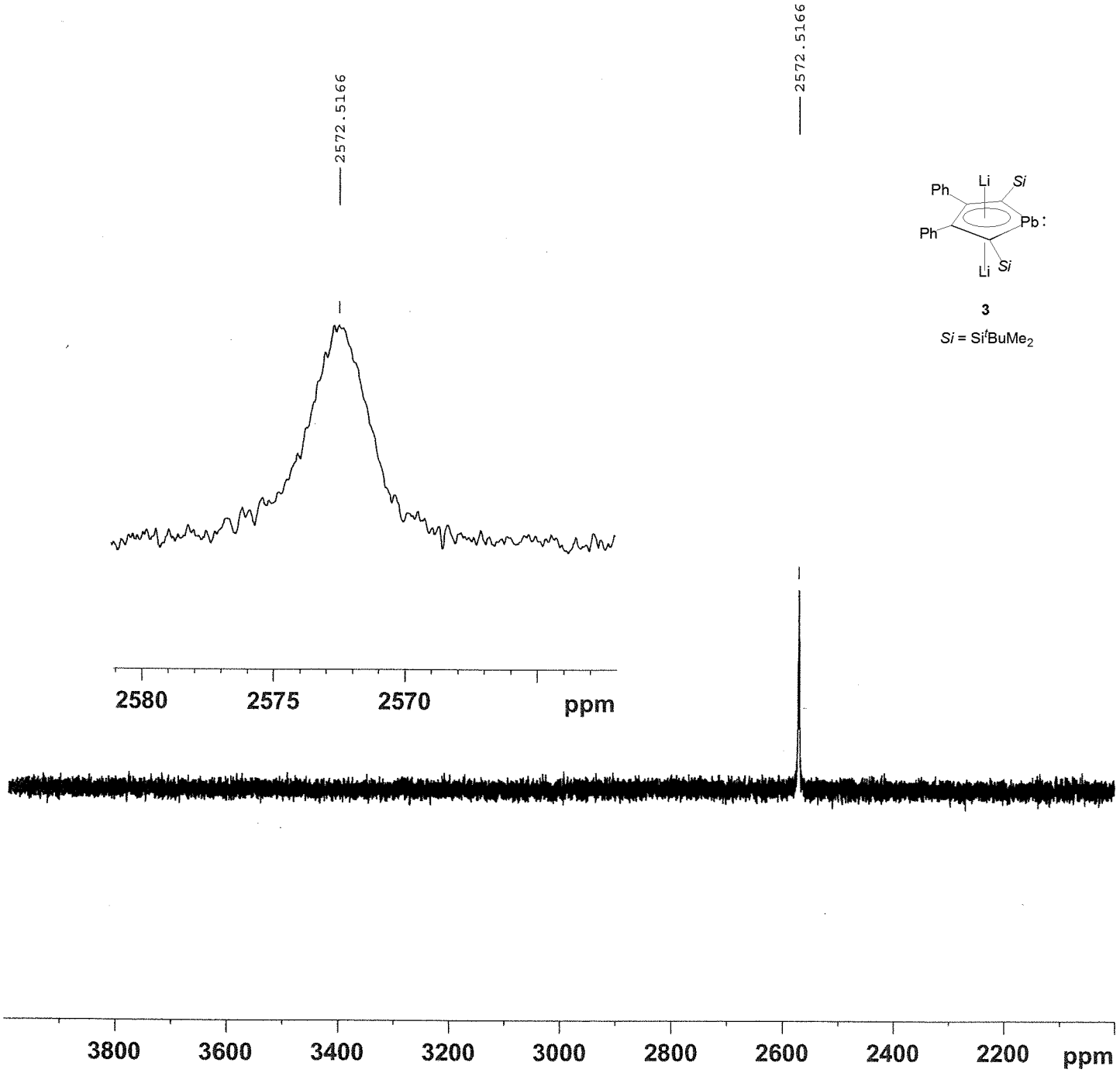
TBDMS Ph dilithioplumbole 207Pb in C6D6



3

Si = Si^{t}BuMe$_2$

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PROCNO	1	
Date_	20130517	
Time	18.02	
INSTRUM	spect	
PROBHD	5 mm PABBO BB-	
PULPROG	zgpg	
TD	65536	
SOLVENT	C6D6	
NS	3016	
DS	2	
SWH	208333.328	Hz
FIDRES	3.178914	Hz
AQ	0.1573364	sec
RG	203	
DW	2.400	usec
DE	65.00	usec
TE	300.6	K
D1	2.00000000	sec
D11	0.03000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	207Pb	
P1	12.00	usec
PL1	0.00	dB
SFO1	104.9230988	MHz
======== CHANNEL f2 ========		
CPDPRG2	waltz16	
NUC2	1H	
PCPD2	80.00	usec
PL2	2.40	dB
PL12	19.02	dB
PL13	22.02	dB
PL2W	15.17711735	W
PL12W	0.33051354	W
PL13W	0.16564916	W
SFO2	500.0320005	MHz
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SF	104.6094793	MHz
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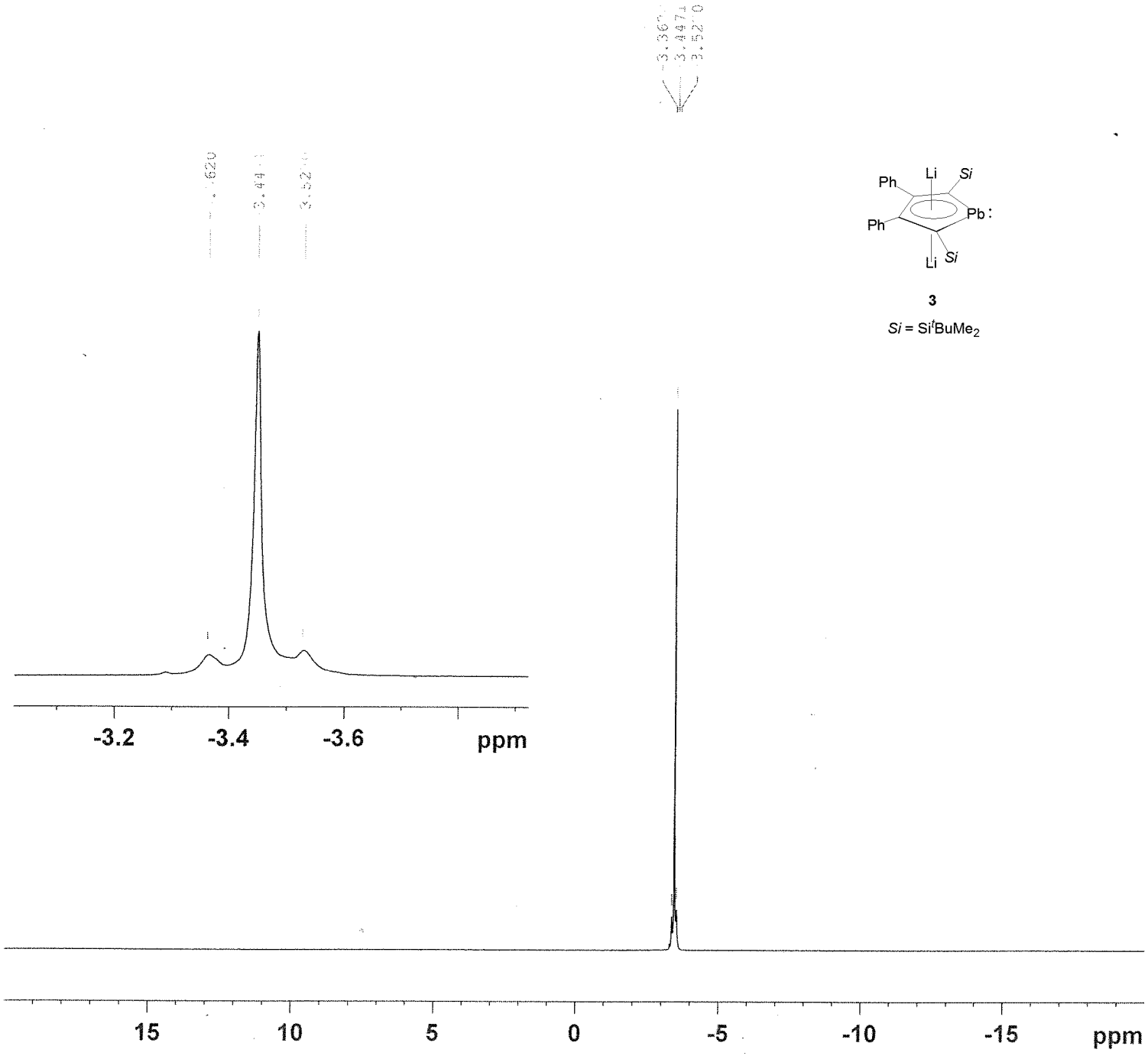
TBDMS Ph dilithioplumbole 7Li in C6D6



3

Si = Si^{t}BuMe$_2$

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PROCNO	1
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PULPROG	zgpg30
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SOLVENT	C6D6
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SWH	19531.250 Hz
FIDRES	0.074506 Hz
AQ	6.7109365 sec
RG	203
DW	25.600 usec
DE	6.50 usec
TE	301.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
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NUC1	7Li
P1	20.00 usec
PL1	1.00 dB
SFO1	194.3695082 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.75 dB
PL12	19.50 dB
PL13	19.50 dB
PL2W	18.63472366 W
PL12W	0.31284049 W
PL13W	0.31284049 W
SFO2	500.1330885 MHz
SI	131072
SF	194.3695384 MHz
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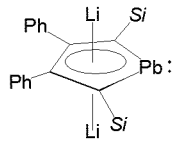
TBDMS Ph dilithioplumbole 7Li in THF-d8 at RT



3

Si = Si^{t}BuMe$_2$

NAME	A13MC116Li	
EXPNO	1	
PROCNO	1	
Date_	20141224	
Time	17.04	
INSTRUM	spect	
PROBHD	5 mm PABBO BB-	
PULPROG	zg30	
TD	65536	
SOLVENT	THF	
NS	16	
DS	4	
SWH	96153.844	Hz
FIDRES	1.467191	Hz
AQ	0.3408372	sec
RG	203	
DW	5.200	usec
DE	6.50	usec
TE	299.9	K
D1	2.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	7Li	
P1	15.00	usec
PL1	3.00	dB
PL1W	37.76776123	W
SFO1	194.3299925	MHz
SI	32768	
SF	194.3306440	MHz
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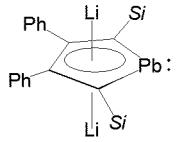
PC	1.40	

