Boron Cluster-based Development of Potent Non-Secosteroidal Vitamin D Receptor Ligands: Direct Observation of Hydrophobic Interaction between Protein Surface and Carborane.

Shinya Fujii,[†,‡] Hiroyuki Masuno,[‡] Yoshiyuki Taoda,[§] Atsushi Kano,[†,‡] Angsuma Wongmayura,[#] Makoto Nakabayashi,[†] Nobutoshi Ito,[†] Masato Shimizu,[†,‡] Emiko Kawachi,[†,‡] Tomoya Hirano,[‡] Yasuyuki Endo,[‖] Aya Tanatani,[#] Hiroyuki Kagechika[*,†,‡]

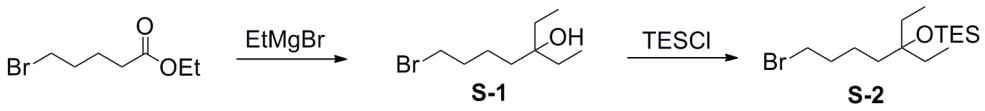
[†]Graduate School of Biomedical Science, and [‡]Institute of Biomaterials and Bioengineering, Tokyo Medical and Dental University, 2-3-10, Kanda-Surugadai, Chiyoda-ku, Tokyo 101-0062, Japan, [§]Graduate School of Pharmaceutical Sciences, University of Tokyo, 7-3-1, Hongo, Bunkyo-ku, Tokyo 113-0033, Japan, [#]Graduate School of Humanities and Sciences, Ochanomizu University, 2-1-1 Ohtsuka, Bunkyo-ku, Tokyo 112-8610, Japan, [‖]Faculty of Pharmaceutical Sciences, Tohoku Pharmaceutical University, 4-4-1 Komatsushima, Aoba-ku, Sendai 981-8558, Japan

*Corresponding author: kage.omc@tmd.ac.jp

Supporting Information

Contents

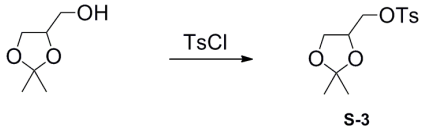
Scheme S1. Synthesis of 3-Ethyl-3-triethylsilyloxy-7-bromoheptane (S-2)..... p. S2
Scheme S2. Synthesis of 4-(*p*-toluenesulfonyloxymethyl)-2,2-dimethyl-1,3-dioxolane (S-3)..... p. S2
Scheme S3. Synthesis of (2-Phenyl-1,3-dioxan-4-yl)methyl p-toluenesulfonate (S-5) ..... P. S3
NMR Spectra..... p. S4 – S25



Scheme S1. Synthesis of 3-Ethyl-3-triethylsilyloxy-7-bromoheptane (**S-2**).

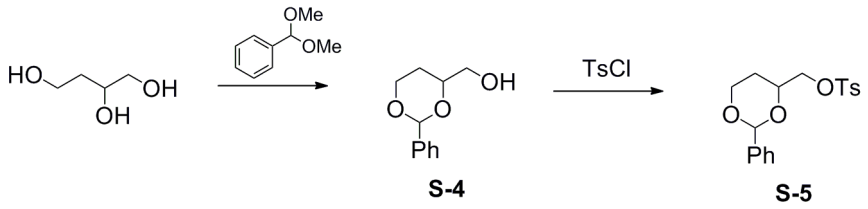
7-Bromo-3-ethyl-3-heptanol (S-1) A 0.89 M solution of EtMgBr in THF (80 mL, 90 mmol) was added to a solution of ethyl 5-bromovalerate (8.54 g, 40.9 mmol) in THF (160 mL) at 0 °C and the mixture was stirred at room temperature for 3 h. The mixture was poured into water and extracted with ethyl acetate. The organic layer was washed with brine, dried over magnesium sulfate and then concentrated. Purification by silica gel column chromatography (eluent; hexane/ethyl acetate, 4:1) gave **S-1** (68%) as colorless oil; ^{1}H-NMR ($CDCl_3$, 400 MHz) δ 3.41 (t, J = 6.8 Hz, 2 H), 1.85 (quint, J = 6.8 Hz, 2 H), 1.45 (q, J = 6.8 Hz, 4 H), 1.39 (m, 4 H), 1.36 (s, 1 H), 0.85 (t, J = 7.5 Hz, 6 H).

3-Ethyl-3-triethylsilyloxy-7-bromoheptane (S-2) Triethylsilyl chloride (9.52 g, 36.0 mmol) and 2,6-lutidine (5.94 g, 55.4 mmol) were added to a solution of **S-1** (6.18 g, 27.7 mmol) at 0 °C and the mixture was stirred at room temperature for 6 h. The mixture was poured into water and extracted with dichloromethane. The organic layer was washed with brine, dried over magnesium sulfate and then concentrated. Purification by silica gel column chromatography (eluent; hexane/ethyl acetate) gave **S-2** (72%) as colorless oil; ^{1}H-NMR ($CDCl_3$, 400 MHz) δ 3.40 (t, J = 6.8 Hz, 2 H), 1.82 (m, 2 H), 1.47-1.39 (m, 6 H), 0.93 (t, J = 8.1 Hz, 9 H), 0.81 (t, J = 7.3 Hz, 6 H), 0.55 (q, J = 7.9 Hz, 6 H).



Scheme S2. Synthesis of 4-(*p*-toluenesulfonyloxymethyl)-2,2-dimethyl-1,3-dioxolane (S-3).

4-(*p*-toluenesulfonyloxymethyl)-2,2-dimethyl-1,3-dioxolane (S-3) A mixture of 2,2-dimetyl-1,3-dioxolane-4-methanol (5.04 g, 38.1 mmol), pyridine (10 mL) and *p*-toluenesulfonyl chloride in dichloromethane (30 mL) was stirred at room temperature for 14 h. Insoluble materials were filtered off, the solvent was removed under reduced pressure. Purification by silica gel column chromatography (eluent; dichloromethane) gave **S-3** (94%) as colorless soild. ^{1}H-NMR ($CDCl_3$, 400 MHz) δ 7.80 (d, J = 8.4 Hz, 2 H), 7.35 (d, J = 8.4Hz, 2 H), 4.30-4.24 (m, 1 H), 4.06-3.95 (m, 3H), 3.79-3.74 (m, 1 H), 2.45 (s, 3 H), 1.34 (s, 3 H), 1.31 (s, 3 H).



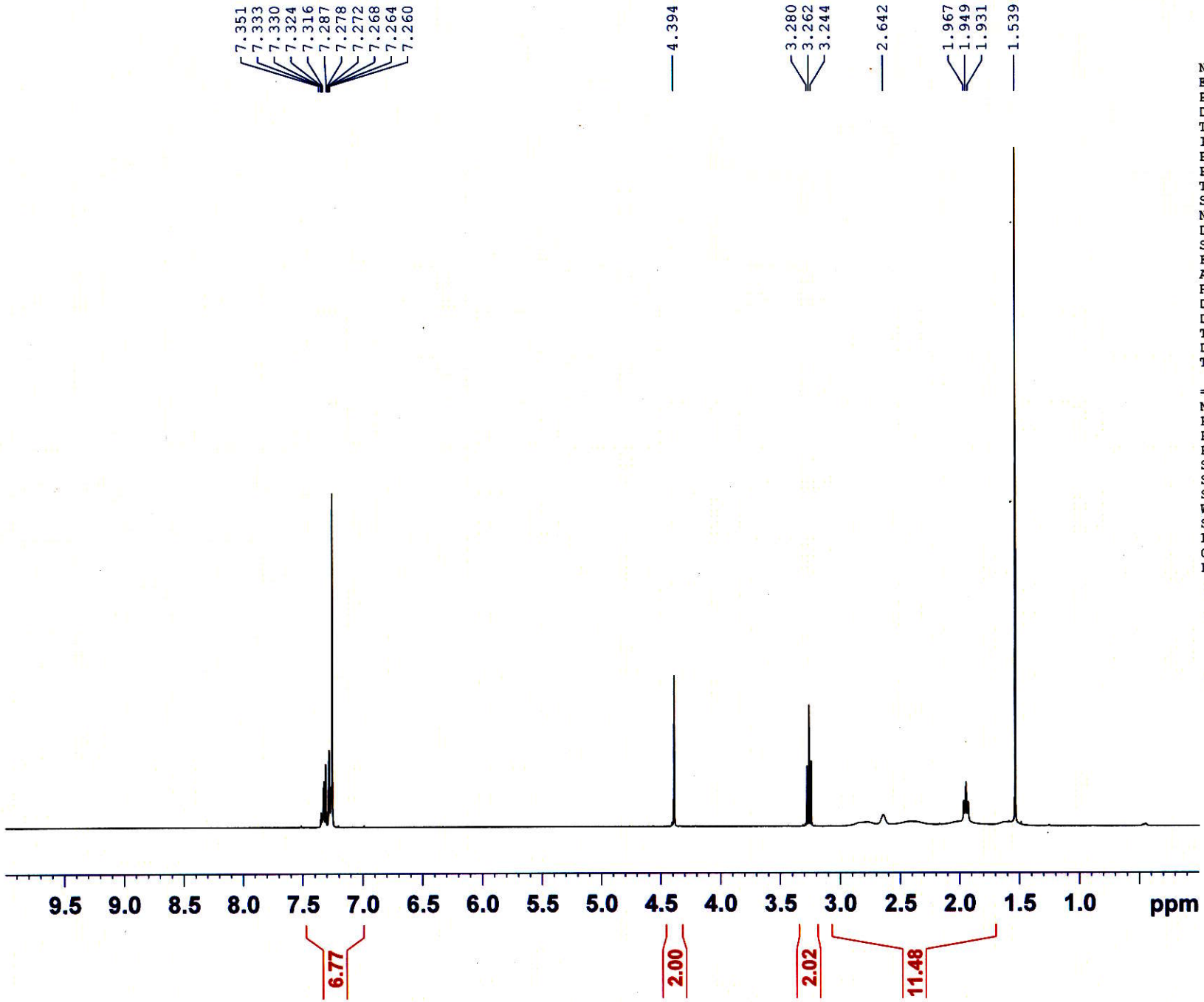
Scheme S3. Synthesis of (2-Phenyl-1,3-dioxan-4-yl)methyl p-toluenesulfonate (S-5)

(2-Phenyl-1,3-dioxan-4-yl)methanol (S-4) Benzaldehyde dimethylacetal (1.58 g, 10.4 mmol) and a catalytic amount of p-toluenesulfonic acid was added to a solution of 1,2,4-butanetriol (1.00 g, 9.42 mmol) in DMF (30 mL). The mixture was stirred at 60 °C for 3 h and then concentrated under reduced pressure. Purification by silica gel column chromatography (eluent; hexane/ethyl acetate, 1:1) gave **S-4** (91%); ^{1}H-NMR ($CDCl_3$, 400 MHz) δ 7.49-7.47 (m, 2 H), 7.38-7.31 (m, 5 H), 5.54 (s, 1 H), 4.29 (dd, *J* = 11.4 Hz, 4.2 Hz, 1 H), 4.04-3.97 (m, 2 H), 3.70-3.64 (m, 2 H), 2.06 (t, *J* = 5.7 Hz, 1 H), 1.92 (dq, *J* = 12.5 Hz, 5.1 Hz, 1 H), 1.45 (d, *J* = 13.2 Hz, 1 H).

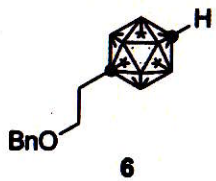
(2-Phenyl-1,3-dioxan-4-yl)methyl p-toluenesulfonate (S-5) Compound **S-5** was prepared from **S-4** in a similar manner used for preparation of **S-3**. Purification by silica gel column chromatography (eluent; hexane/ethyl acetate, 2:1) gave **S-5** (86%); ^{1}H-NMR (CDCl3, 400 MHz) δ 7.77 (d, *J* = 8.2 Hz, 2 H), 7.39-7.31 (m, 5 H), 7.26 (d, *J* = 8.2 Hz, 2 H), 5.44 (s, 1 H), 4.26 (dd, *J* = 11.5 Hz, 4.9 Hz, 1 H), 4.15-4.03 (m, 3 H), 3.93 (dt, *J* = 12.3 Hz, 2.6 Hz, 1 H), 2.41 (s, 3 H), 1.79 (dq, *J* = 12.8 Hz, 5.3 Hz, 1 H), 1.53 (d, *J* = 13.2 Hz, 1 H).