TBDMS Ph dilithioplumbole 7Li in THF-d8 at 203K



3

Si = Si^{t}BuMe$_2$

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EXPNO	10	
PROCNO	1	
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PROBHD	5 mm PABBO BB-	
PULPROG	zg30	
TD	65536	
SOLVENT	THF	
NS	32	
DS	4	
SWH	96153.844	Hz
FIDRES	1.467191	Hz
AQ	0.3408372	sec
RG	203	
DW	5.200	usec
DE	6.50	usec
TE	206.8	K
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TD0	1	
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P1	15.00	usec
PL1	3.00	dB
PL1W	37.76776123	W
SFO1	194.3299925	MHz
SI	32768	
SF	194.3306440	MHz
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GB	0	
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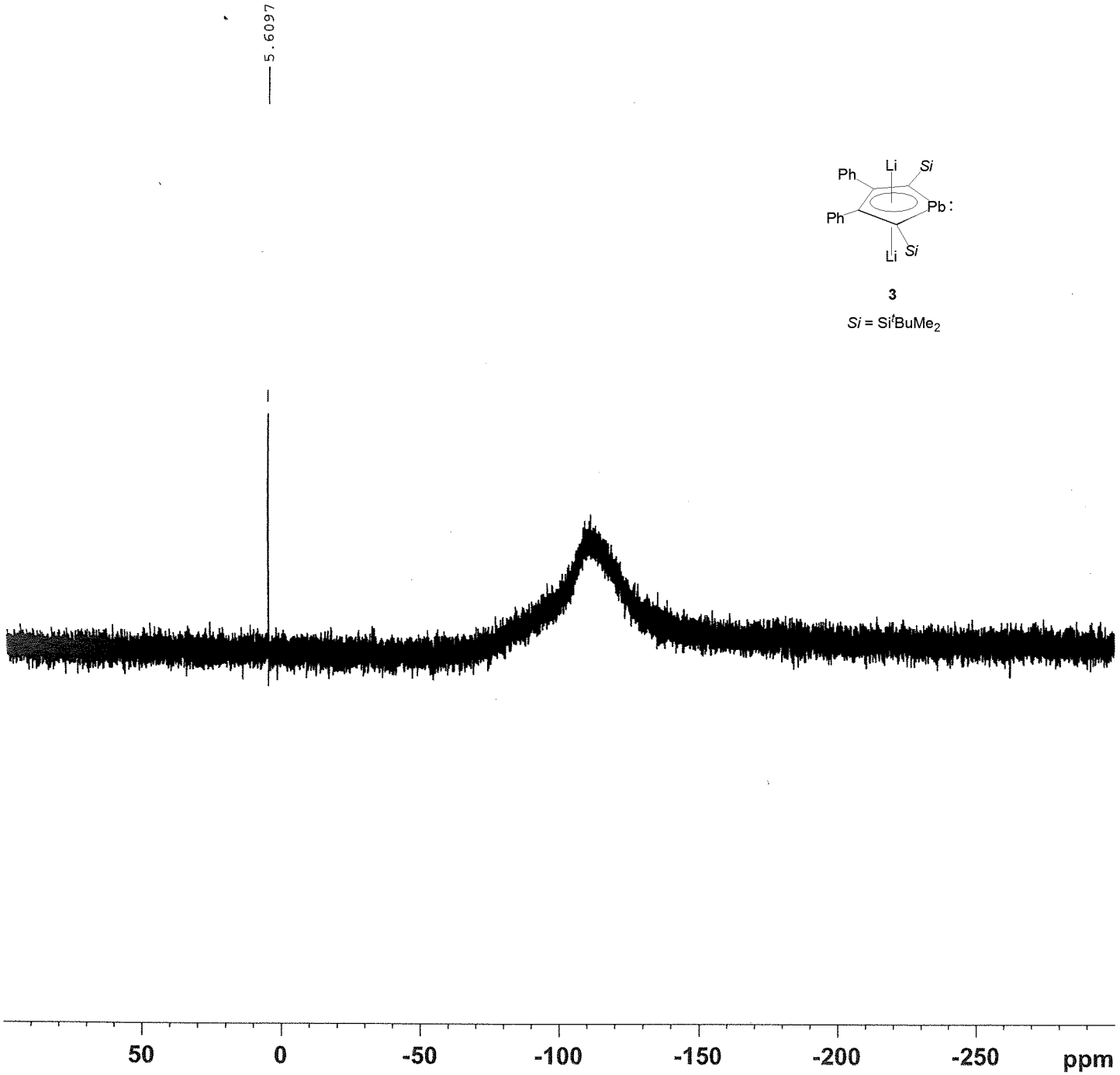
TBDMS Ph dilithioplumbole 29Si in C6D6



3

Si = Si^{t}BuMe$_2$

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PROCNO	1	
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Time	15.06	
INSTRUM	spect	
PROBHD	5 mm PABBO BB-	
PULPROG	zgig	
TD	65536	
SOLVENT	C6D6	
NS	440	
DS	4	
SWH	39682.539	Hz
FIDRES	0.605507	Hz
AQ	0.8258036	sec
RG	203	
DW	12.600	usec
DE	6.50	usec
TE	300.4	K
D1	5.00000000	sec
D11	0.03000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	29Si	
P1	15.00	usec
PL1	3.00	dB
PL1W	37.76776123	W
SFO1	99.3319610	MHz
======== CHANNEL f2 ========		
CPDPRG2	waltz16	
NUC2	1H	
PCPD2	80.00	usec
PL2	2.40	dB
PL12	19.02	dB
PL2W	15.17711735	W
PL12W	0.33051354	W
SFO2	500.0320001	MHz
SI	32768	
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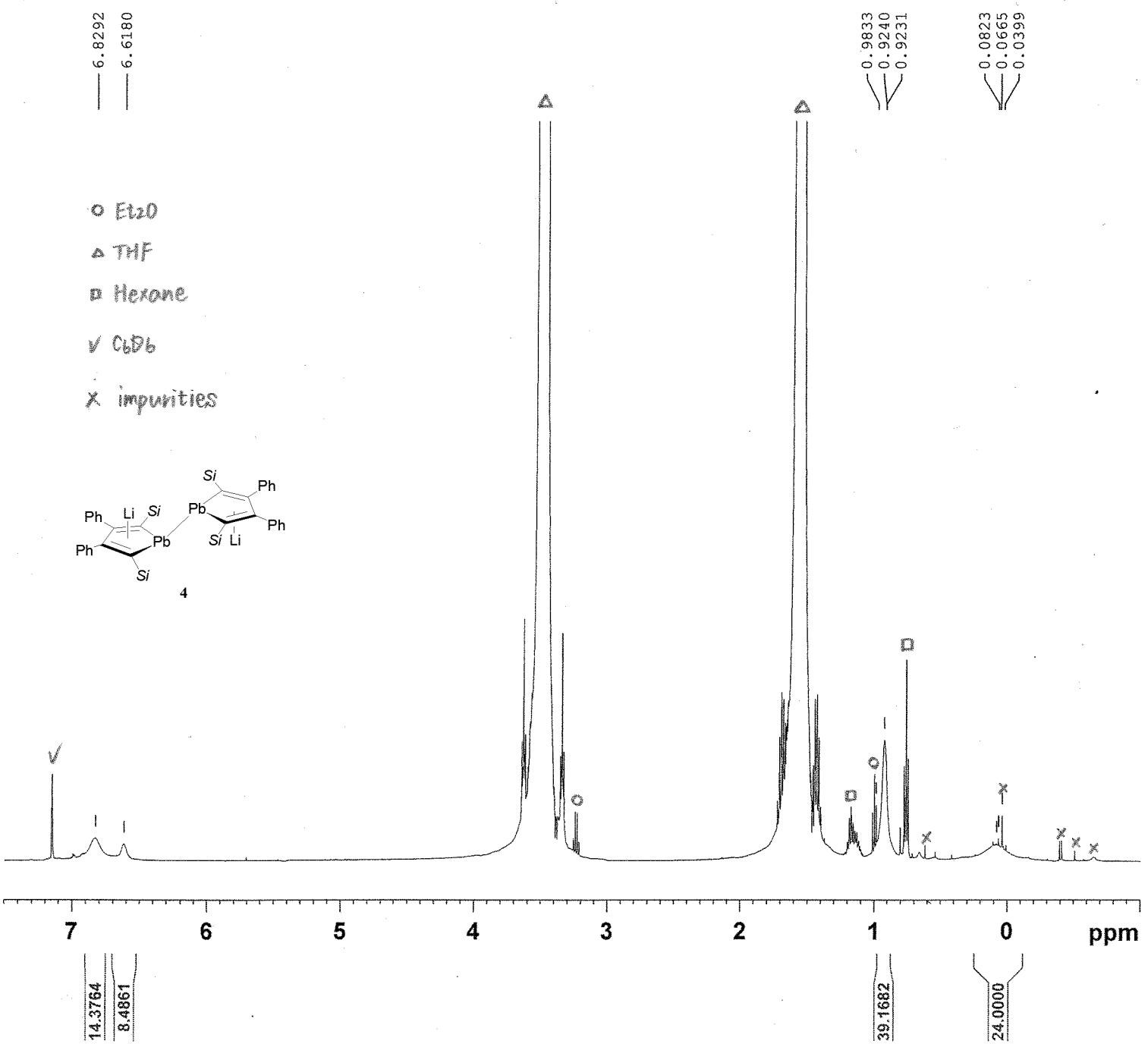
Wx 319 1,1' dilithiobiplumbole comp. 1H in THF a C6D6



Current Data Parameters

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EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

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PULPROG	zg30
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SOLVENT	C6D6
NS	8
DS	2
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FIDRES	0.157632 Hz
AQ	3.1719923 sec
RG	2.8
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DE	6.50 usec
TE	299.3 K
D1	1.00000000 sec
TD0	1

======== CHANNEL f1 ========

NUC1	1H
P1	12.00 usec
PL1	2.60 dB
PL1W	15.32226563 W
SFO1	500.1330885 MHz

F2 - Processing parameters

SI	32768
SF	500.1300011 MHz
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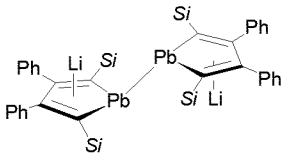

4

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PROCNO	1	
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PULPROG	zgpg	
TD	65536	
SOLVENT	C6D6	
NS	2040	
DS	2	
SWH	208333.328	Hz
FIDRES	3.178914	Hz
AQ	0.1573364	sec
RG	203	
DW	2.400	usec
DE	65.00	usec
TE	300.7	K
D1	2.00000000	sec
D11	0.03000000	sec
TD0	1	
========	CHANNEL f1	========
NUC1	207Pb	
P1	12.00	usec
PL1	0.00	dB
SFO1	104.7138803	MHz
========	CHANNEL f2	========
CPDPRG2	waltz16	
NUC2	1H	
PCPD2	80.00	usec
PL2	2.40	dB
PL12	19.02	dB
PL13	22.02	dB
PL2W	15.17711735	W
PL12W	0.33051354	W
PL13W	0.16564916	W
SFO2	500.0320005	MHz
SI	65536	
SF	104.6094793	MHz
WDW	EM	
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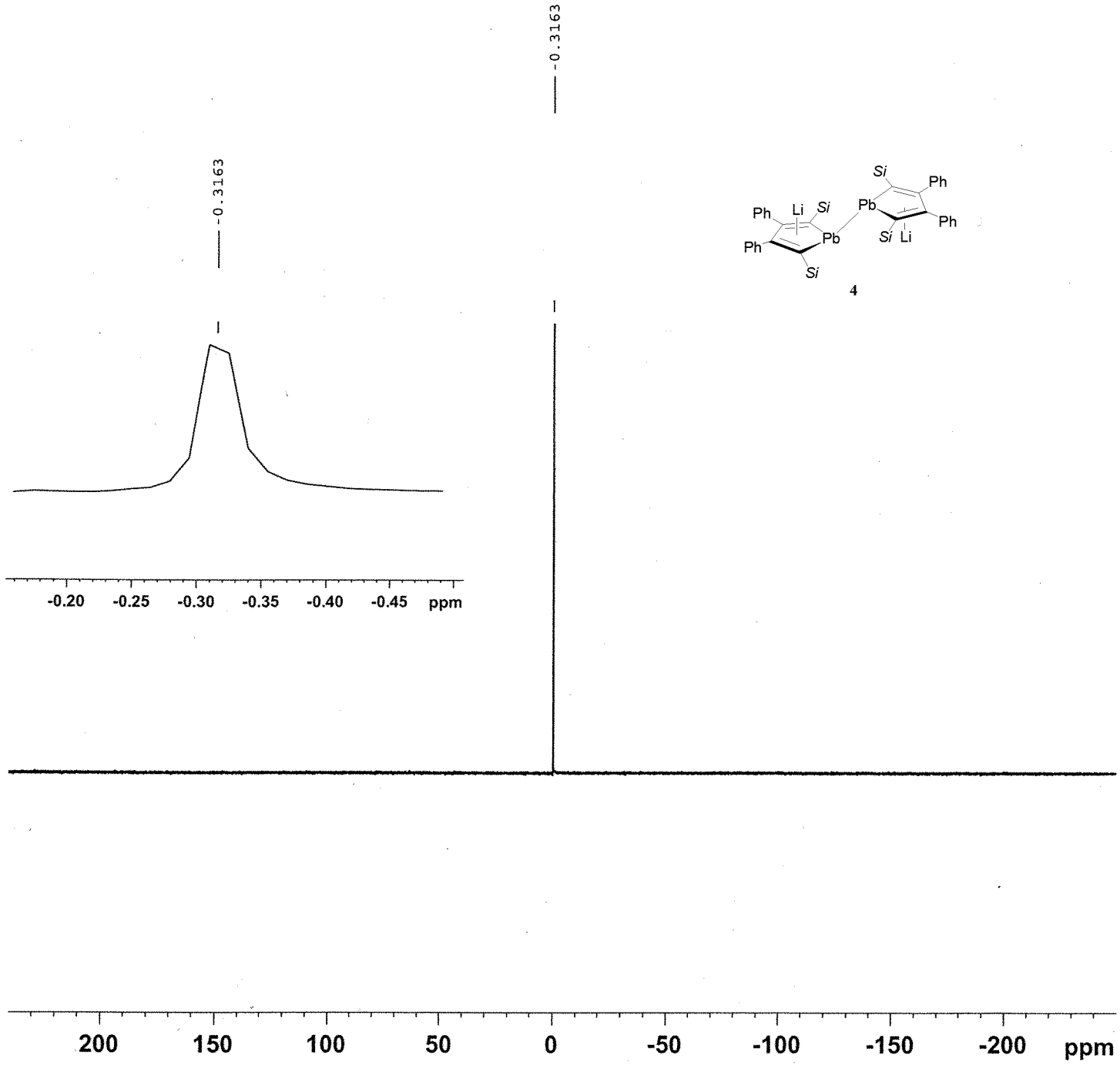
PC	1.40	