carborane-VDR

IBB-nmr Analysis



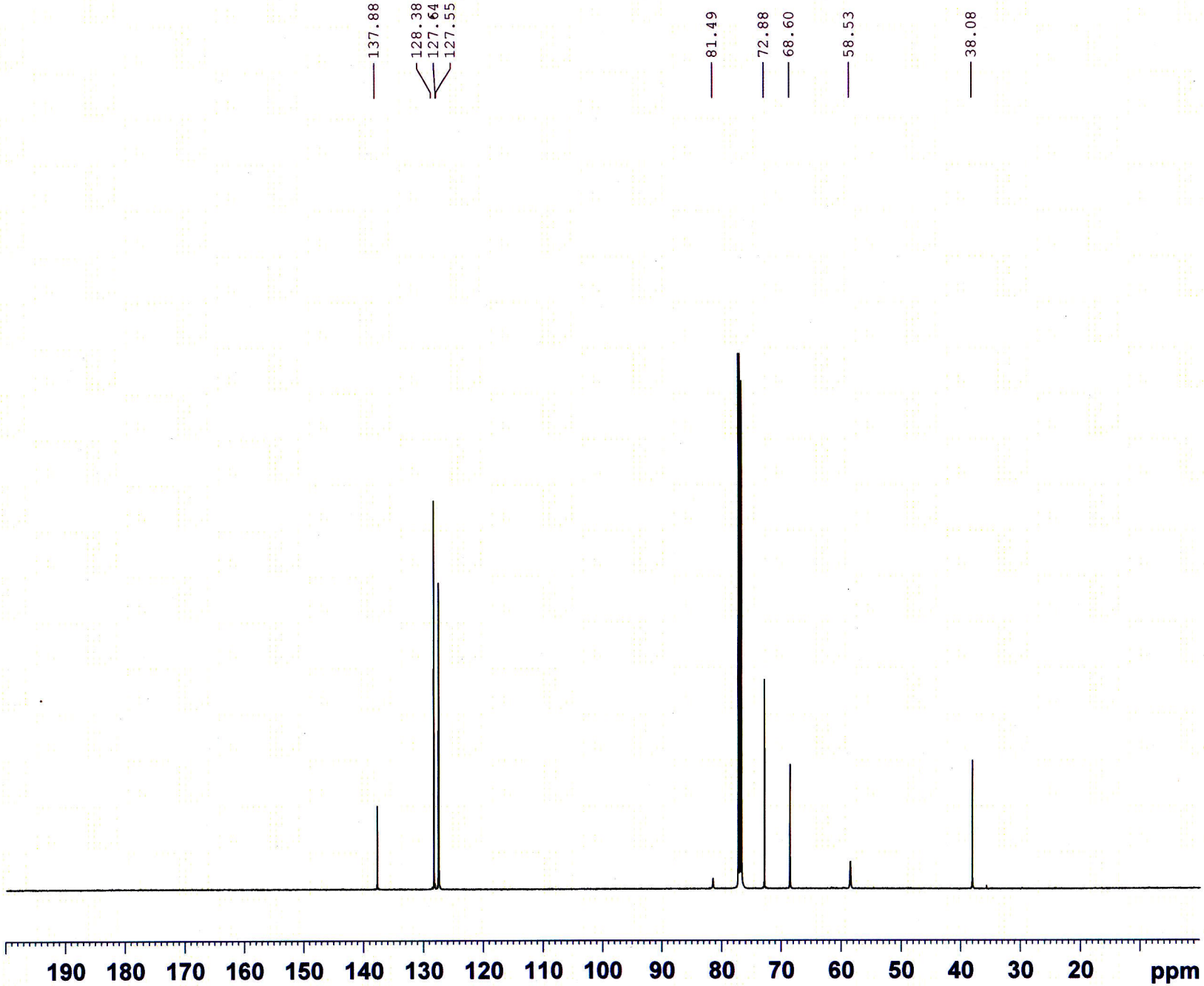
NAME	201001-064-carborane-clm
EXPNO	1
PROCNO	1
Date_	20110706
Time	18.43
INSTRUM	av400
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	203
DW	60.800 usec
DE	6.50 usec
TE	301.2 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.00 usec
PL1	-1.80 dB
PL1W	14.82738590 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300099 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



6

Carborane-VDR

IBB-nmr Analysis

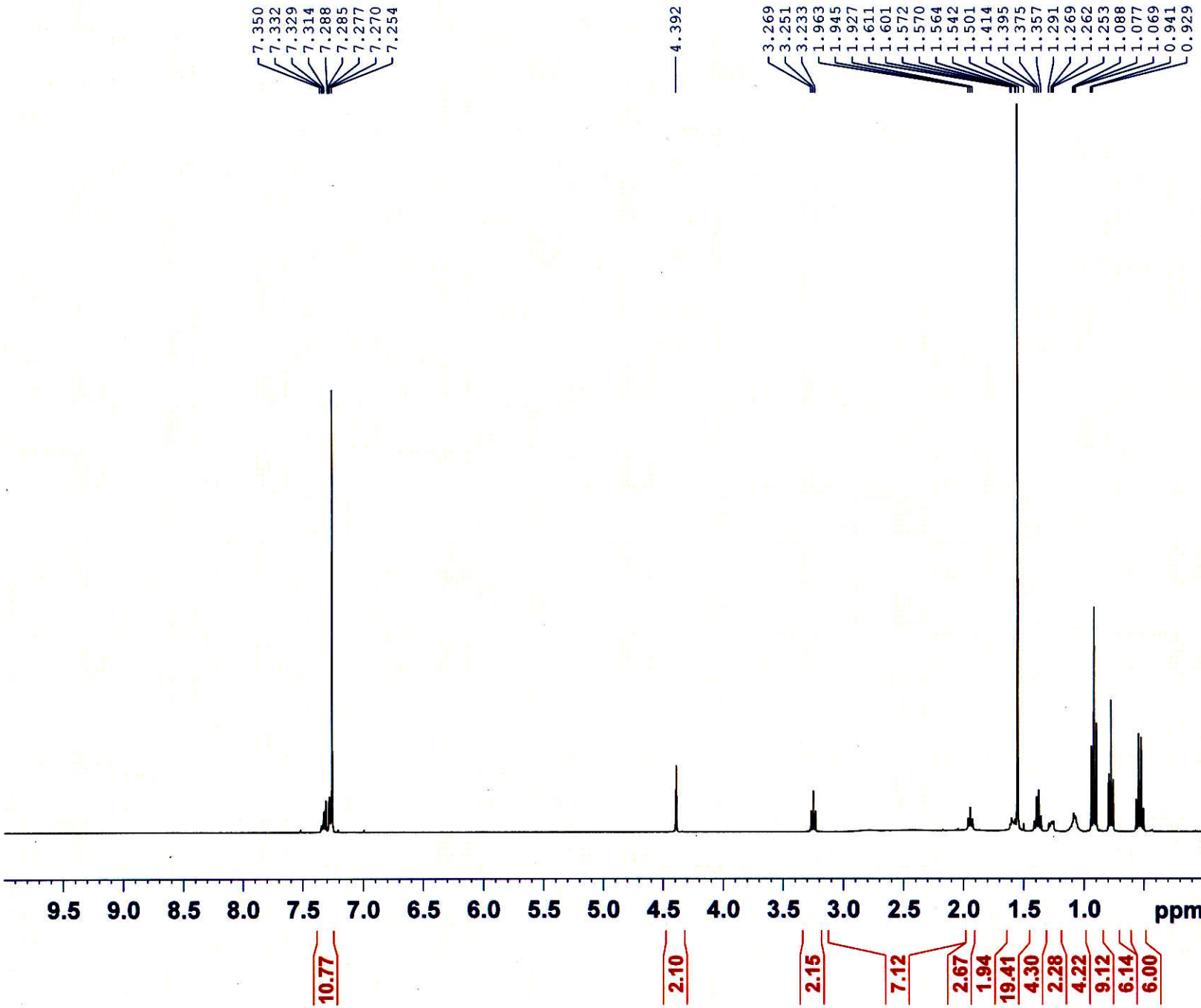


NAME	201001-64-p-carborane-CH2CH2OBn-13C
EXPNO	1
PROCNO	1
Date_	20110707
Time	11.53
INSTRUM	av500
PROBHD	5 mm CPDCH 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	256
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	90.5
DW	16.650 usec
DE	20.00 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
========	CHANNEL f1 ========
NUC1	13C
P1	10.00 usec
PL1	-4.80 dB
PL1W	13.65439701 W
SFO1	125.7703643 MHz
========	CHANNEL f2 ========
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-4.80 dB
PL12	12.80 dB
PL13	16.00 dB
PL2W	7.58577585 W
PL12W	0.13182567 W
PL13W	0.06309573 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577932 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	0.30

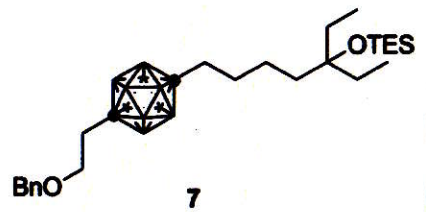
6

carborane-VDR

IBB-nmr Analysis



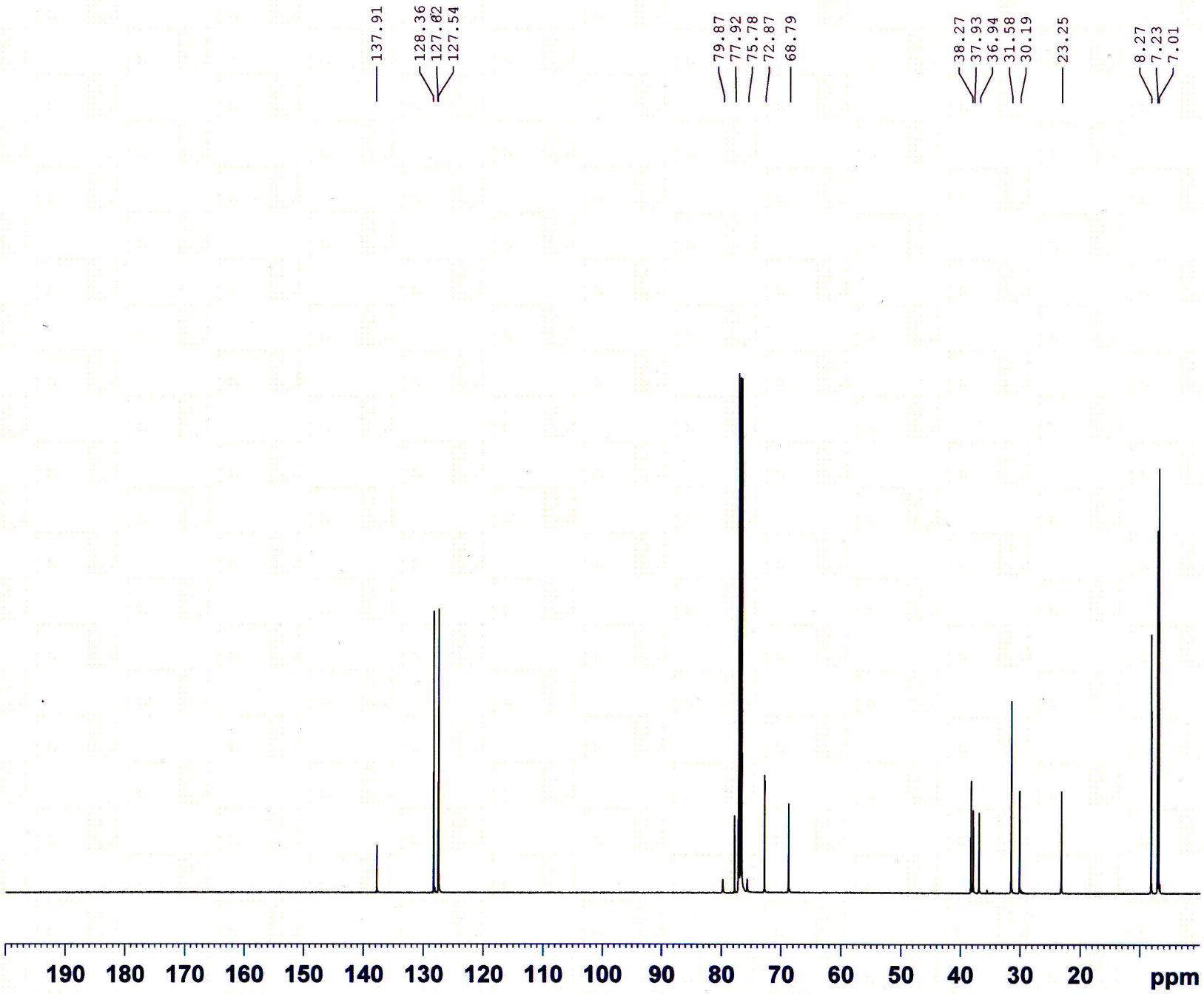
NAME	201001-069-clm-pure-carborane
EXPNO	1
PROCNO	1
Date_	20110803
Time	12.21
INSTRUM	av400
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	203
DW	60.800 usec
DE	6.50 usec
TE	3009.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.00 usec
PL1	-1.80 dB
PL1W	14.82738590 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300083 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