Ex 319 1,1` dilithiobiplumbole comp. 7Li in THF and D6



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PROCNO	1	
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PULPROG	zg30	
TD	65536	
SOLVENT	C6D6	
NS	8	
DS	4	
SWH	96153.844	Hz
FIDRES	1.467191	Hz
AQ	0.3408372	sec
RG	203	
DW	5.200	usec
DE	6.50	usec
TE	299.8	K
D1	2.00000000	sec
TD0	1	
========	CHANNEL f1 ========	
NUC1	7Li	
P1	15.00	usec
PL1	3.00	dB
PL1W	37.76776123	W
SFO1	194.3299925	MHz
SI	32768	
SF	194.3306440	MHz
WDW	EM	
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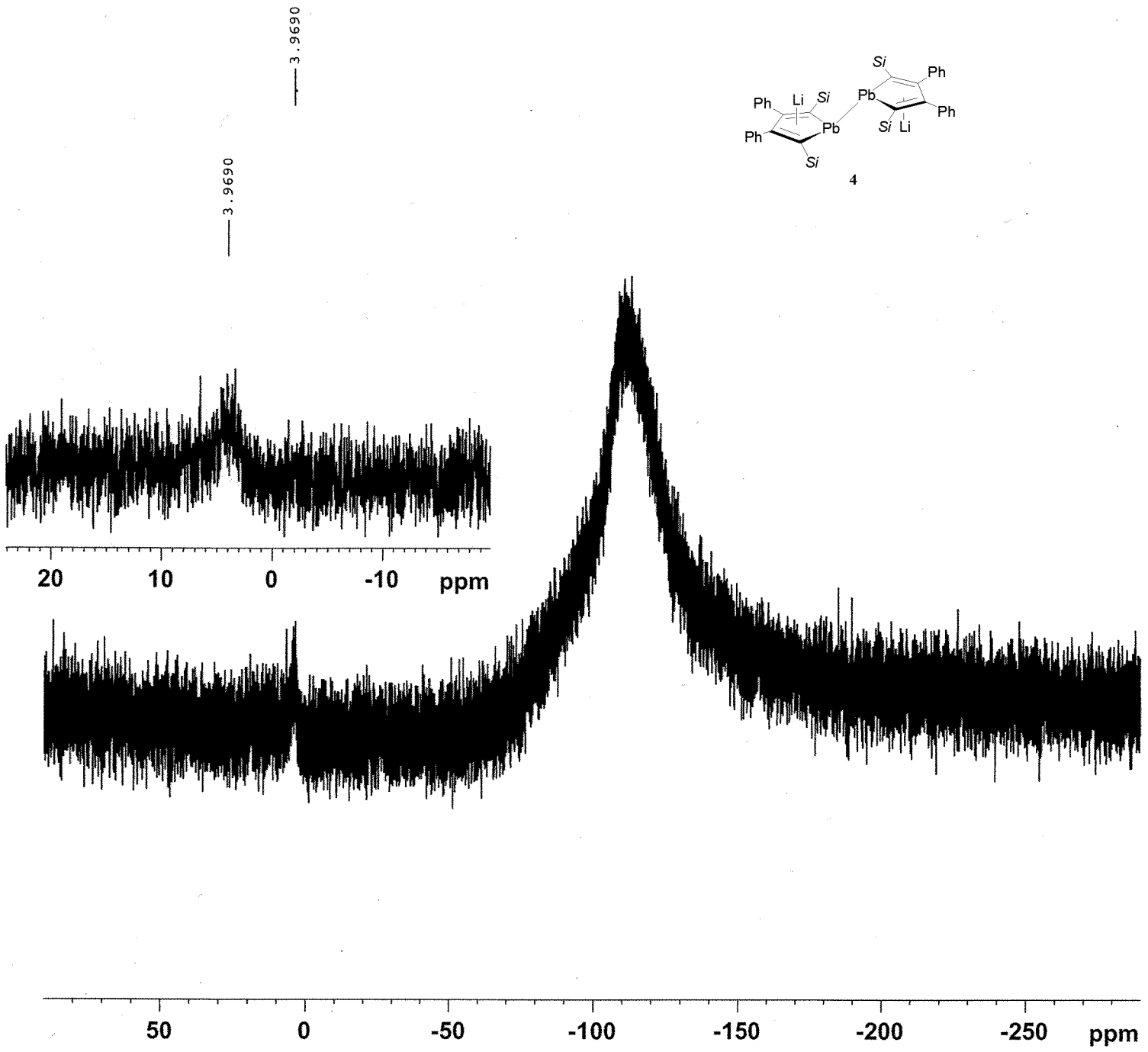
PC	1.40	

Ex 319 1,1` dilithiobiplumbole comp. 29 Si in THF an` C6D6



4

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PROCNO	1	
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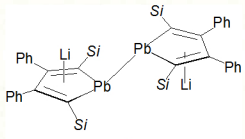
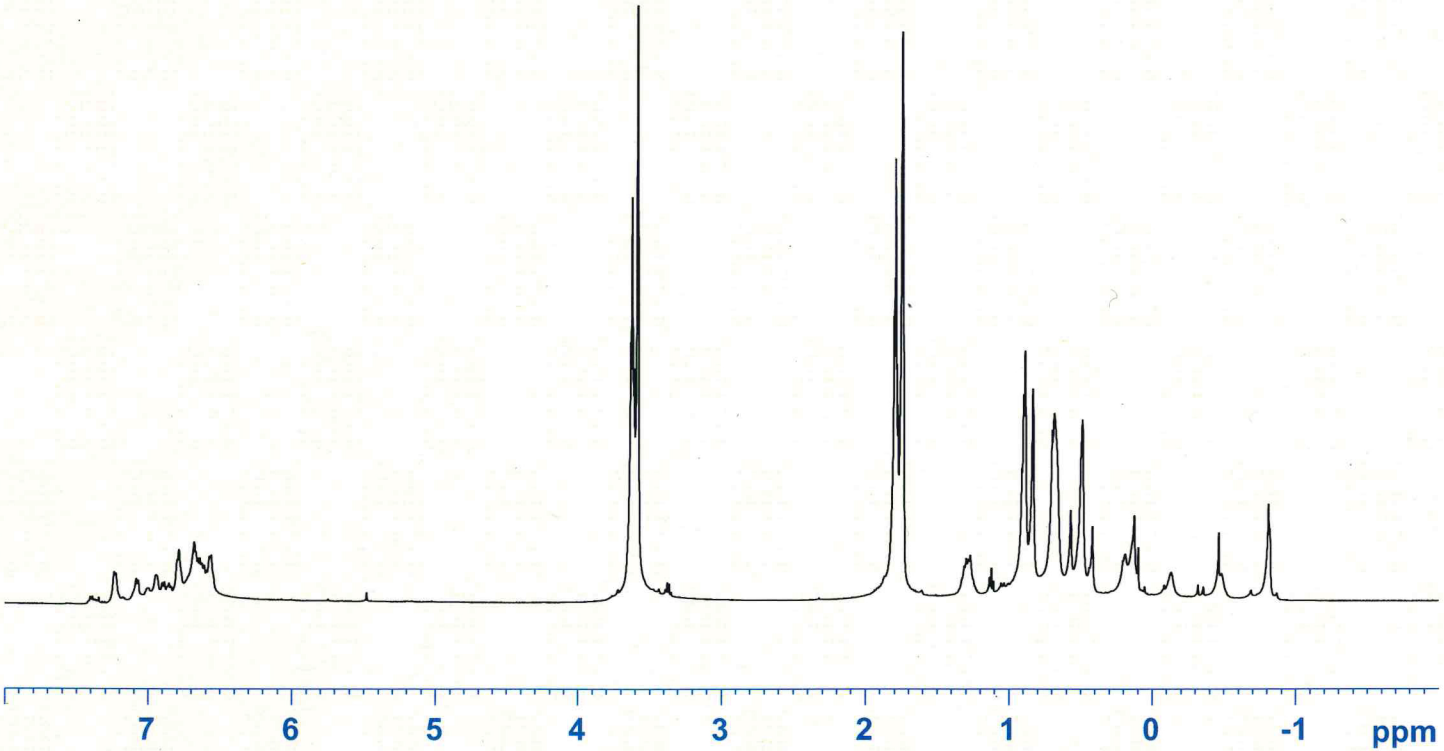
PULPROG	zgig	
TD	65536	
SOLVENT	C6D6	
NS	1004	
DS	4	
SWH	39682.539	Hz
FIDRES	0.605507	Hz
AQ	0.8258036	sec
RG	203	
DW	12.600	usec
DE	6.50	usec
TE	300.0	K
D1	5.00000000	sec
D11	0.03000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	29Si	
P1	15.00	usec
PL1	3.00	dB
PL1W	37.76776123	W
SFO1	99.3319610	MHz
======== CHANNEL f2 ========		
CPDPRG2	waltz16	
NUC2	1H	
PCPD2	80.00	usec
PL2	2.40	dB
PL12	19.02	dB
PL2W	15.17711735	W
PL12W	0.33051354	W
SFO2	500.0320001	MHz
SI	32768	
SF	99.3418950	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	