7

carborane-VDR

IBB-nmr Analysis

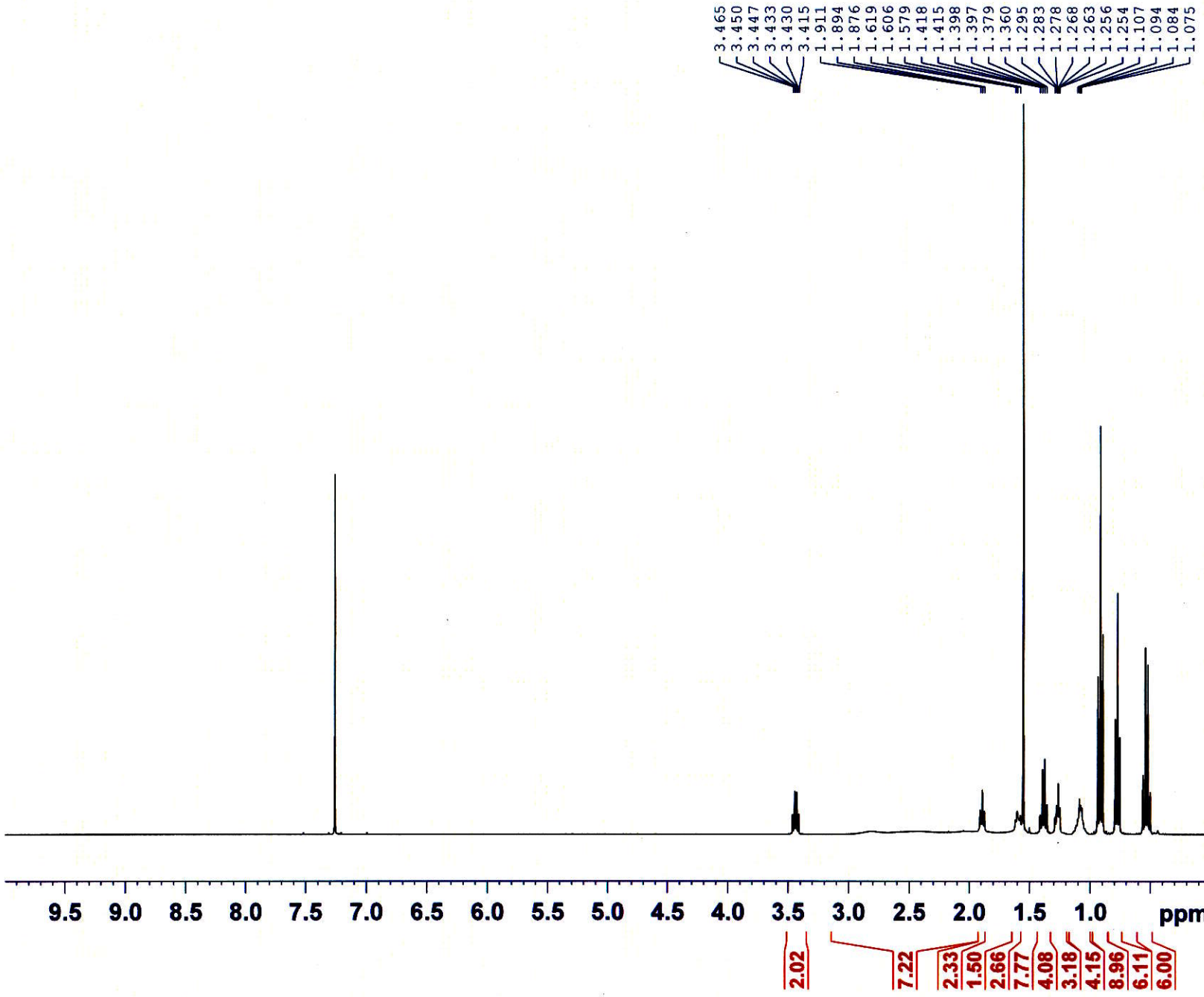


NAME	201001-069-clm-pure-13Ccarborane
EXPNO	1
PROCNO	1
Date_	20110803
Time	16.25
INSTRUM	av500
PROBHD	5 mm CPDCH 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	400
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	90.5
DW	16.650 usec
DE	20.00 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	10.00 usec
PL1	-4.80 dB
PL1W	13.65439701 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-4.80 dB
PL12	12.80 dB
PL13	16.00 dB
PL2W	7.58577585 W
PL12W	0.13182567 W
PL13W	0.06309573 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577932 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	0.30

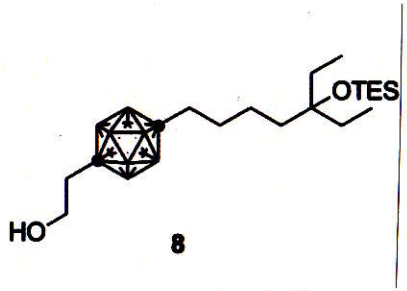
7

carborane-VDR

IBB-nmr Analysis



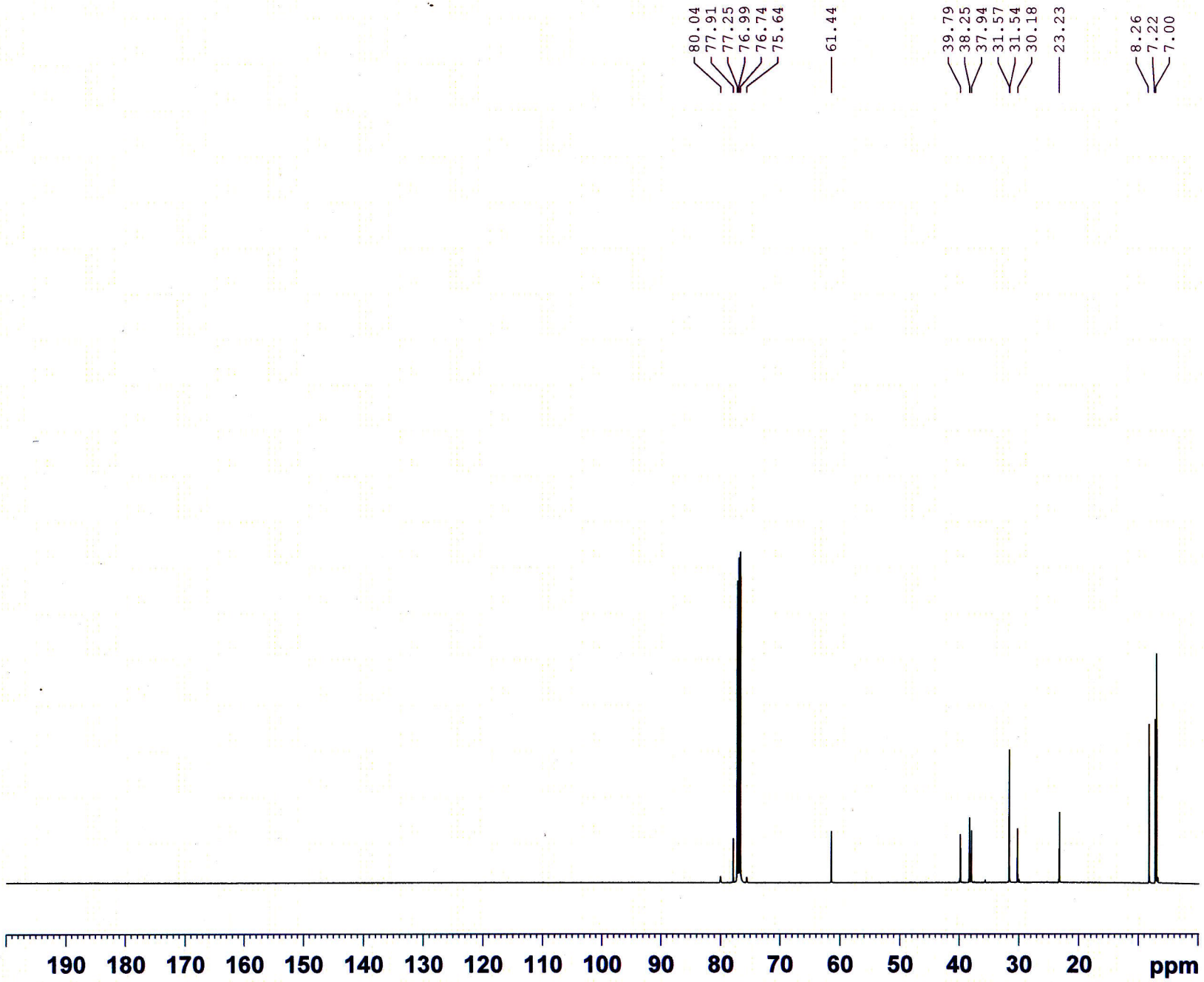
NAME	201001-074-clm-carborane-400MHz
EXPNO	1
PROCNO	1
Date_	20110810
Time	22.59
INSTRUM	av400
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	203
DW	60.800 usec
DE	6.50 usec
TE	302.4 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.00 usec
PL1	-1.80 dB
PL1W	14.82738590 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300083 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



8

carborane-VDR

IBB-nmr Analysis

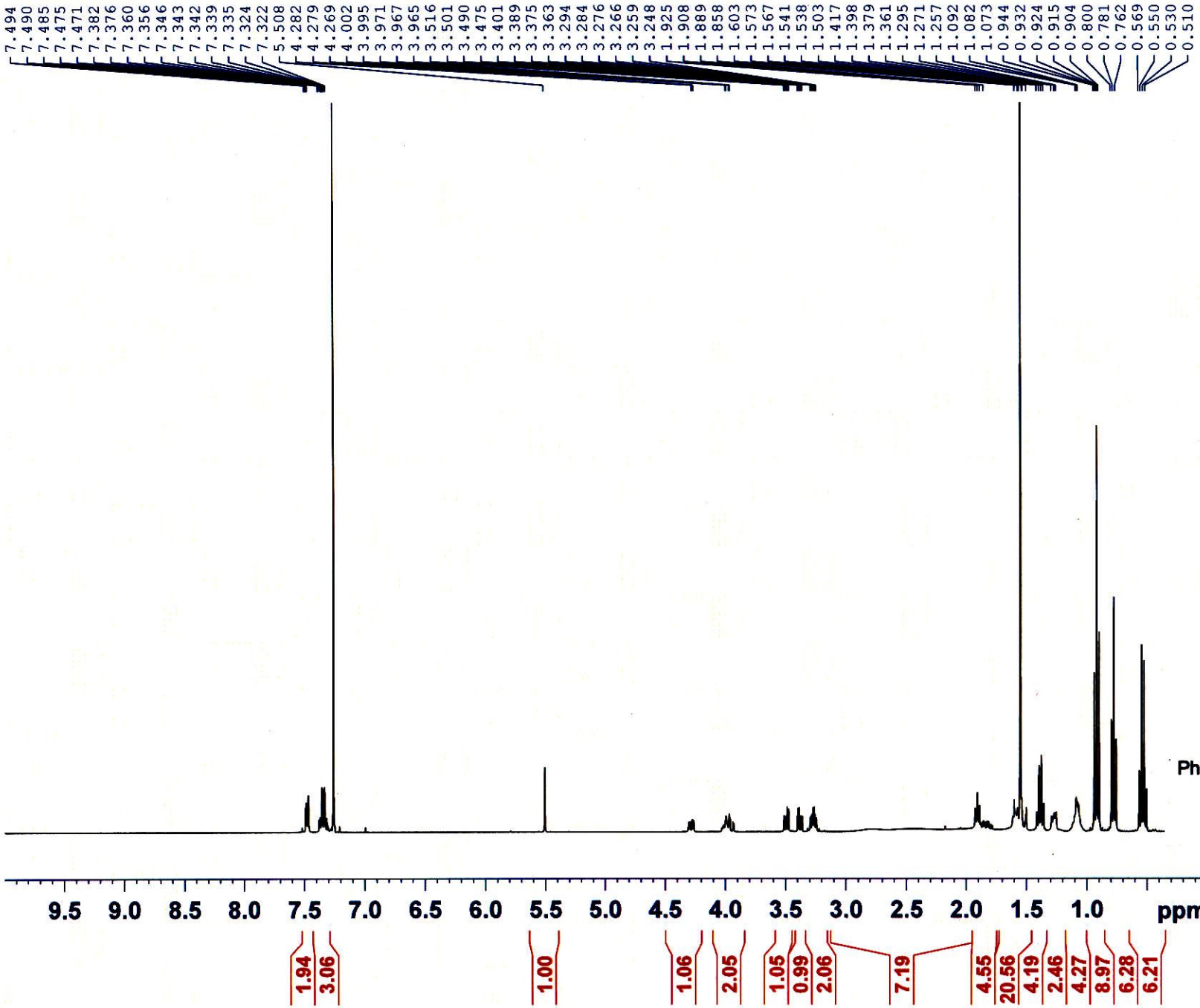


NAME	201001-074-clm-13C-carborane	
EXPNO	1	
PROCNO	1	
Date_	20110808	
Time	21.00	
INSTRUM	av500	
PROBHD	5 mm CPDCH 13C	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	512	
DS	4	
SWH	30030.029	Hz
FIDRES	0.458222	Hz
AQ	1.0912410	sec
RG	90.5	
DW	16.650	usec
DE	20.00	usec
TE	298.0	K
D1	2.00000000	sec
D11	0.03000000	sec
TD0	1	
========	CHANNEL f1	========
NUC1	13C	
P1	10.00	usec
PL1	-4.80	dB
PL1W	13.65439701	W
SFO1	125.7703643	MHz
========	CHANNEL f2	========
CPDPRG2	waltz16	
NUC2	1H	
PCPD2	80.00	usec
PL2	-4.80	dB
PL12	12.80	dB
PL13	16.00	dB
PL2W	7.58577585	W
PL12W	0.13182567	W
PL13W	0.06309573	W
SFO2	500.1320005	MHz
SI	32768	
SF	125.7577932	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	0.30	