1,1'-dilithiobiplumbole 1H in THF-d8 (cal.@1.79) ~~RT~~ 223



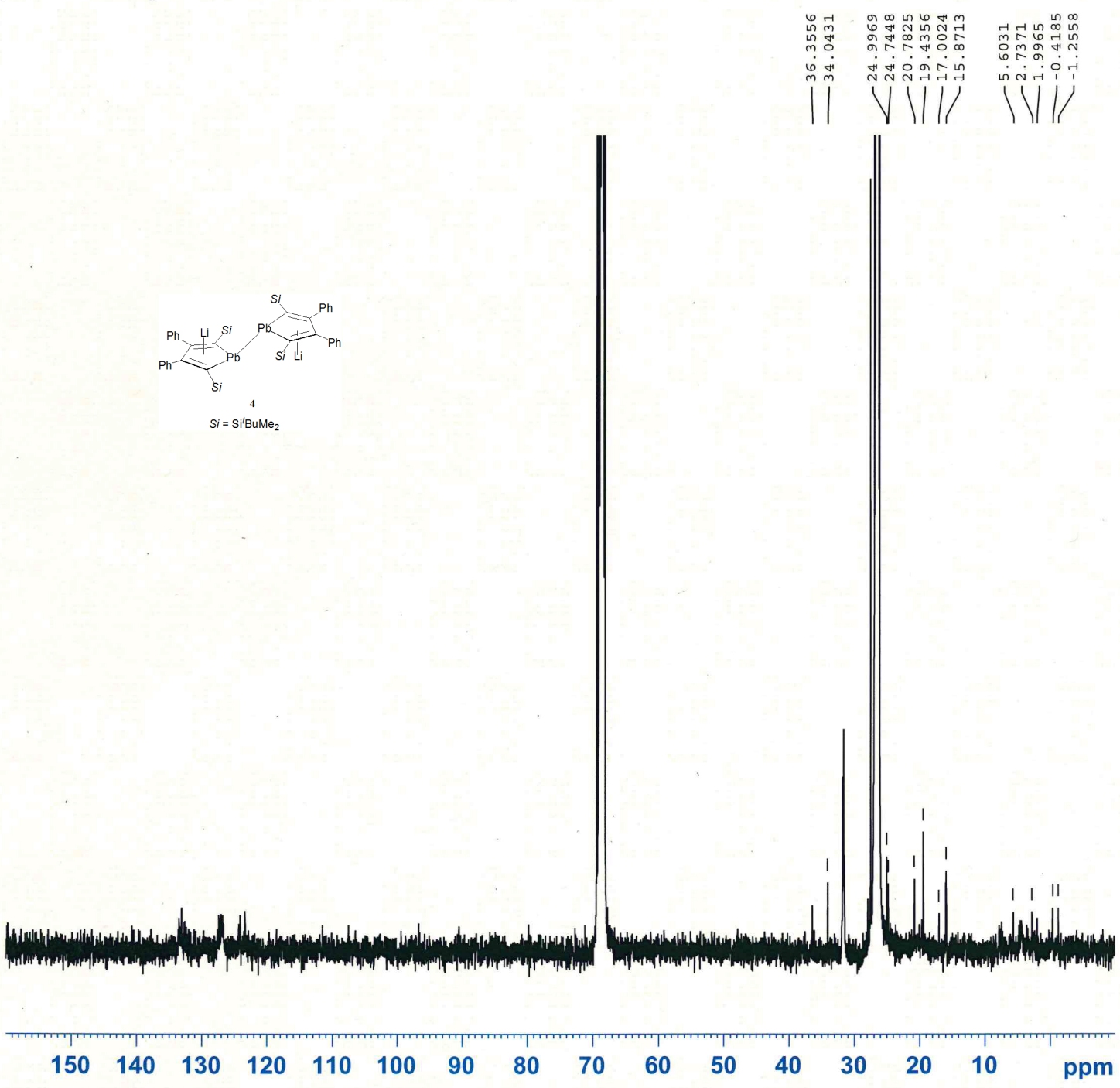
4

Si = Si^{t}BuMe$_2$



NAME	A13MC116VTH223K	
EXPNO	455	
PROCNO	1	
Date_	20150109	
Time	15.26	
INSTRUM	spect	
PROBHD	5 mm PABBO BB-	
PULPROG	zg30	
TD	65536	
SOLVENT	THF	
NS	8	
DS	2	
SWH	10330.578	Hz
FIDRES	0.157632	Hz
AQ	3.1719923	sec
RG	90.5	
DW	48.400	usec
DE	6.50	usec
TE	222.3	K
D1	1.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	1H	
P1	11.80	usec
PL1	2.40	dB
PL1W	15.17711735	W
SFO1	500.0330885	MHz
SI	32768	
SF	500.0290845	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.40	

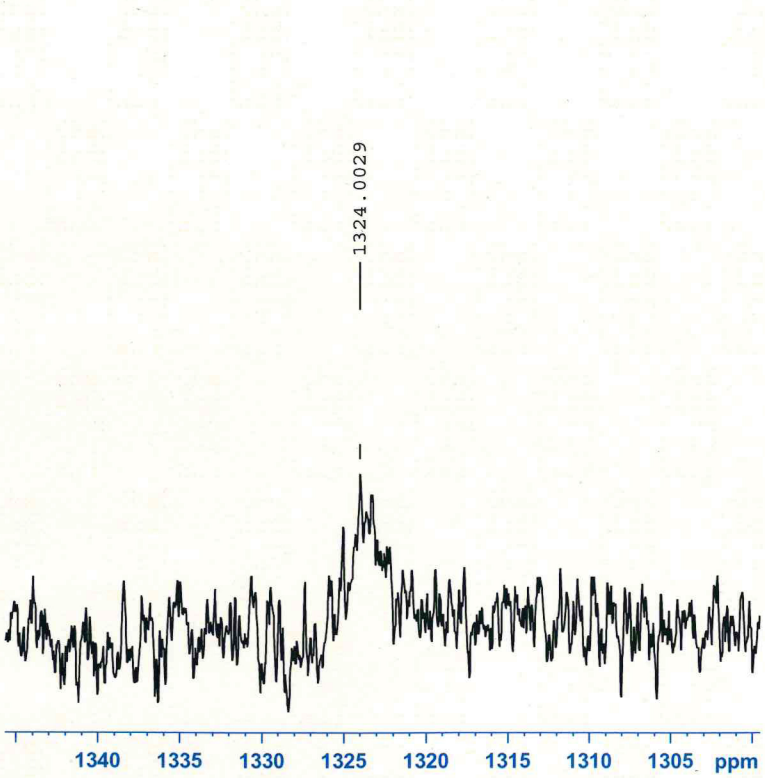
1,1'-dilithiobiplumbole 13C in THF-d8 (cal@26.15, 223K)

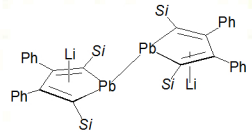


BRUKER

NAME	A13MC116VTC223K
EXPNO	455
PROCNO	1
Date_	20150109
Time	15.36
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	THF
NS	1432
DS	2
SWH	40760.871 Hz
FIDRES	0.621962 Hz
AQ	0.8039582 sec
RG	203
DW	12.267 usec
DE	6.50 usec
TE	222.9 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.80 usec
PL1	0.00 dB
PL1W	100.47545624 W
SFO1	125.7477319 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.40 dB
PL12	19.02 dB
PL13	22.02 dB
PL2W	15.17711735 W
PL12W	0.33051354 W
PL13W	0.16564916 W
SFO2	500.0316016 MHz
SI	16384
SF	125.7321636 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1,1'-dilithiobiplumbole 207Pb in THF-d8 223K