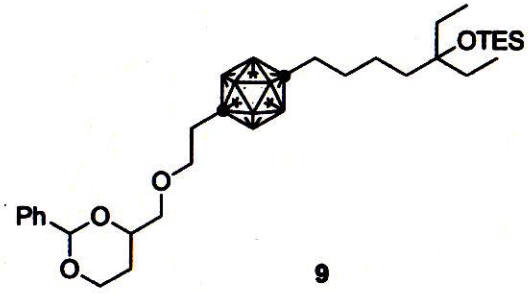
8

carborane-VDR

IBB-nmr Analysis



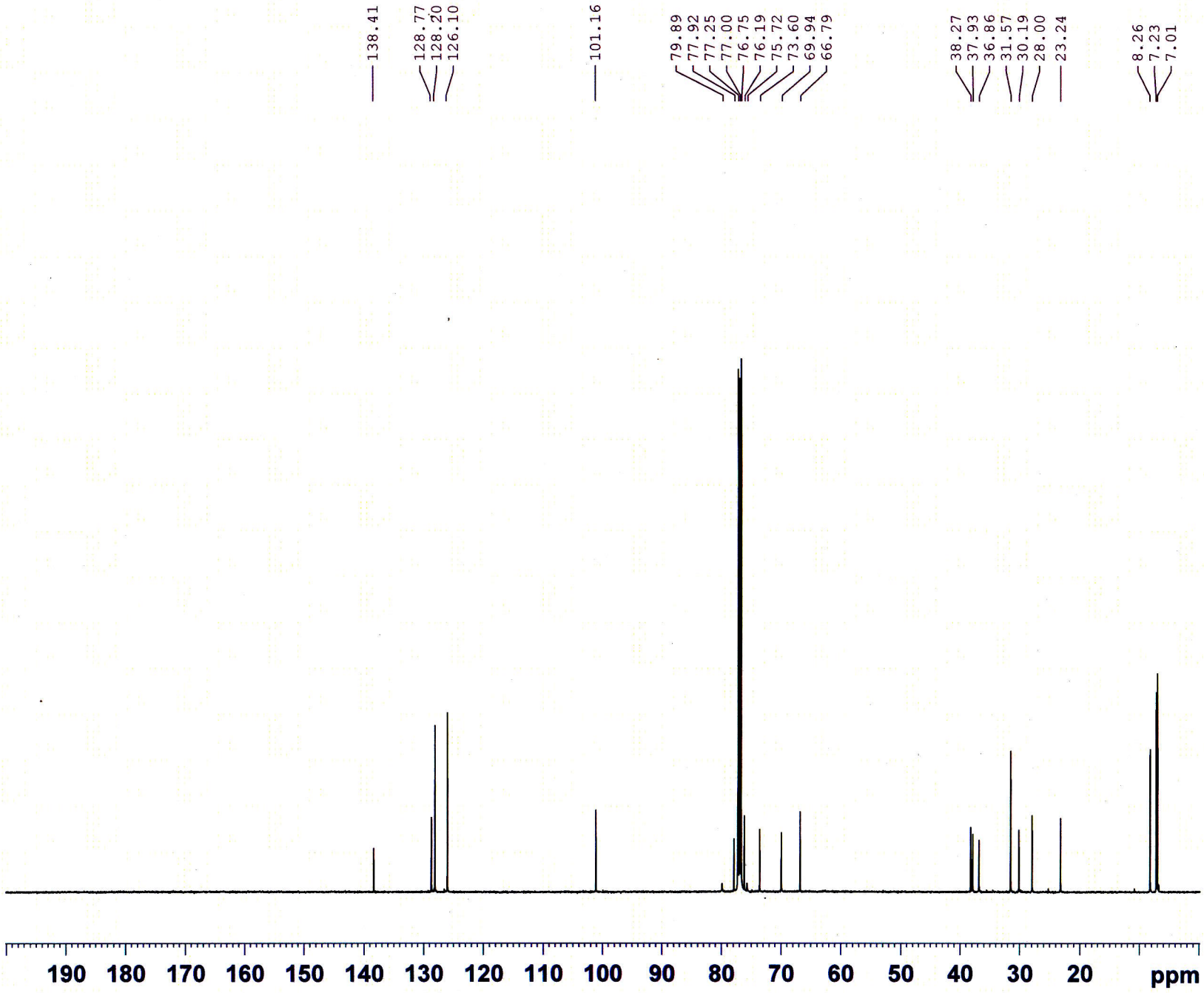
NAME	201001-076-clm2-carborane
EXPNO	1
PROCNO	1
Date_	20110812
Time	14.15
INSTRUM	av400
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	64
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	203
DW	60.800 usec
DE	6.50 usec
TE	301.9 K
D1	1.00000000 sec
TD0	1
========	CHANNEL f1 ========
NUC1	1H
P1	14.00 usec
PL1	-1.80 dB
PL1W	14.82738590 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300083 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



9

carborane-VDR

IBB-nmr Analysis

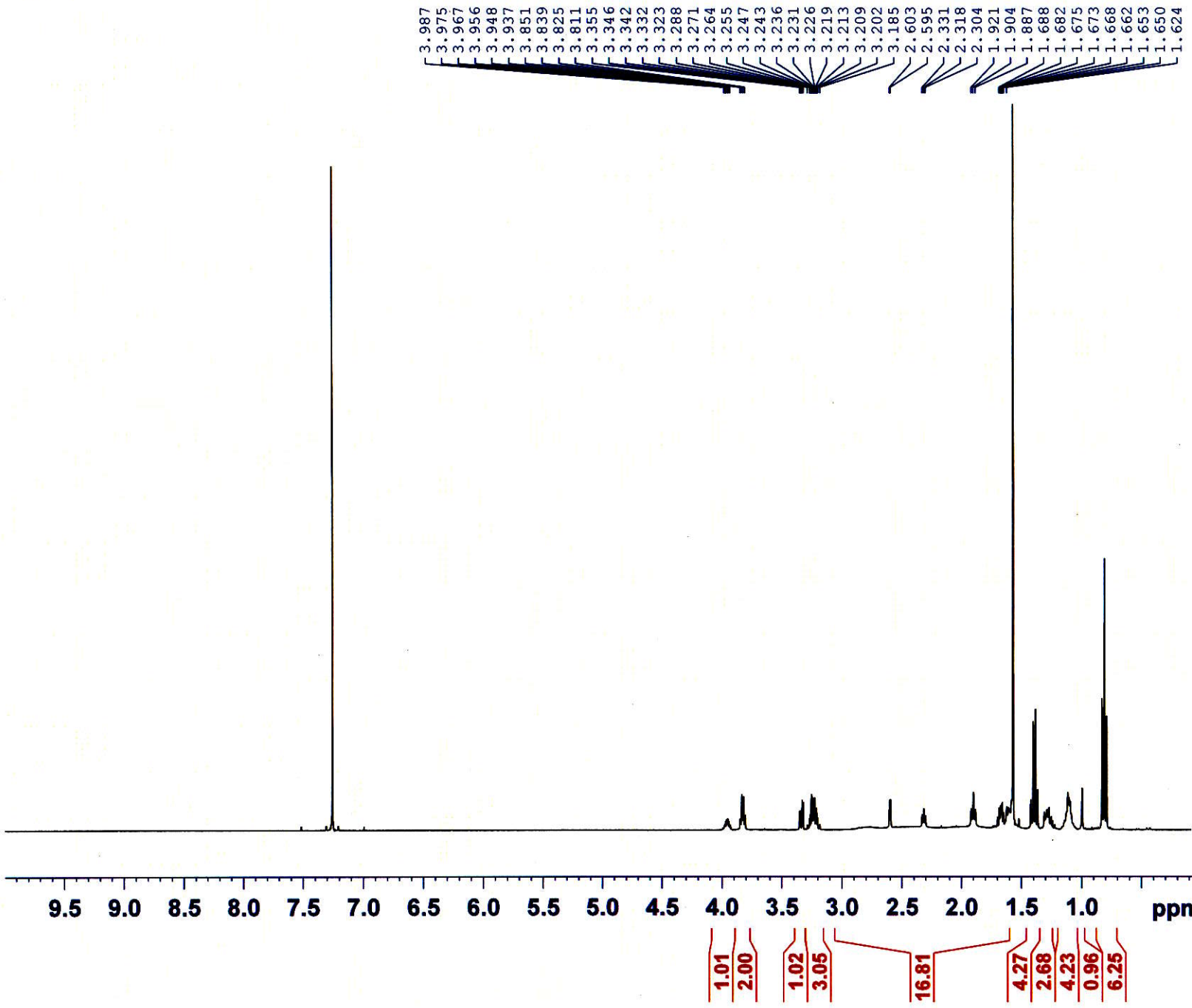


NAME	201001-076-clm2-13C-carborane
EXPNO	1
PROCNO	1
Date_	20110812
Time	14.49
INSTRUM	av500
PROBHD	5 mm CPDCH 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	128
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	90.5
DW	16.650 usec
DE	20.00 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	10.00 usec
PL1	-4.80 dB
PL1W	13.65439701 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-4.80 dB
PL12	12.80 dB
PL13	16.00 dB
PL2W	7.58577585 W
PL12W	0.13182567 W
PL13W	0.06309573 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577932 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	0.30

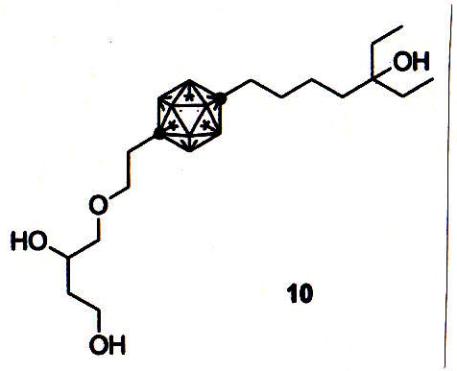
9

carborane-VDR

IBB-nmr Analysis



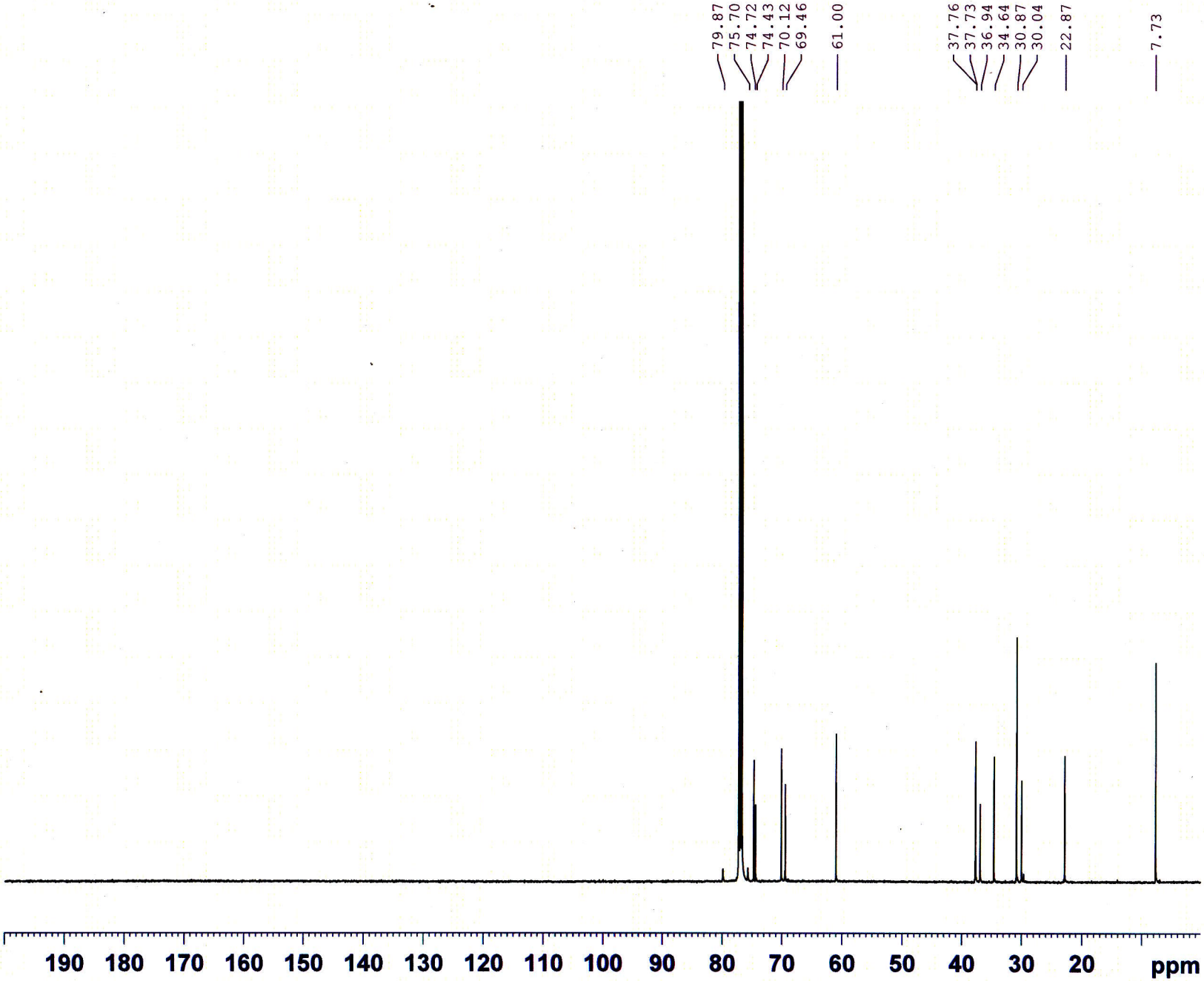
NAME	201001-078-clm-3-carborane
EXPNO	1
PROCNO	1
Date_	20110823
Time	14.05
INSTRUM	av400
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	203
DW	60.800 usec
DE	6.50 usec
TE	300.9 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.00 usec
PL1	-1.80 dB
PL1W	14.82738590 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300083 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



10

carborane-VDR
#78-N128

IBB-nmr Analysis

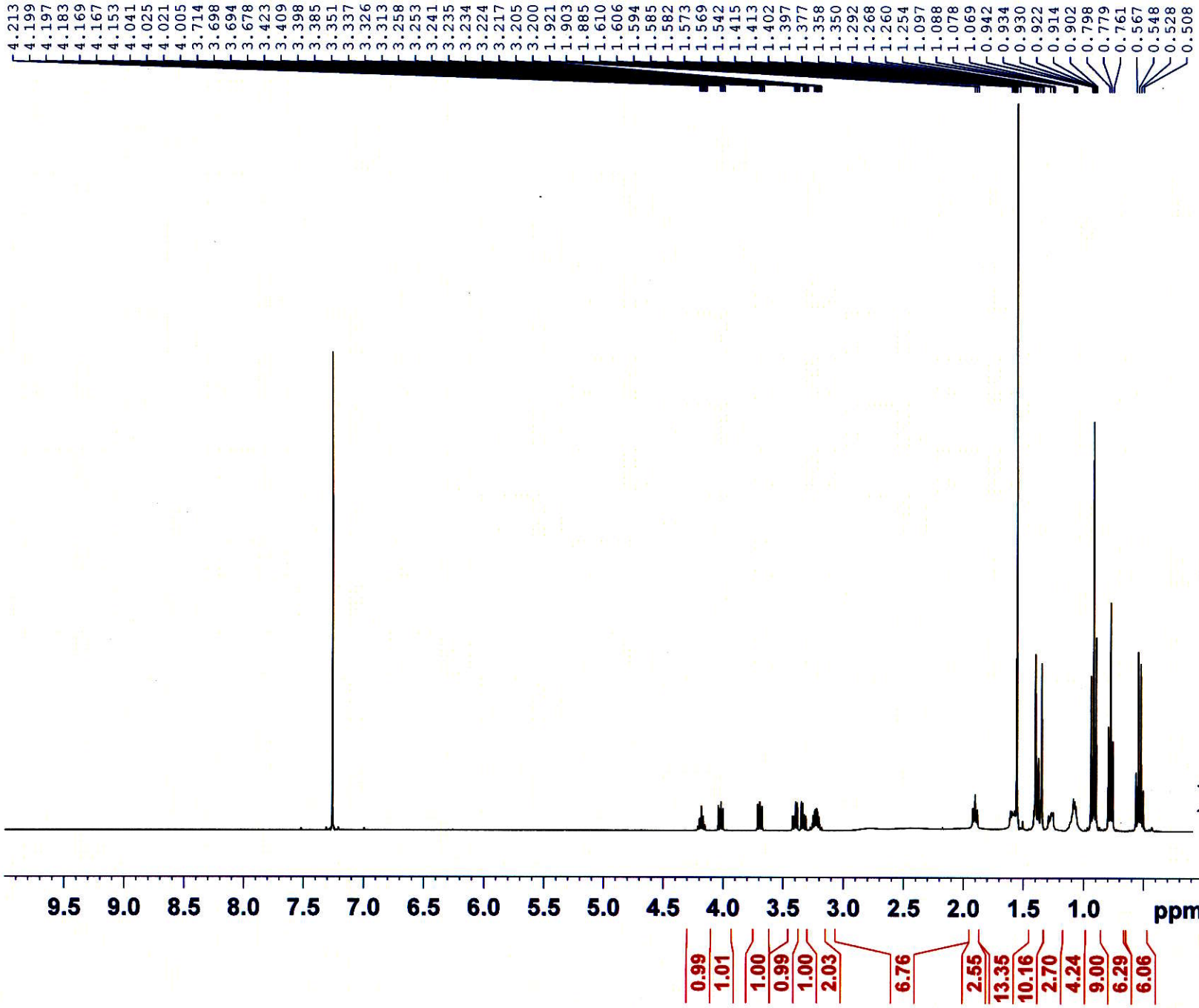


NAME	201001-078-clm-3-13C-carborane
EXPNO	1
PROCNO	1
Date_	20110823
Time	15.10
INSTRUM	av500
PROBHD	5 mm CPDCH 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	256
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	128
DW	16.650 usec
DE	20.00 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
========	CHANNEL f1 ========
NUC1	13C
P1	10.00 usec
PL1	-4.80 dB
PL1W	13.65439701 W
SFO1	125.7703643 MHz
========	CHANNEL f2 ========
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-4.80 dB
PL12	12.80 dB
PL13	16.00 dB
PL2W	7.58577585 W
PL12W	0.13182567 W
PL13W	0.06309573 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577932 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	0.30

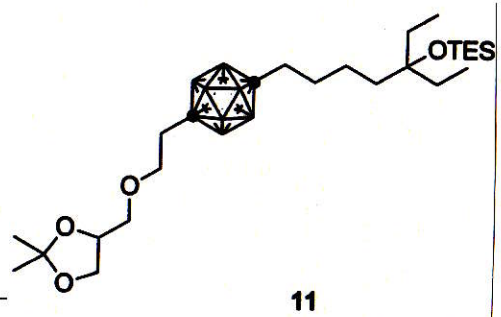
10

carborane-VDR

IBB-nmr Analysis



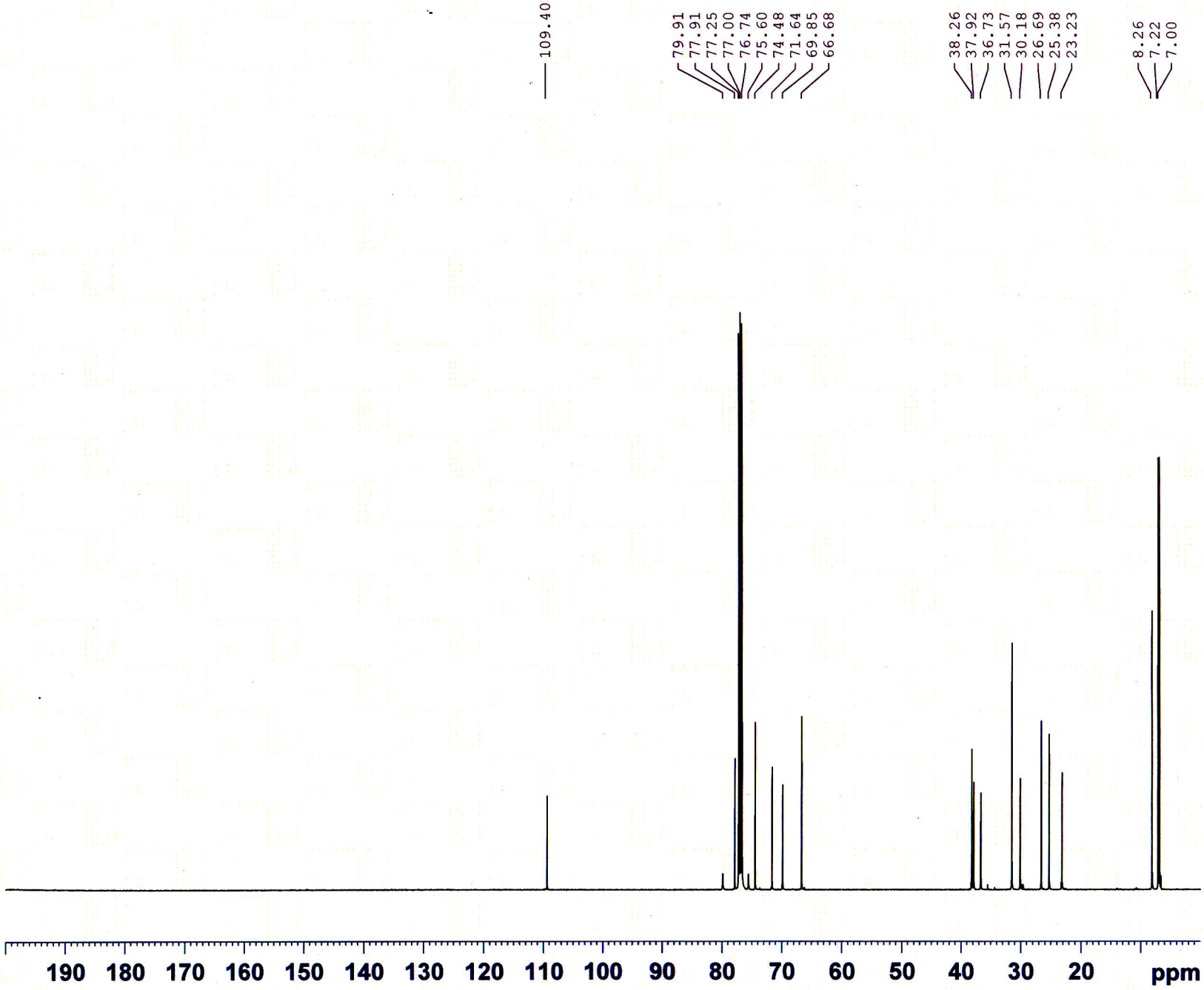
NAME	201001-075-clm-carborane-2
EXPNO	1
PROCNO	1
Date_	20110811
Time	9.25
INSTRUM	av400
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	203
DW	60.800 usec
DE	6.50 usec
TE	301.7 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.00 usec
PL1	-1.80 dB
PL1W	14.82738590 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300083 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



11

carborane-VDR

IBB-nmr Analysis

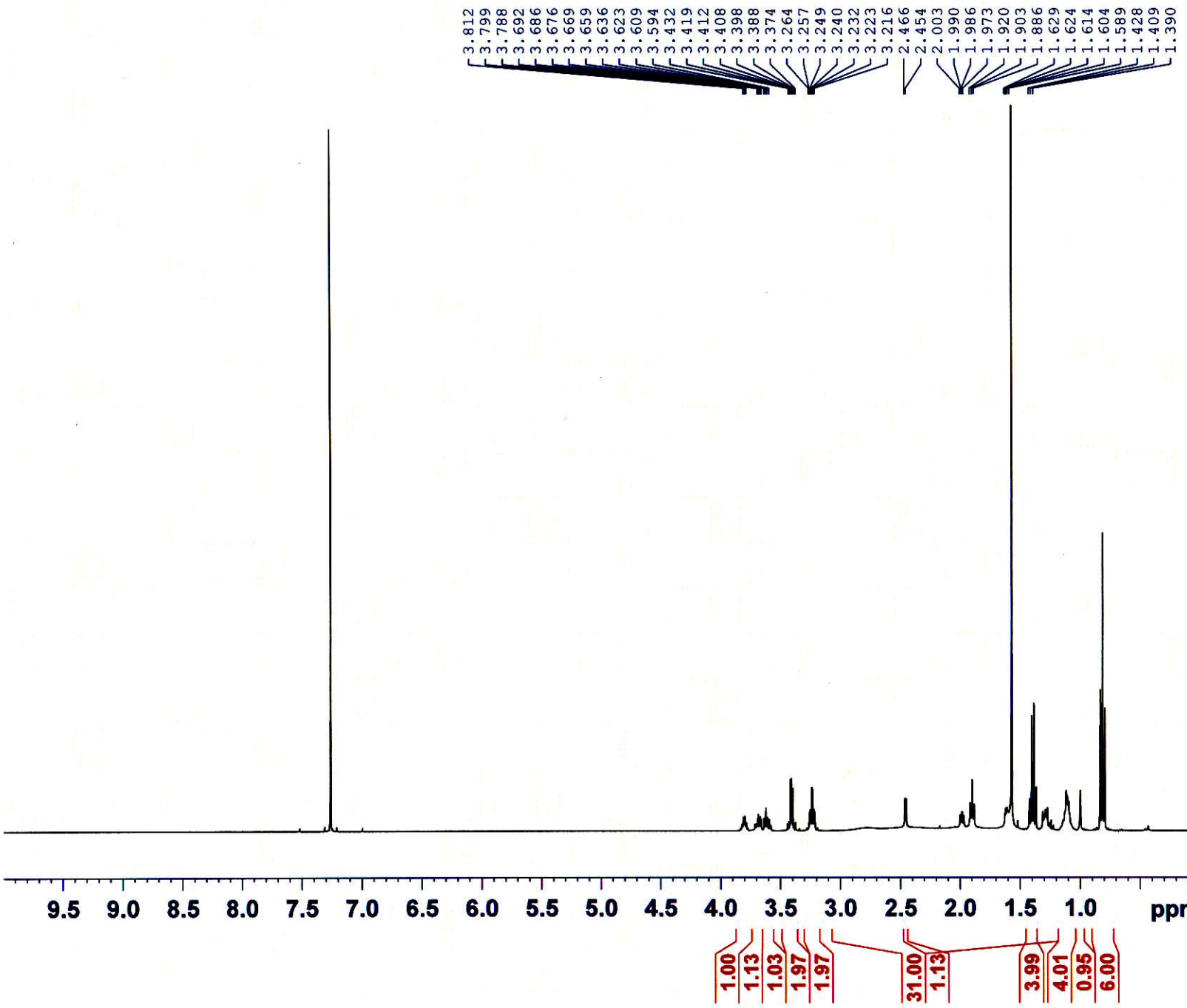


NAME	201001-075-clm-13C-carborane
EXPNO	1
PROCNO	1
Date_	20110811
Time	9.30
INSTRUM	av500
PROBHD	5 mm CPDCH 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	512
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	90.5
DW	16.650 usec
DE	20.00 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	10.00 usec
PL1	-4.80 dB
PL1W	13.65439701 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-4.80 dB
PL12	12.80 dB
PL13	16.00 dB
PL2W	7.58577585 W
PL12W	0.13182567 W
PL13W	0.06309573 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577932 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	0.30