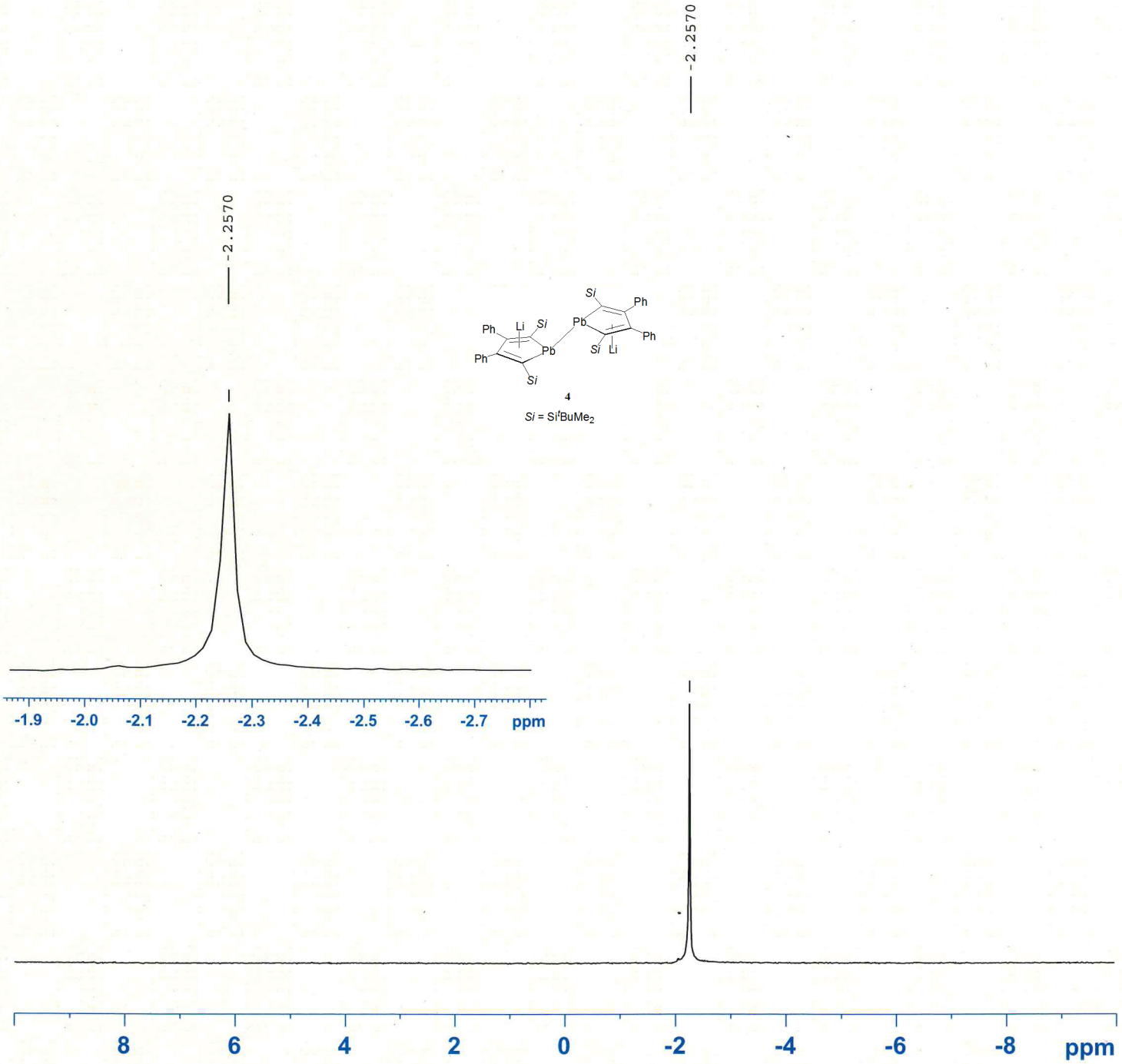


4

Si = Si^{t}BuMe$_2$

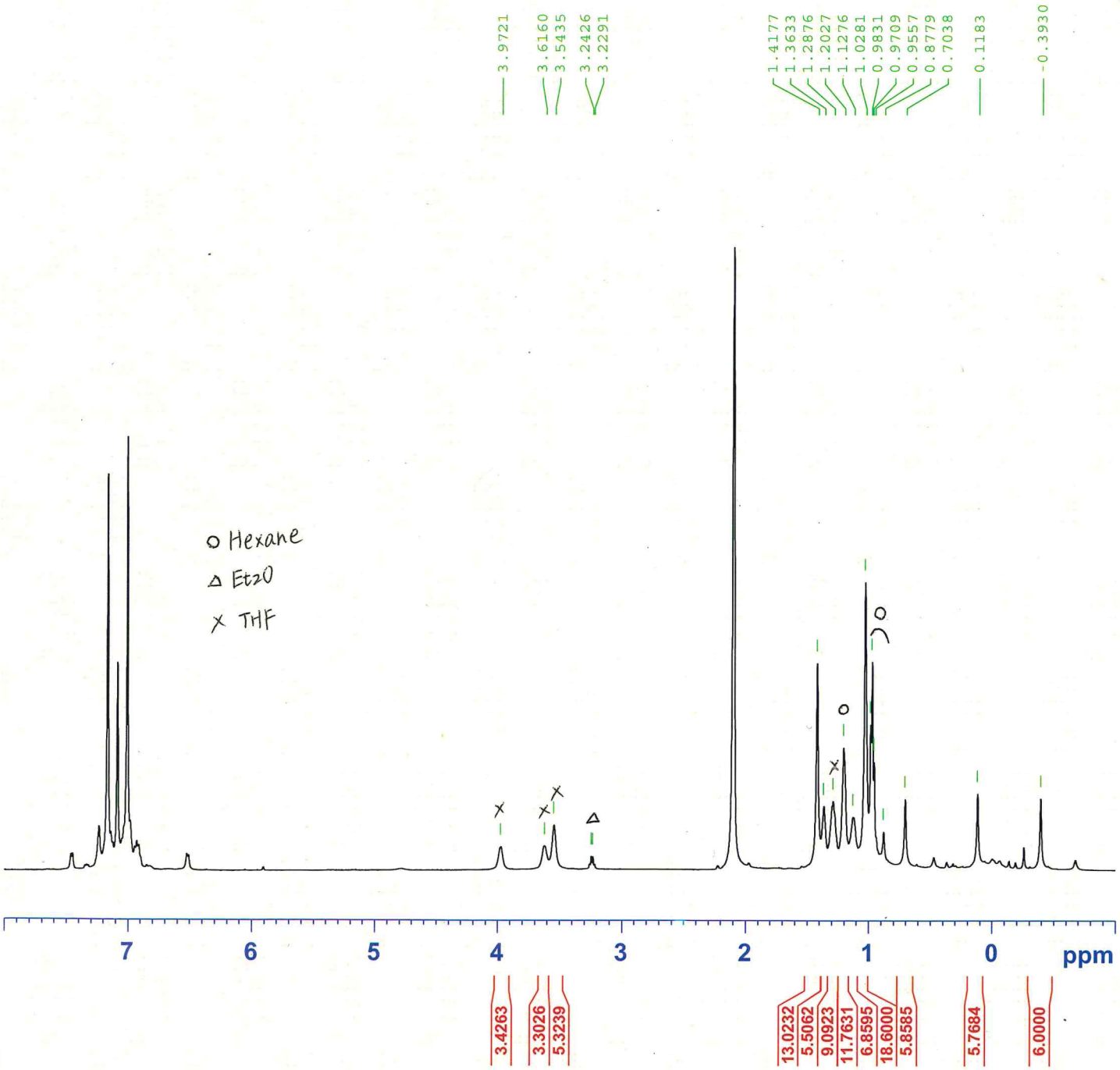
NAME	A13MC116VTPb223K	
EXPNO	455	
PROCNO	1	
Date_	20150109	
Time	16.44	
INSTRUM	spect	
PROBHD	5 mm PABBO BB-	
PULPROG	zgpg	
TD	65536	
SOLVENT	THF	
NS	936	
DS	2	
SWH	208333.328	Hz
FIDRES	3.178914	Hz
AQ	0.1573364	sec
RG	203	
DW	2.400	usec
DE	65.00	usec
TE	219.1	K
D1	2.00000000	sec
D11	0.03000000	sec
TD0	1	
========	CHANNEL f1	========
NUC1	207Pb	
P1	12.00	usec
PL1	0.00	dB
SFO1	104.7661849	MHz
========	CHANNEL f2	========
CPDPRG2	waltz16	
NUC2	1H	
PCPD2	80.00	usec
PL2	2.40	dB
PL12	19.02	dB
PL13	22.02	dB
PL2W	15.17711735	W
PL12W	0.33051354	W
PL13W	0.16564916	W
SFO2	500.0320005	MHz
SI	65536	
SF	104.6094793	MHz
WDW	EM	
SSB	0	
LB	10.00	Hz
GB	0	
PC	1.40	

1,1'-dilithiobiplumbole 7Li 223K in THF-d8



NAME	A13MC116VTLi223K	
EXPNO	455	
PROCNO	1	
Date_	20150109	
Time	15.30	
INSTRUM	spect	
PROBHD	5 mm PABBO BB-	
PULPROG	zg30	
TD	65536	
SOLVENT	THF	
NS	32	
DS	4	
SWH	96153.844	Hz
FIDRES	1.467191	Hz
AQ	0.3408372	sec
RG	203	
DW	5.200	usec
DE	6.50	usec
TE	222.7	K
D1	2.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	7Li	
P1	15.00	usec
PL1	3.00	dB
PL1W	37.76776123	W
SFO1	194.3299925	MHz
SI	32768	
SF	194.3306440	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	

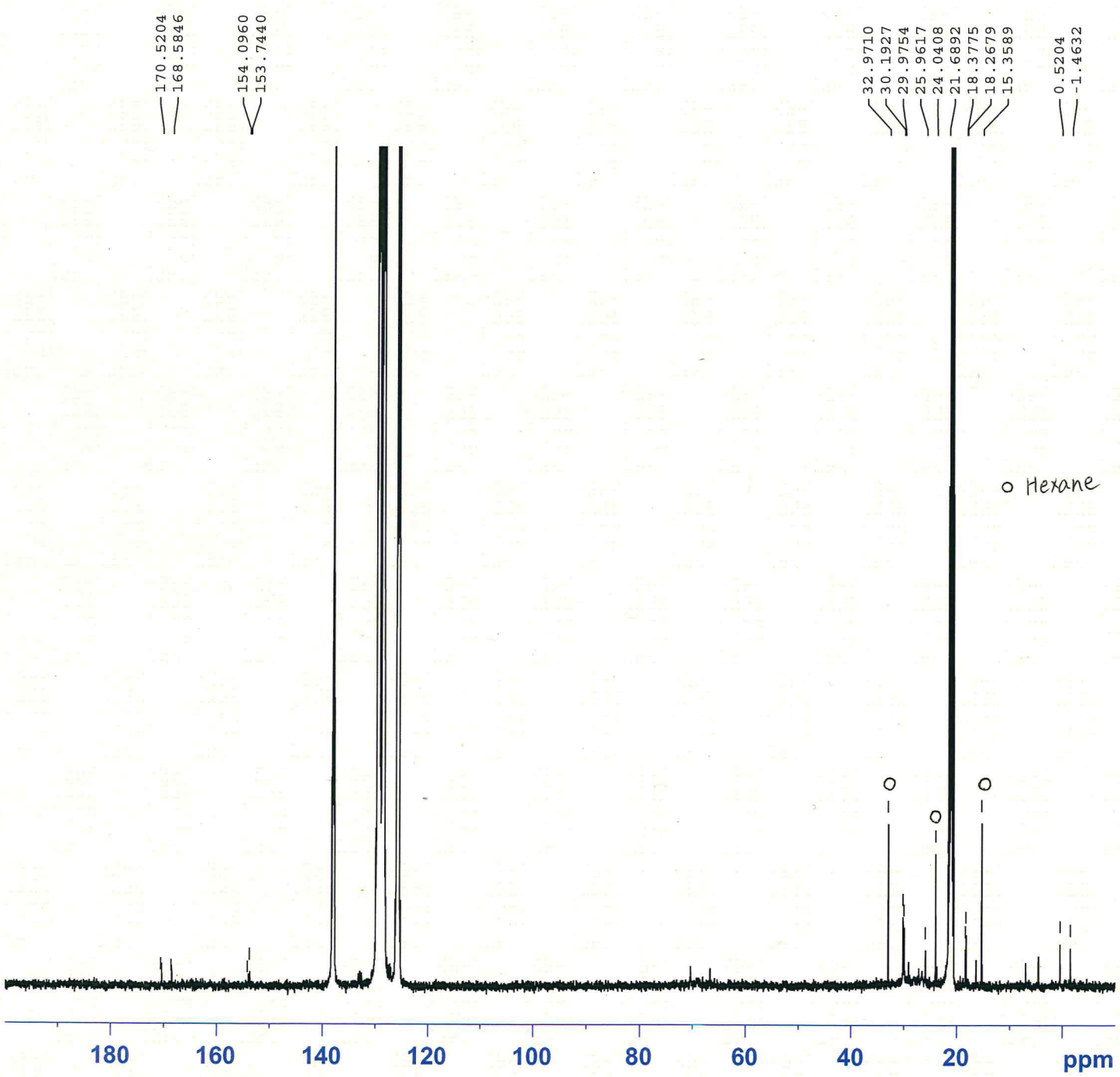
1,1'-dilithiobiplumbole 1H in tol-d8 at. 223K (cal@2.1)



BRUKER

NAME	SAITMN-DilithiobiVTH
EXPNO	1
PROCNO	1
Date_	20141104
Time	13.32
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	Tol
NS	8
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1719923 sec
RG	101
DW	48.400 usec
DE	6.50 usec
TE	222.9 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	11.80 usec
PL1	2.40 dB
PL1W	15.17711735 W
SFO1	500.0330885 MHz
SI	32768
SF	500.0300145 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

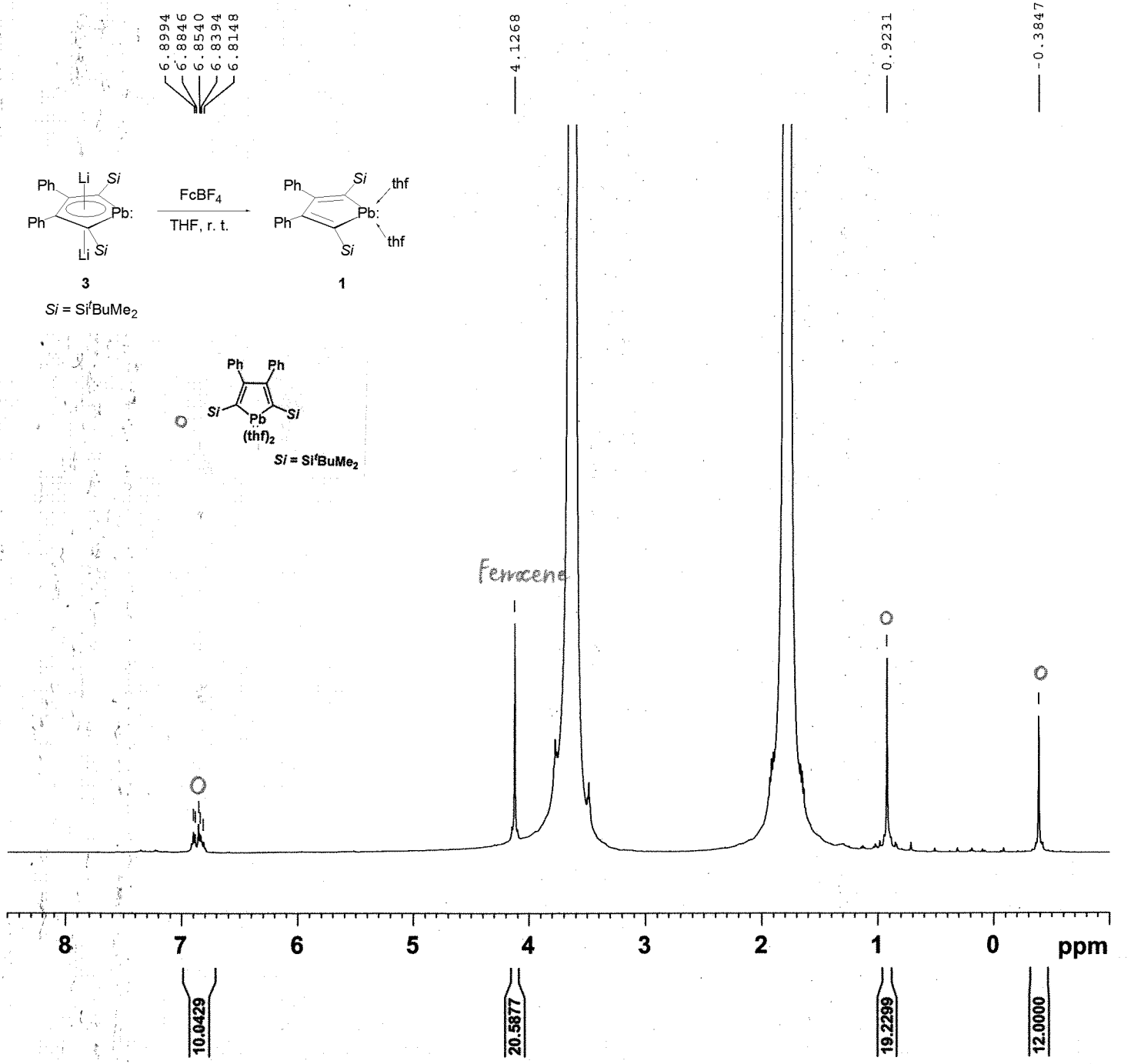
1,1'-dilithiobiplumbole 13C in tol-d8 at.223K (cal@21.0)