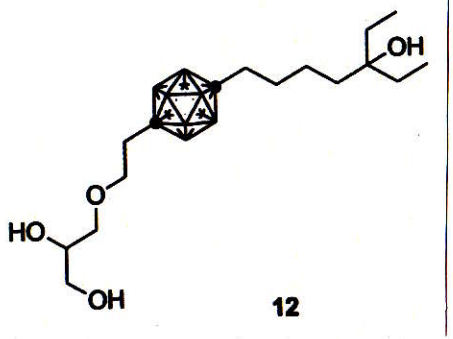
11

carborane-VDR

IBB-nmr Analysis

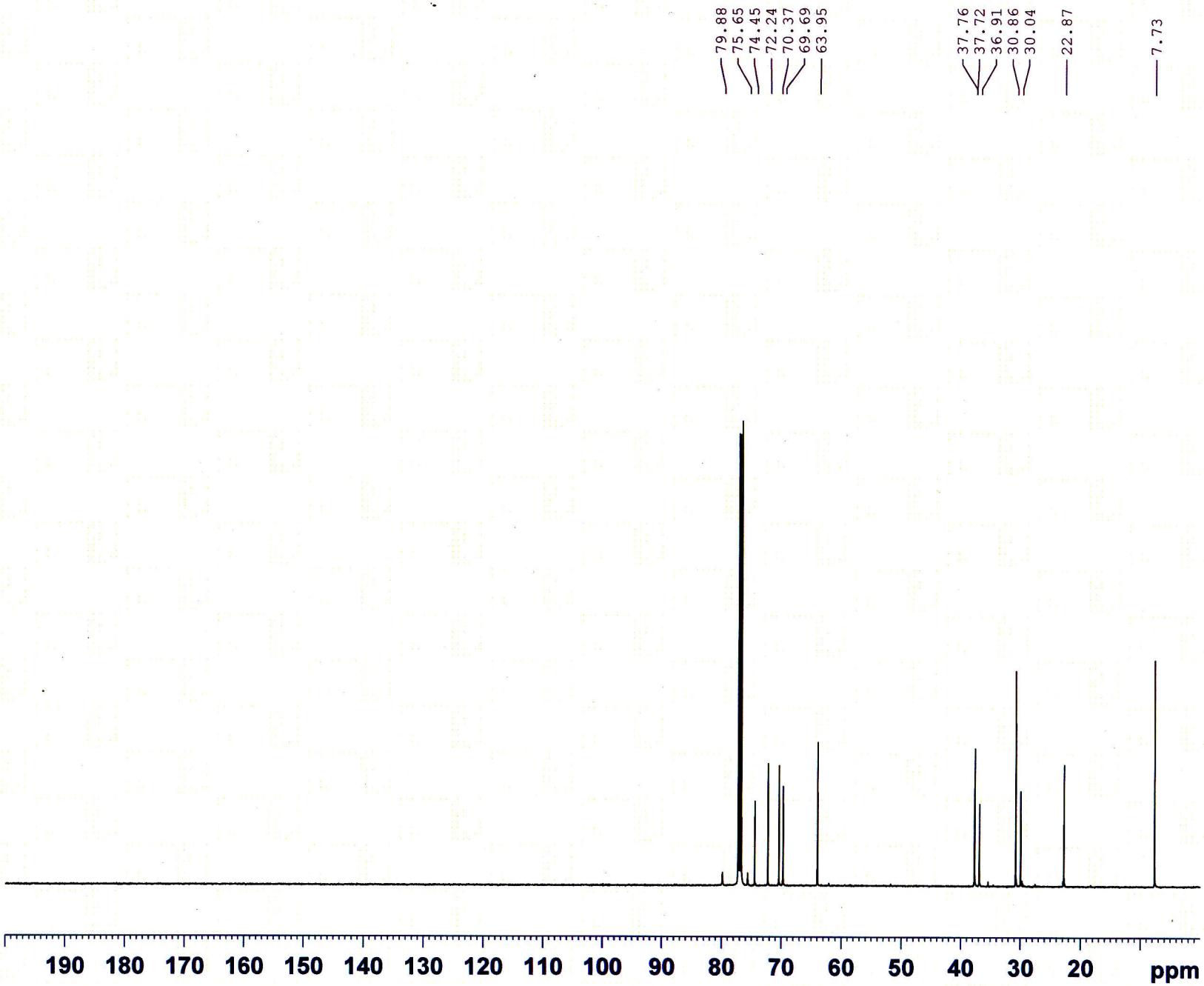


NAME	201001-077-clm-carborane
EXPNO	1
PROCNO	1
Date_	20110822
Time	12.40
INSTRUM	av400
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	203
DW	60.800 usec
DE	6.50 usec
TE	300.8 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.00 usec
PL1	-1.80 dB
PL1W	14.82738590 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300083 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



12

carborane-VDR



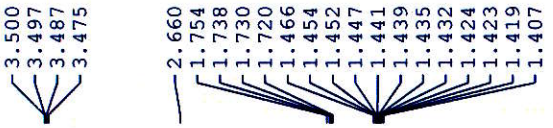
IBB-nmr Analysis

NAME	201001-077-clm-13C-carborane
EXPNO	1
PROCNO	1
Date_	20110822
Time	13.44
INSTRUM	av500
PROBHD	5 mm CPDCH 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	128
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	90.5
DW	16.650 usec
DE	20.00 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	10.00 usec
PL1	-4.80 dB
PL1W	13.65439701 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-4.80 dB
PL12	12.80 dB
PL13	16.00 dB
PL2W	7.58577585 W
PL12W	0.13182567 W
PL13W	0.06309573 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577932 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	0.30

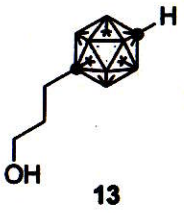
12

Carborane-VDR

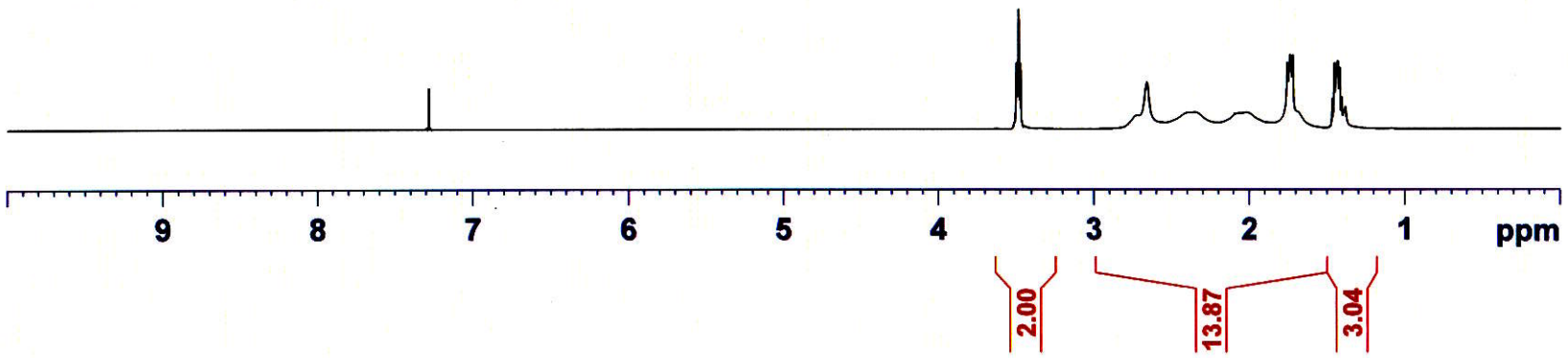
IBB-nmr Analysis



NAME	201001-p-carborane-CH2CH2CH2OH
EXPNO	1
PROCNO	1
Date_	20110707
Time	10.59
INSTRUM	av500
PROBHD	5 mm CPDCH 13C
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	4
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	14.3
DW	48.400 usec
DE	6.00 usec
TE	298.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	10.00 usec
PL1	-4.90 dB
PL1W	7.76247120 W
SFO1	500.1330885 MHz
SI	32768
SF	500.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	0.50