NAME	SAITMN-DilithiobiVTC	
EXPNO	1	
PROCNO	1	
Date_	20141104	
Time	13.35	
INSTRUM	spect	
PROBHD	5 mm PABBO BB-	
PULPROG	zgpg30	
TD	65536	
SOLVENT	Tol	
NS	3891	
DS	2	
SWH	40760.871	Hz
FIDRES	0.621962	Hz
AQ	0.8039582	sec
RG	203	
DW	12.267	usec
DE	6.50	usec
TE	223.0	K
D1	2.00000000	sec
D11	0.03000000	sec
TD0	1	
========	CHANNEL f1 ========	
NUC1	13C	
P1	9.80	usec
PL1	0.00	dB
PL1W	100.47545624	W
SFO1	125.7477319	MHz
========	CHANNEL f2 ========	
CPDPRG2	waltz16	
NUC2	1H	
PCPD2	80.00	usec
PL2	2.40	dB
PL12	19.02	dB
PL13	22.02	dB
PL2W	15.17711735	W
PL12W	0.33051354	W
PL13W	0.16564916	W
SFO2	500.0316016	MHz
SI	16384	
SF	125.7325542	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	

6

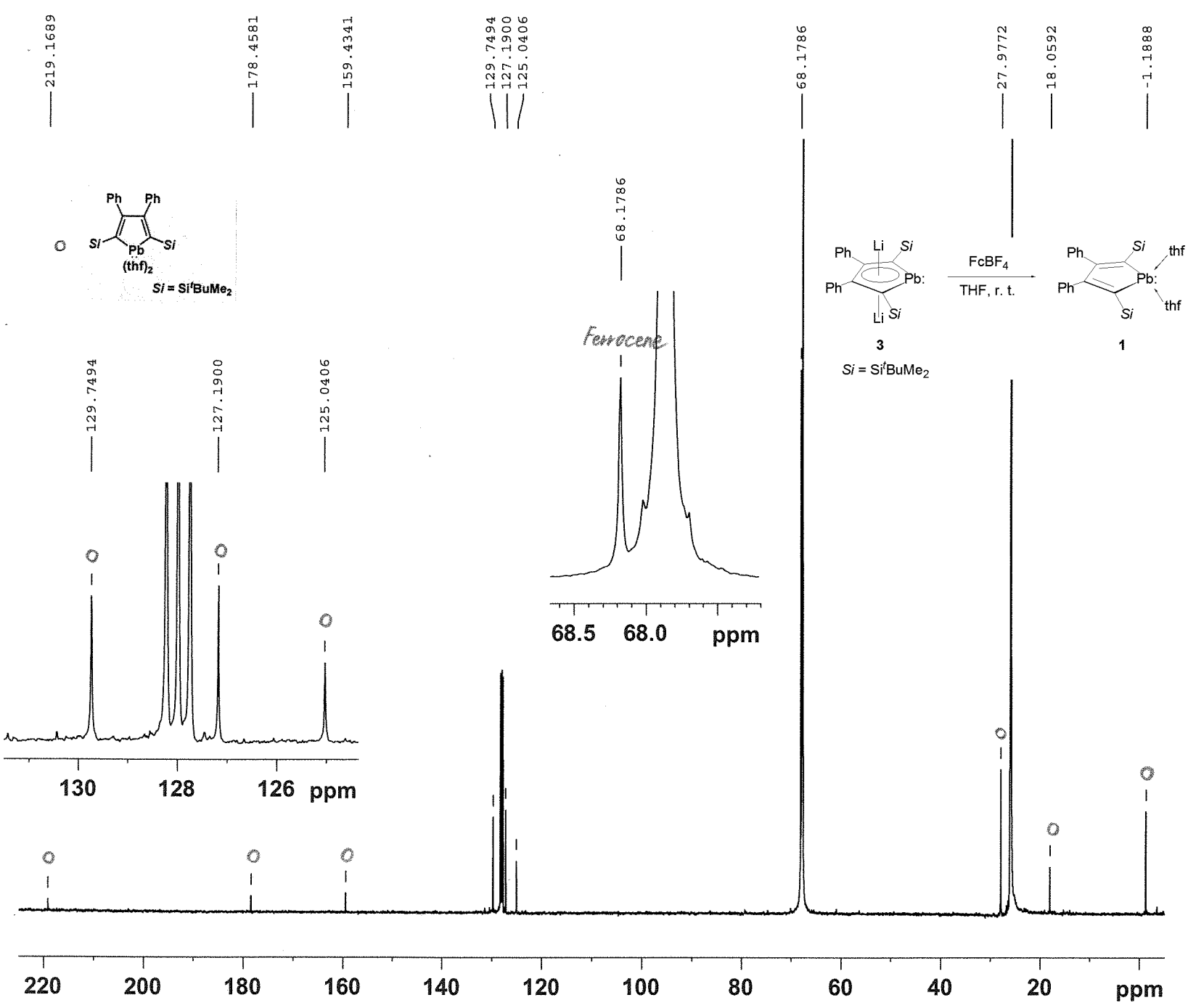
Ex 225 1H in THF



BRUKER

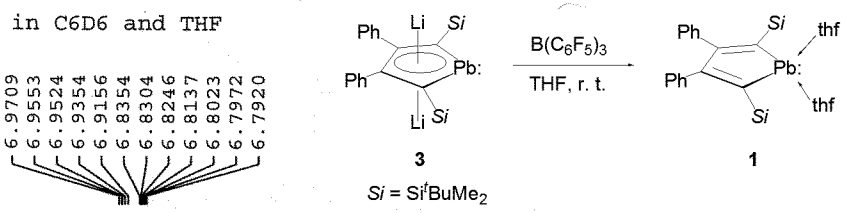
NAME	A13MC116EF
EXPNO	1
PROCNO	1
Date_	20130912
Time	11.47
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	C6D6
NS	8
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1719923 sec
RG	3.2
DW	48.400 usec
DE	6.50 usec
TE	300.4 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	11.80 usec
PL1	2.40 dB
PL1W	15.17711735 W
SFO1	500.0330885 MHz
SI	32768
SF	500.0301467 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

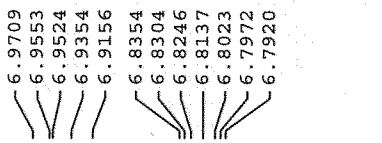
Ex 226 13C in THF

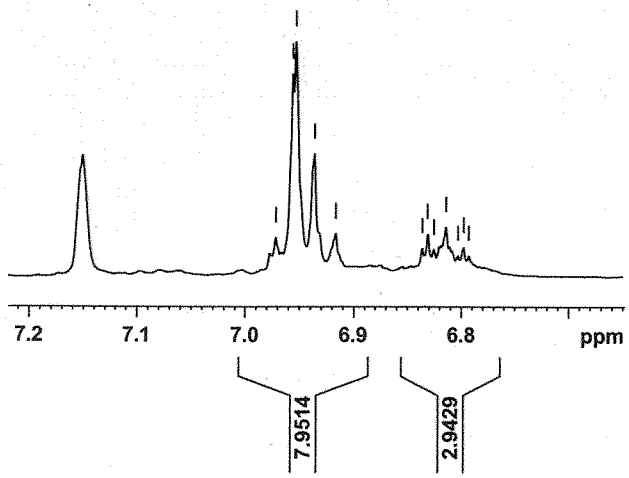


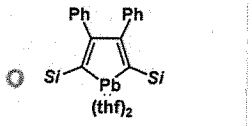
NAME	A12MC108of
EXPNO	1
PROCNO	1
Date_	20130912
Time	12.20
INSTRUM	spect
PROBHD	5 mm CPQNP 1H/
PULPROG	zgpg30
TD	65536
SOLVENT	C6D6
NS	72
DS	2
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	126.99
DW	16.800 usec
DE	18.00 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.00 usec
SI	32768
SF	100.6127374 MHz
WDW	EM
SSB	0
LB	2.00 Hz
GB	0
PC	1.40

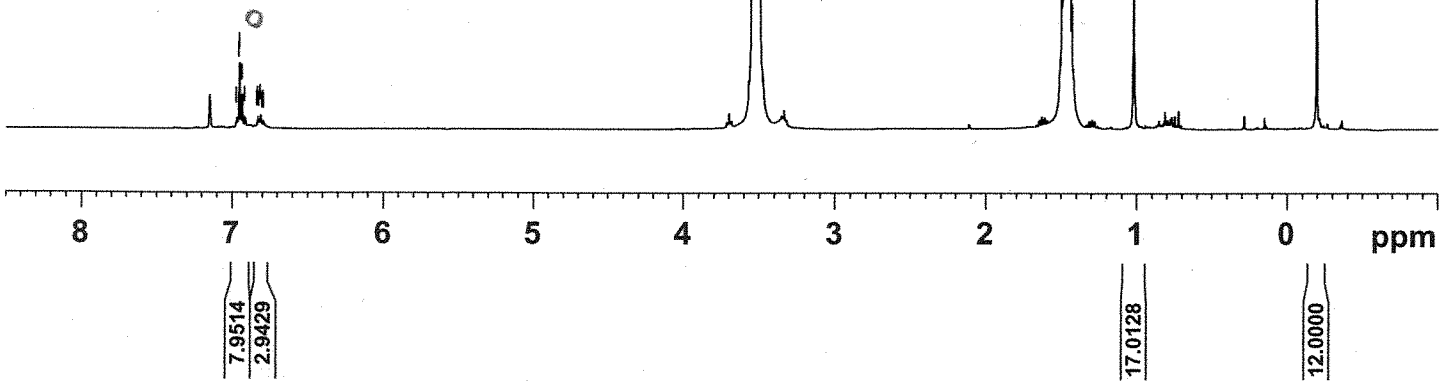
Ex 376 1H in C6D6 and THF

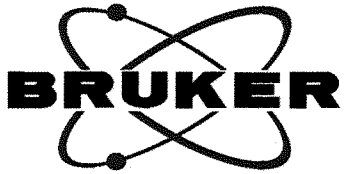












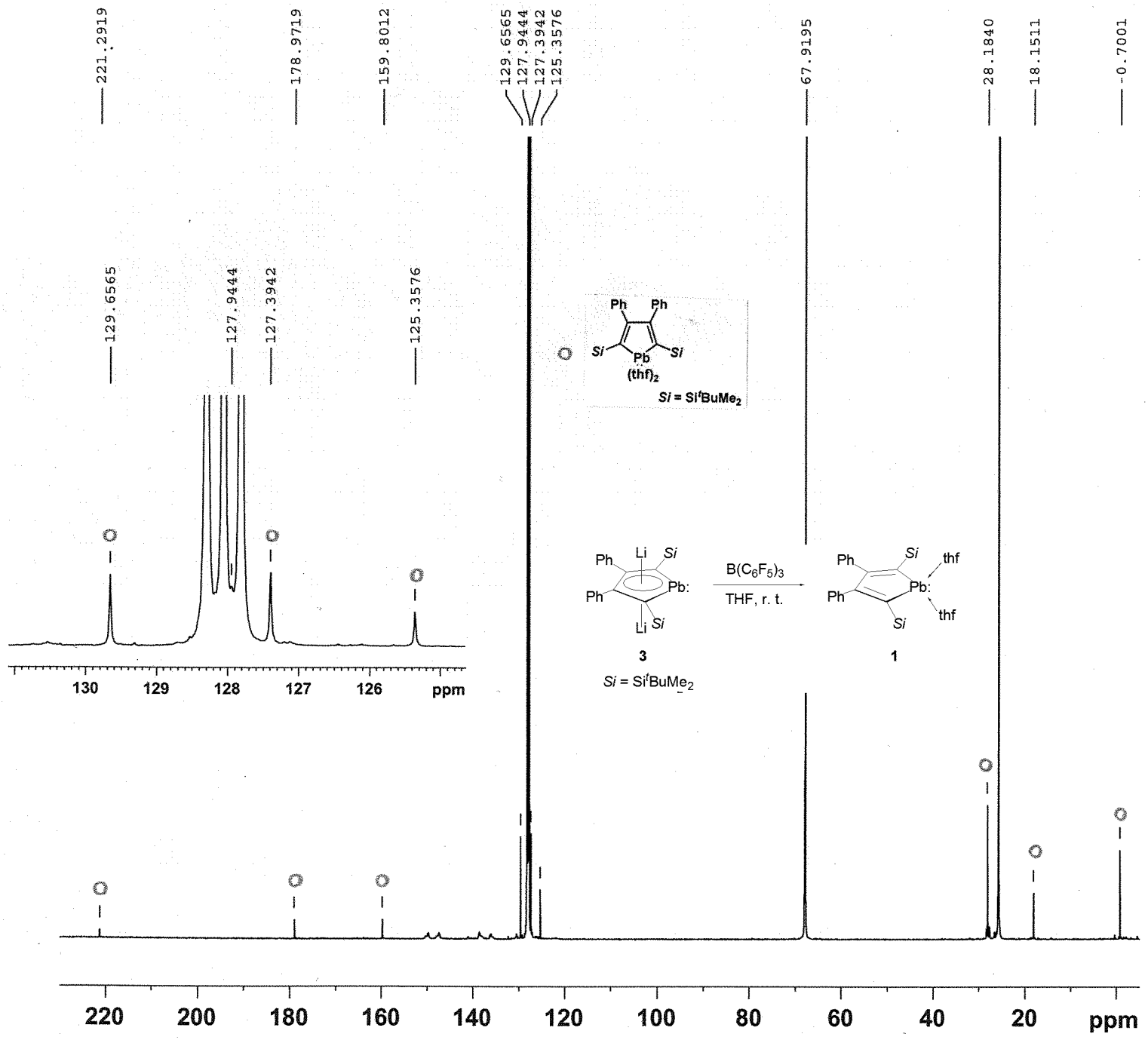

```
Current Data Parameters
NAME                  A13MC116H
EXPNO                       376
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20140611
Time                      12.08
INSTRUM                   spect
PROBHD    5 mm CPQNP 1H/
PULPROG                    zg30
TD                        65536
SOLVENT                     C6D6
NS                             4
DS                             2
SWH                    8305.647 Hz
FIDRES                 0.126734 Hz
AQ                     3.9453173 sec
RG                          4.17
DW                        60.200 usec
DE                        10.00 usec
TE                        300.0 K
D1                     1.00000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                          1H
P1                        15.00 usec
PLW1                   5.19999981 W
SFO1                 400.1324708 MHz

F2 - Processing parameters
SI                            32768
SF                   400.1300006 MHz
WDW                            EM
SSB                0
LB                            0.30 Hz
GB                 0
PC                            1.00
```

Ex 376 13C in C6D6 and THF



BRUKER

Current Data Parameters
NAME A13MC116C
EXPNO 376
PROCNO 1

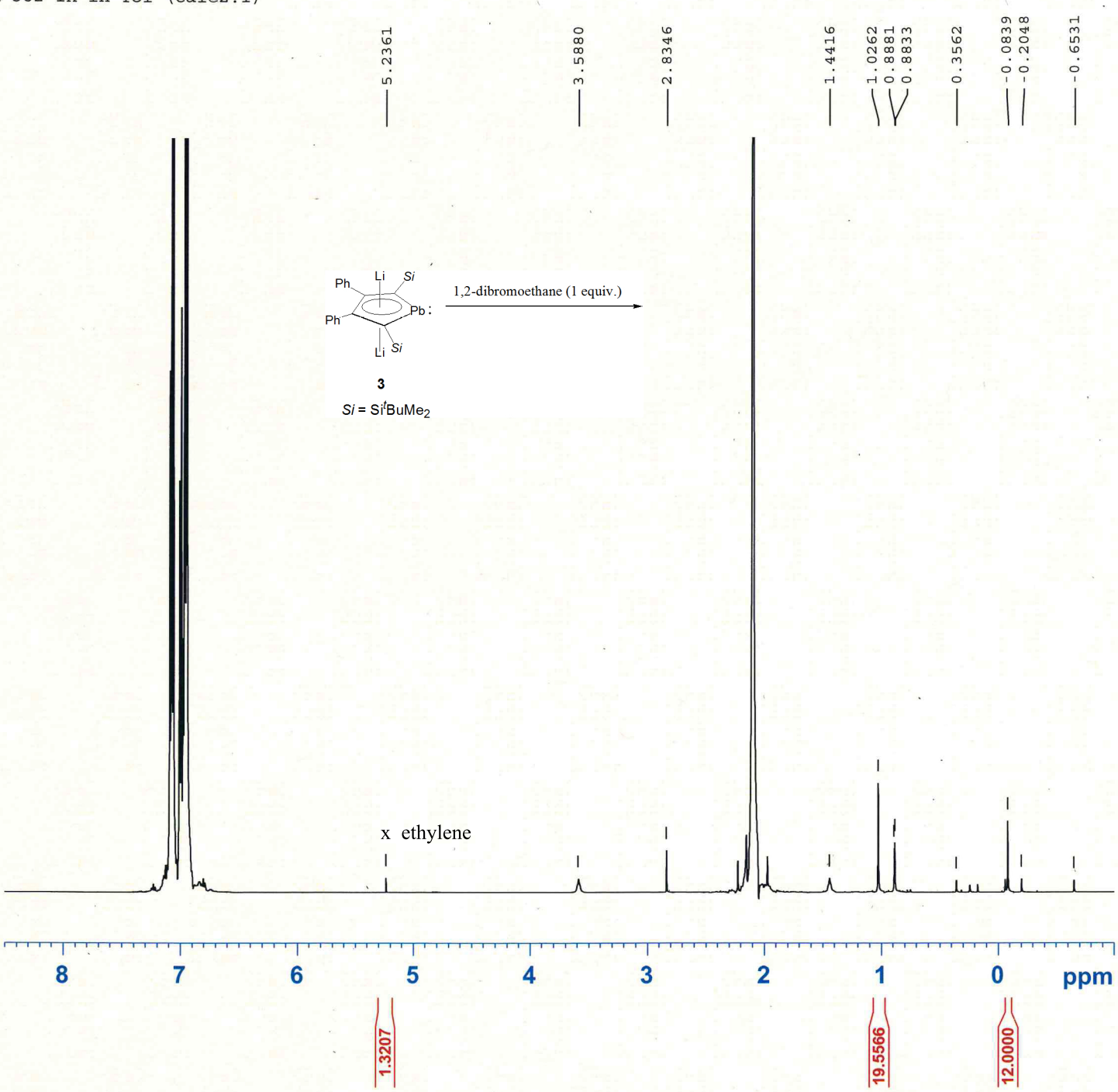
F2 - Acquisition Parameters
Date_ 20140611
Time 20.47
INSTRUM spect
PROBHD 5 mm CPQNP 1H/
PULPROG zgpg30
TD 65536
SOLVENT C6D6
NS 560
DS 2
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 126.99
DW 16.800 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PLW1 15.50000000 W
SFO1 100.6248425 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 5.19999981 W
PLW12 0.14444000 W
PLW13 0.11700000 W
SFO2 400.1316005 MHz

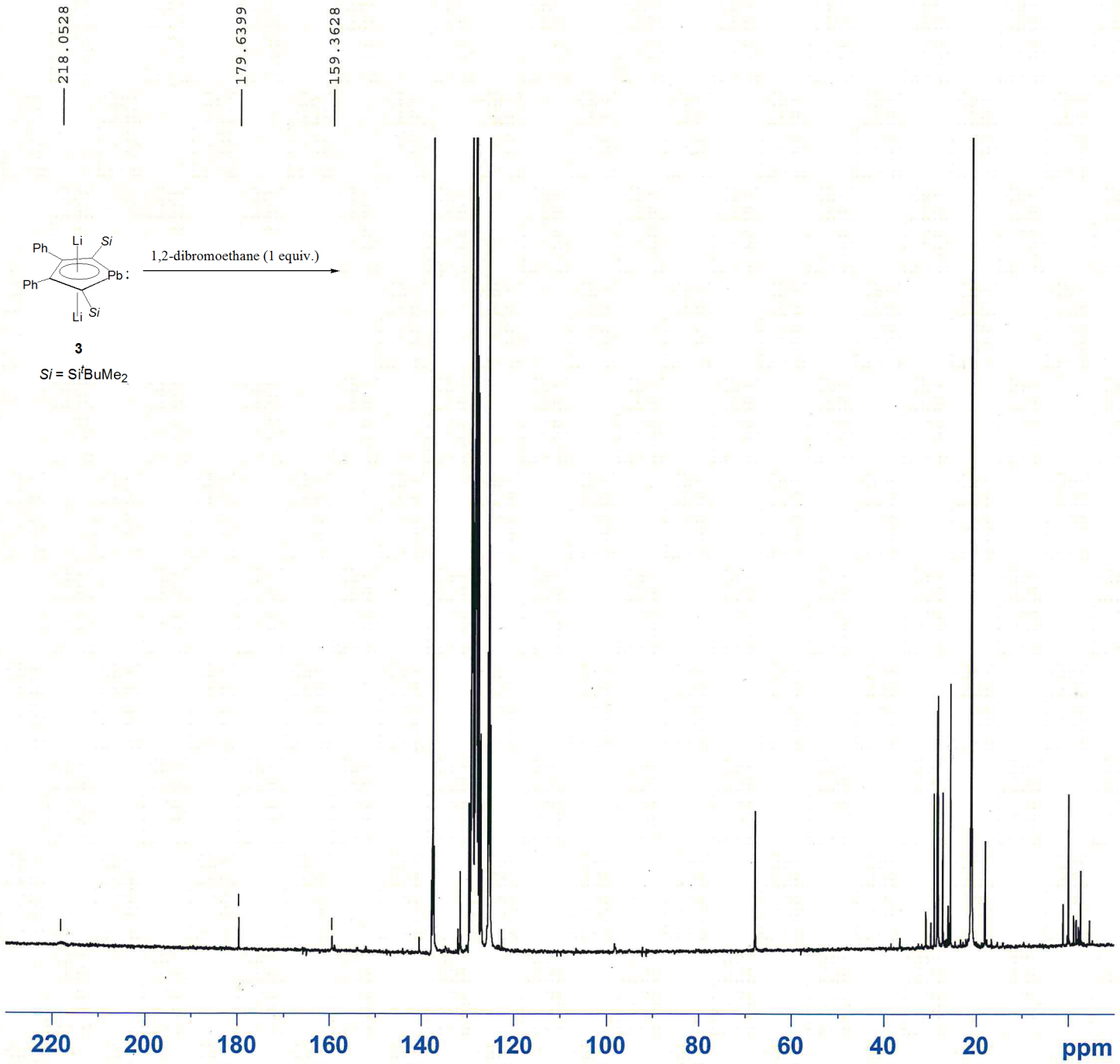
F2 - Processing parameters
SI 32768
SF 100.6127216 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