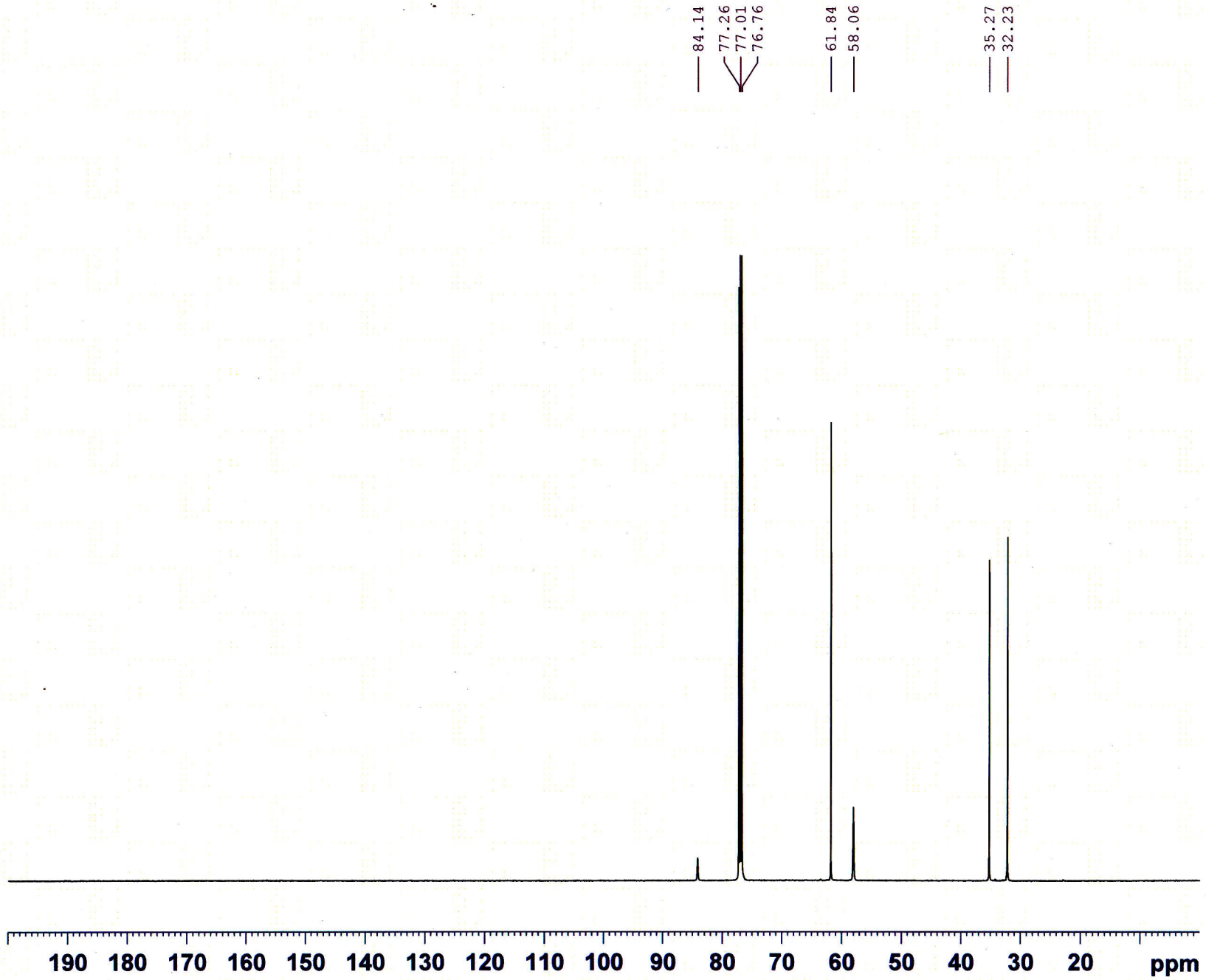


13



Carborane-VDR

IBB-nmr Analysis

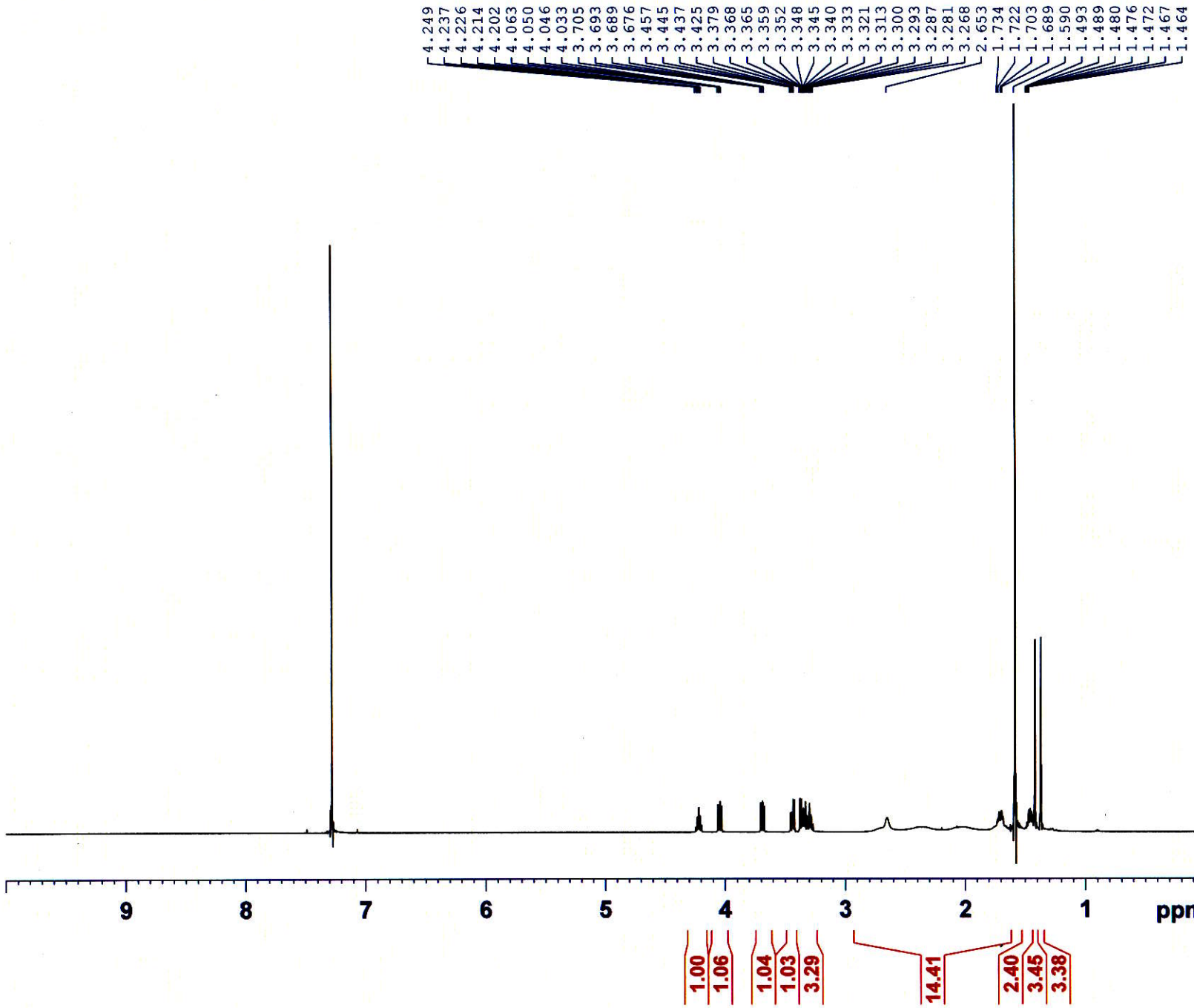


NAME	201001-p-carborane-CH2CH2CH2OH-13C
EXPNO	1
PROCNO	1
Date_	20110707
Time	11.03
INSTRUM	av500
PROBHD	5 mm CPDCH 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	560
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	90.5
DW	16.650 usec
DE	20.00 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	10.00 usec
PL1	-4.80 dB
PL1W	13.65439701 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-4.80 dB
PL12	12.80 dB
PL13	16.00 dB
PL2W	7.58577585 W
PL12W	0.13182567 W
PL13W	0.06309573 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577932 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	0.30

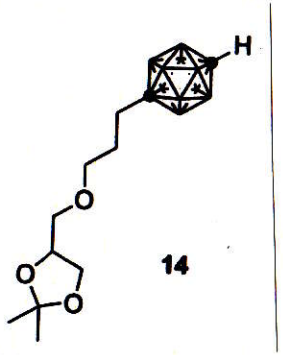
13

carborane-VDR

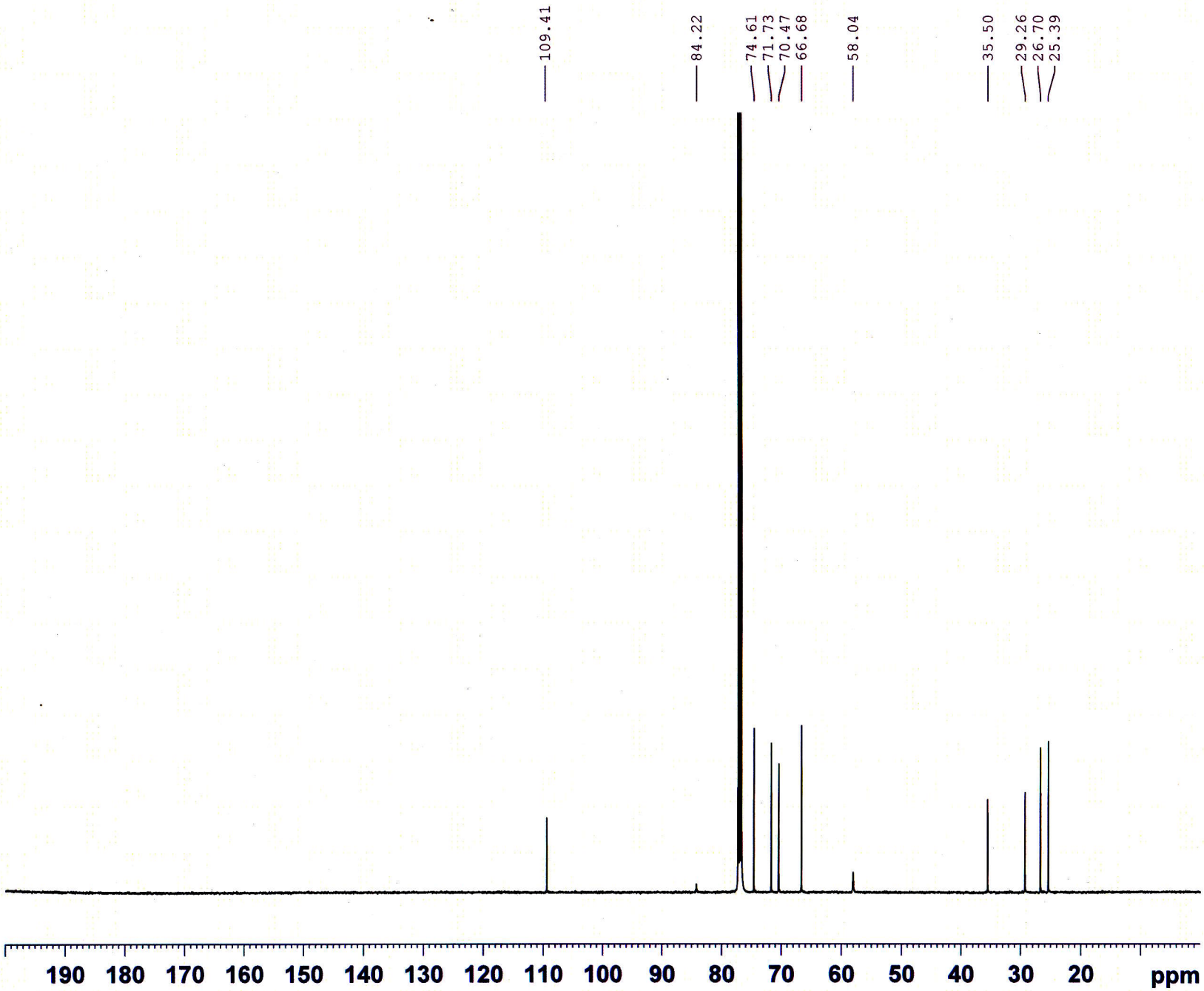
IBB-nmr Analysis



NAME	201001-071-clm-carborane
EXPNO	1
PROCNO	1
Date_	20110723
Time	15.20
INSTRUM	av500
PROBHD	5 mm CPDCH 13C
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	14.3
DW	48.400 usec
DE	6.00 usec
TE	298.0 K
D1	1.00000000 sec
TD0	1
========	CHANNEL f1 ========
NUC1	1H
P1	10.00 usec
PL1	-4.90 dB
PL1W	7.76247120 W
SFO1	500.1330885 MHz
SI	32768
SF	500.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	0.50



carborane-VDR



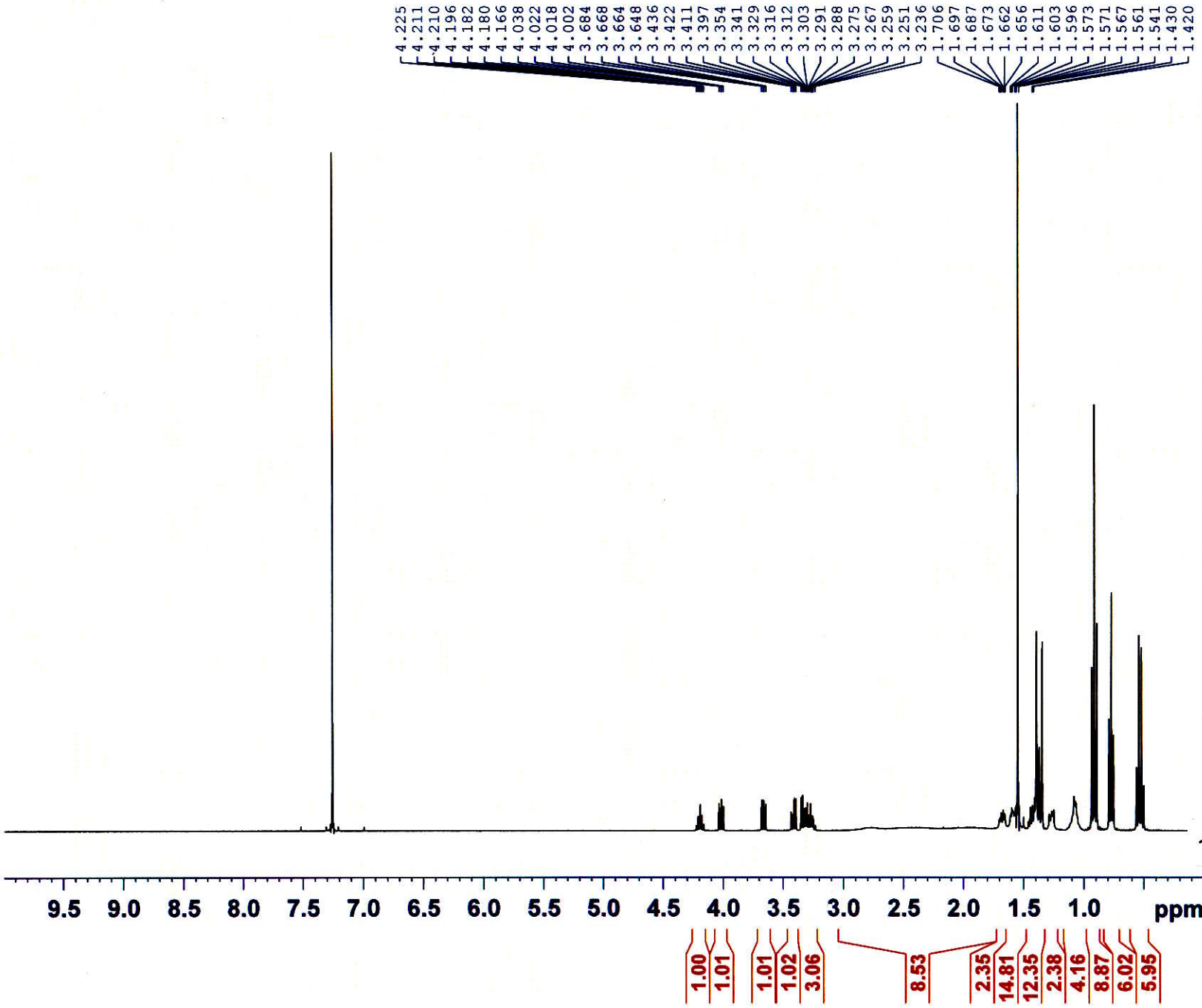
IBB-nmr Analysis

NAME	201001-071-clm-carborane-13C	
EXPNO	1	
PROCNO	1	
Date_	20110728	
Time	12.28	
INSTRUM	av500	
PROBHD	5 mm CPDCH 13C	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	256	
DS	4	
SWH	30030.029	Hz
FIDRES	0.458222	Hz
AQ	1.0912410	sec
RG	90.5	
DW	16.650	usec
DE	20.00	usec
TE	298.0	K
D1	2.00000000	sec
D11	0.03000000	sec
TD0	1	
========	CHANNEL f1	========
NUC1	13C	
P1	10.00	usec
PL1	-4.80	dB
PL1W	13.65439701	W
SFO1	125.7703643	MHz
========	CHANNEL f2	========
CPDPRG2	waltz16	
NUC2	1H	
PCPD2	80.00	usec
PL2	-4.80	dB
PL12	12.80	dB
PL13	16.00	dB
PL2W	7.58577585	W
PL12W	0.13182567	W
PL13W	0.06309573	W
SFO2	500.1320005	MHz
SI	32768	
SF	125.7577932	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	0.30	

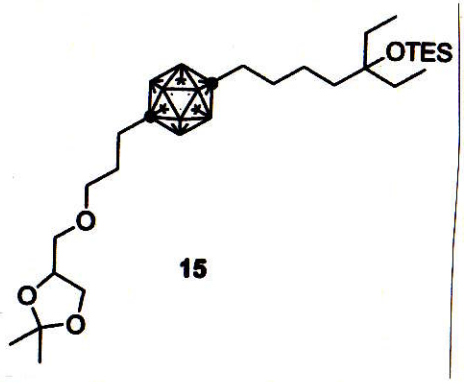
14

carborane-VDR

IBB-nmr Analysis



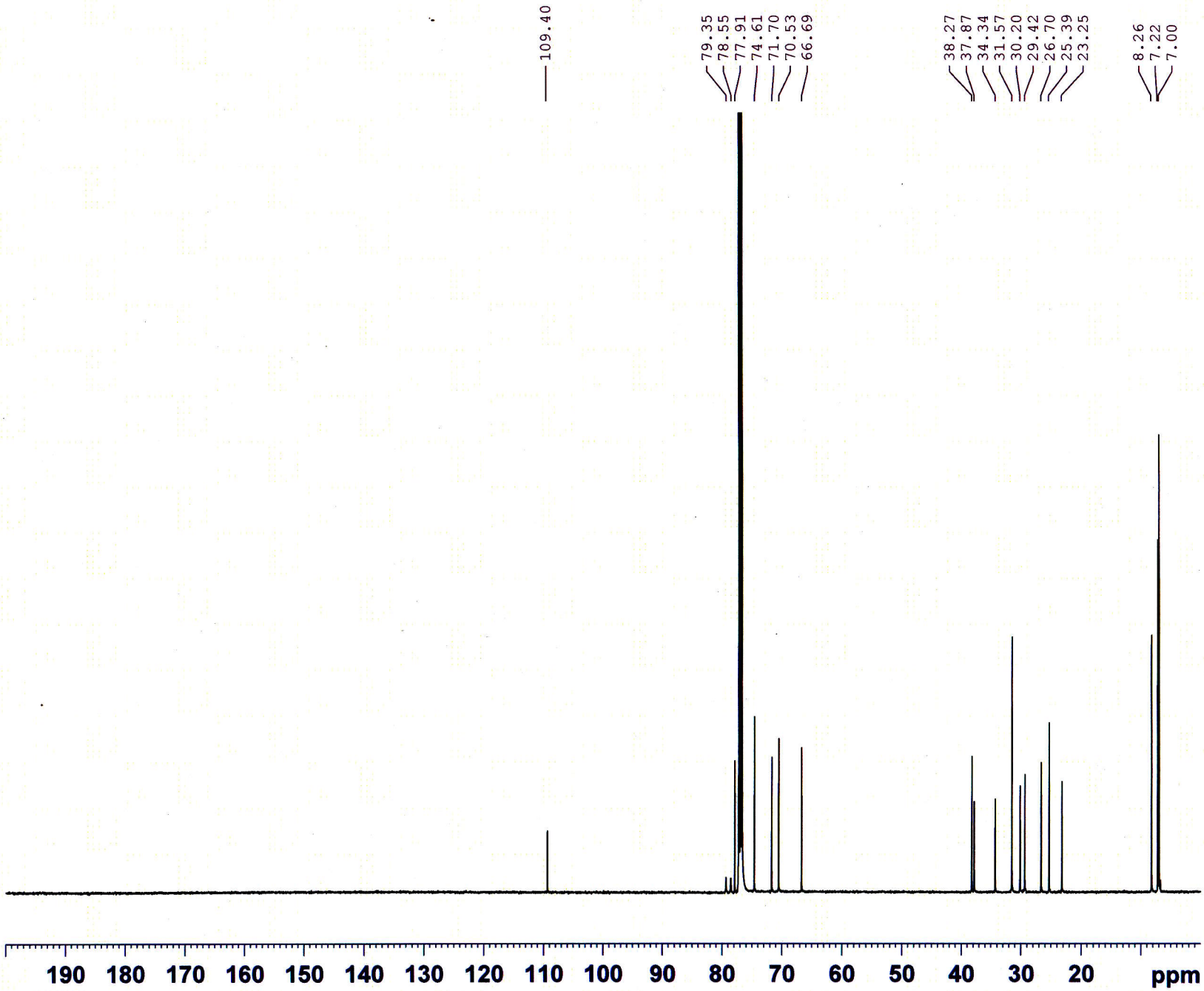
NAME	201001-072-clm-carborane
EXPNO	1
PROCNO	1
Date_	20110802
Time	13.54
INSTRUM	av400
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	203
DW	60.800 usec
DE	6.50 usec
TE	3007.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.00 usec
PL1	-1.80 dB
PL1W	14.82738590 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300083 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



15

carborane-VDR

IBB-nmr Analysis

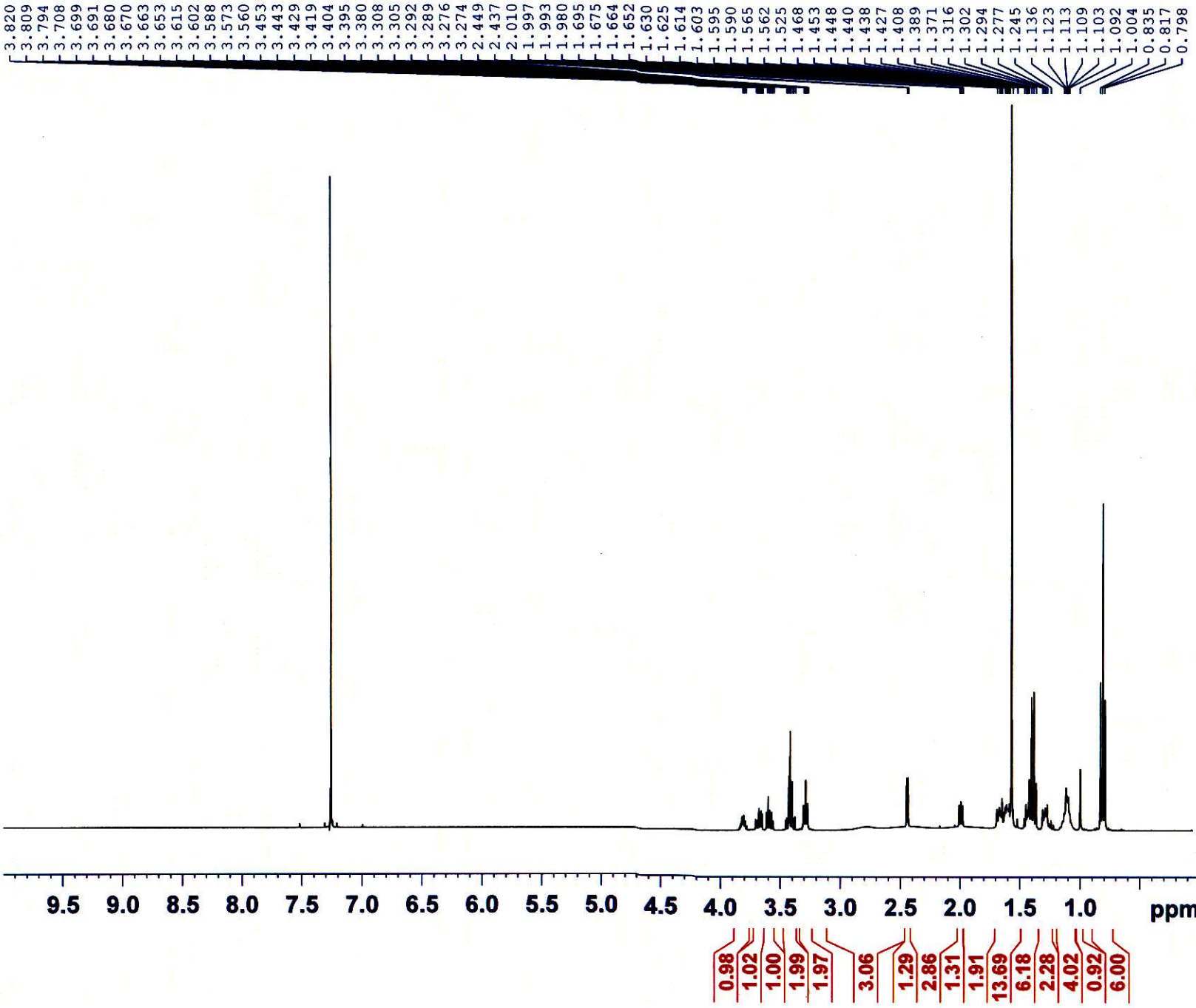


NAME	201001-072-clm-13C-carborane
EXPNO	1
PROCNO	1
Date_	20110802
Time	14.29
INSTRUM	av500
PROBHD	5 mm CPDCH 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	512
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	90.5
DW	16.650 usec
DE	20.00 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	10.00 usec
PL1	-4.80 dB
PL1W	13.65439701 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-4.80 dB
PL12	12.80 dB
PL13	16.00 dB
PL2W	7.58577585 W
PL12W	0.13182567 W
PL13W	0.06309573 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577932 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	0.30

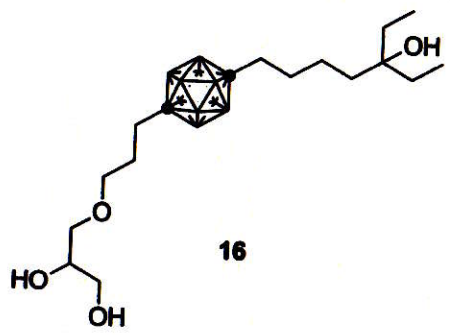
15

carborane-VDR

IBB-nmr Analysis



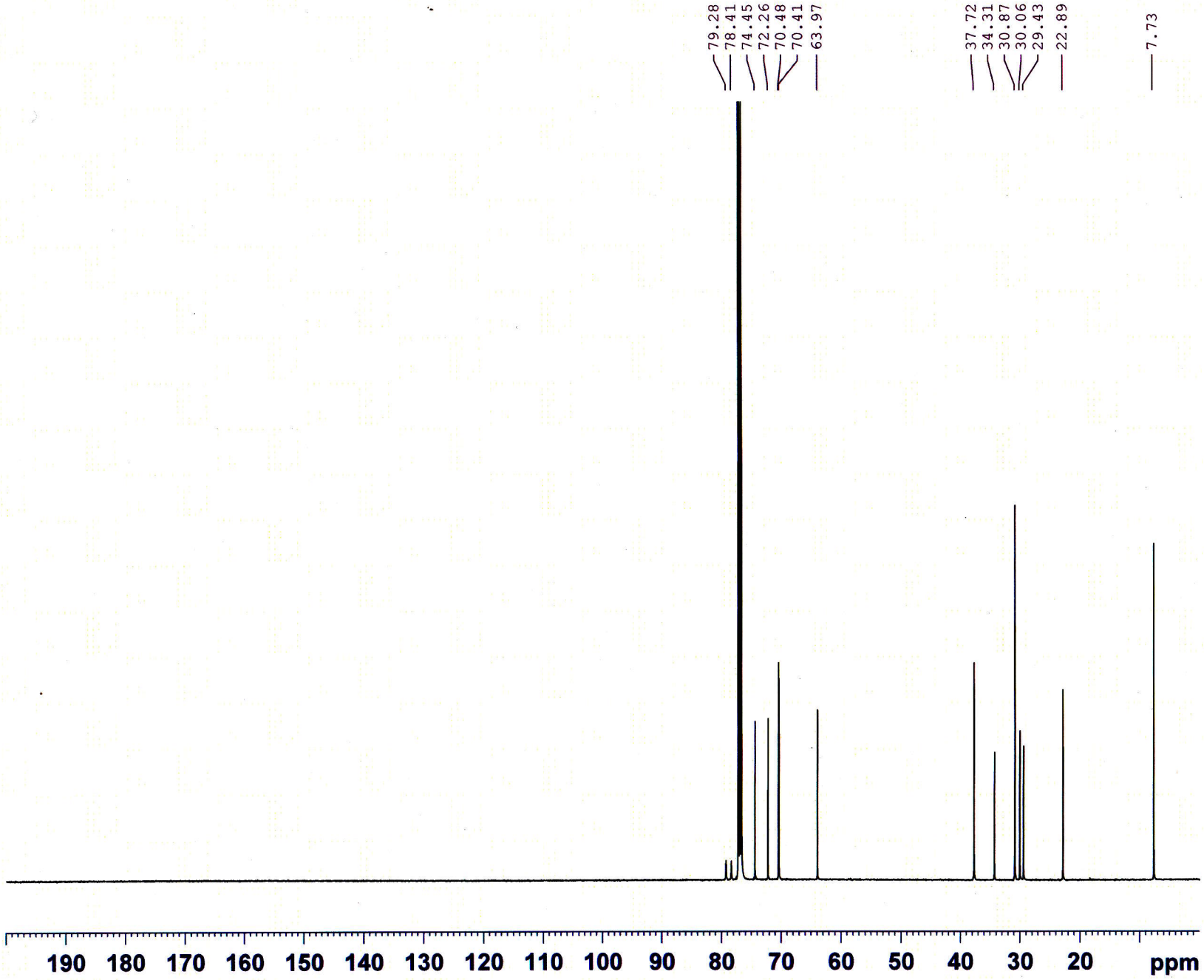
NAME	201001-073-clm-carborane
EXPNO	1
PROCNO	1
Date_	20110804
Time	11.26
INSTRUM	av400
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	203
DW	60.800 usec
DE	6.50 usec
TE	3008.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.00 usec
PL1	-1.80 dB
PL1W	14.82738590 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300083 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



16

carborane-VDR

IBB-nmr Analysis



NAME	201001-073-clm-13C-carborane
EXPNO	1
PROCNO	1
Date_	20110804
Time	12.41
INSTRUM	av500
PROBHD	5 mm CPDCH 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	448
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	90.5
DW	16.650 usec
DE	20.00 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	10.00 usec
PL1	-4.80 dB
PL1W	13.65439701 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-4.80 dB
PL12	12.80 dB
PL13	16.00 dB
PL2W	7.58577585 W
PL12W	0.13182567 W
PL13W	0.06309573 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577932 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	0.30

16