Ex 382 1H in Tol (cal@2.1)



NAME	A13MC116H
EXPNO	382
PROCNO	1
Date_	20140619
Time	14.49
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	C6D6
NS	8
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1719923 sec
RG	4
DW	48.400 usec
DE	6.50 usec
TE	300.6 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	11.80 usec
PL1	2.40 dB
PL1W	15.17711735 W
SFO1	500.0330885 MHz
SI	32768
SF	500.0300272 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

Ex 382 13C inTol cal@21.1




```
Current Data Parameters
NAME            A13MC116H
EXPNO                3822
PROCNO                  1

F2 - Acquisition Parameters
Date_            20140619
Time                15.09
INSTRUM             spect
PROBHD    5 mm CPQNP 1H/
PULPROG            zgpg30
TD                  65536
SOLVENT              C6D6
NS                     400
DS                       2
SWH             29761.904 Hz
FIDRES          0.454131 Hz
AQ              1.1010548 sec
RG                  126.99
DW                16.800 usec
DE                18.00 usec
TE                 300.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   13C
P1                  12.00 usec
PLW1         15.50000000 W
SFO1         100.6248425 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2               90.00 usec
PLW2          5.19999981 W
PLW12         0.14444000 W
PLW13         0.11700000 W
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                  32768
SF           100.6127592 MHz
WDW                    EM
SSB       0
LB                  2.00 Hz
GB        0
PC                  1.40
```

TBDMS Ph plumbacyclo~ (cal @ 2.1) in tol-d8

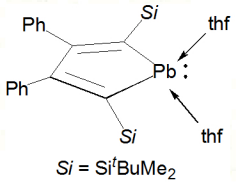
6.9976 6.9860 6.9824 6.9758 6.9704 6.9575 6.9378 6.8458 6.8416 6.8371 6.8292 6.8268 6.8240 6.8213 6.8187 6.8106 6.8066 6.8023

3.6863 3.6847 3.6800 3.6760 3.6697 3.6636 3.6595 3.6532

1.4818 1.4741 1.4653 1.4602 1.4566 1.4488 1.0311

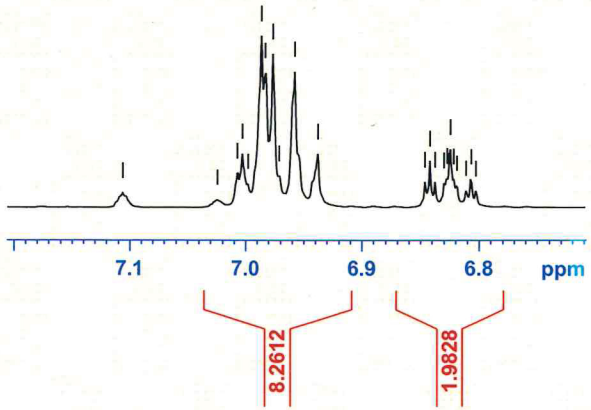
-0.1199

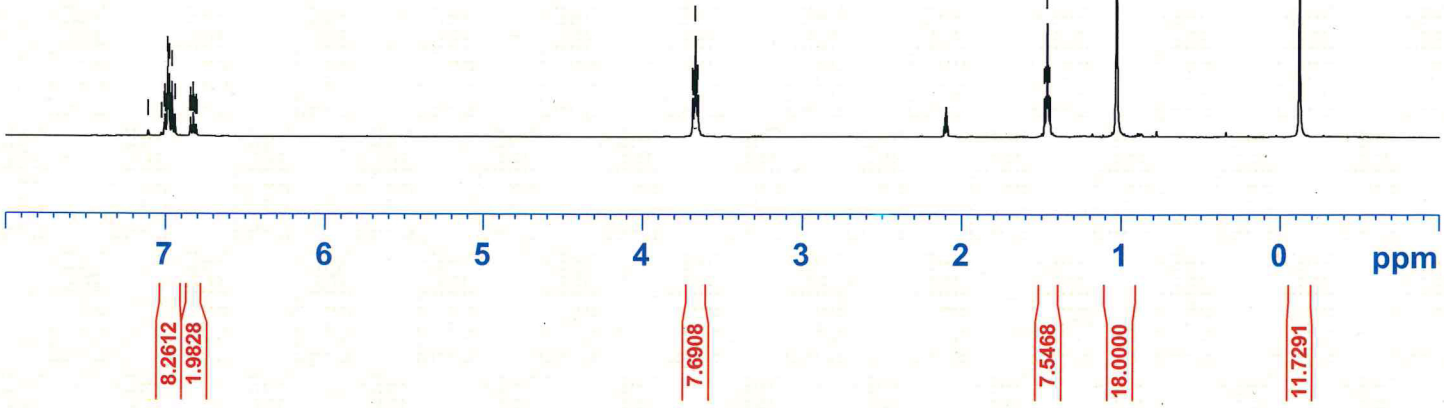
7.1056 7.0240 7.0066 7.0024 6.9976 6.9860 6.9824 6.9758 6.9704 6.9575 6.9378 6.8458 6.8416 6.8371 6.8292 6.8268 6.8240 6.8213 6.8187 6.8106



Si = Si^{t}BuMe$_2$

1





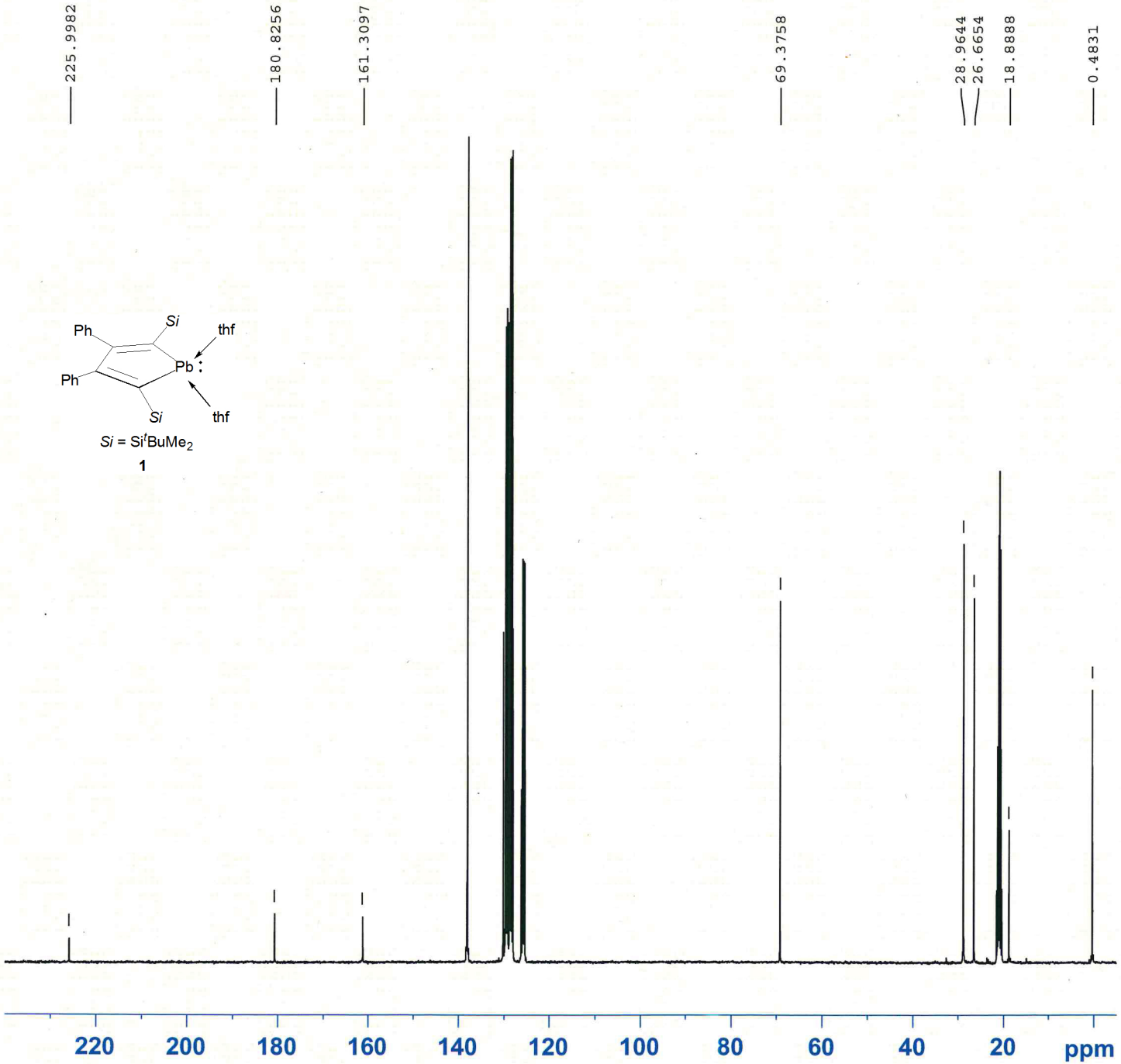
Current Data Parameters
NAME A13MC116H
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150108
Time 18.29
INSTRUM spect
PROBHD 5 mm CPQNP 1H/
PULPROG zg30
TD 65536
SOLVENT Tol
NS 4
DS 2
SWH 8305.647 Hz
FIDRES 0.126734 Hz
AQ 3.9453173 sec
RG 4.71
DW 60.200 usec
DE 10.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PLW1 5.19999981 W
SFO1 400.1324708 MHz

F2 - Processing parameters
SI 32768
SF 400.1300105 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

TBDMS Ph plumbacyclo~ (cal@21.1) in tol-d8



Si = Si^{t}BuMe$_2$

1

```
Current Data Parameters
NAME            A13MC116C
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20150108
Time                18.32
INSTRUM             spect
PROBHD   5 mm CPQNP 1H/
PULPROG            zgpg30
TD                  65536
SOLVENT               Tol
NS                    112
DS                      2
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ             1.1010548 sec
RG                126.99
DW                16.800 usec
DE                18.00 usec
TE                300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                 12.00 usec
PLW1         15.50000000 W
SFO1         100.6248425 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              90.00 usec
PLW2          5.19999981 W
PLW12         0.14444000 W
PLW13         0.11700000 W
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6126618 MHz
WDW                   EM
SSB       0
LB                 2.00 Hz
GB        0
PC                 1.40
```