

Electronic Supplementary Material (ESI) for *Organic & Biomolecular Chemistry*  
This journal is (c) The Royal Society of Chemistry 2015

## Supplementary Information for

# Direct construction of 2-alkylbenzo-1,3-azoles via C-H activation of alkanes for C-C and C-X (X = O, S) bond formation

Arvind K. Yadav and Lal Dhar S. Yadav\*

*Green Synthesis Lab, Department of Chemistry, University of Allahabad,*

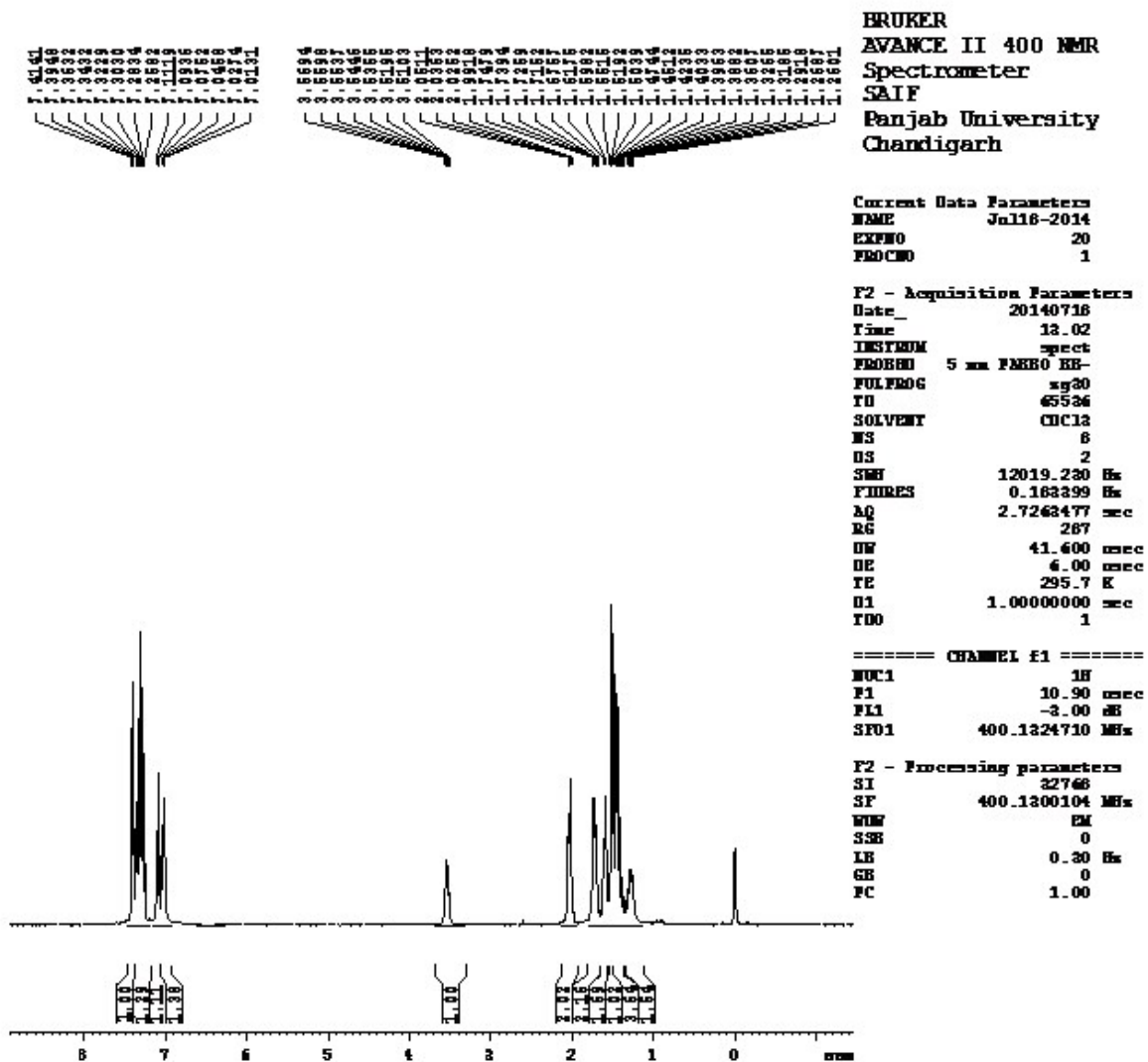
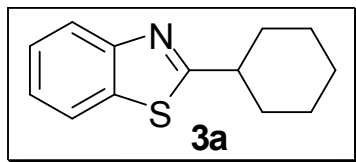
*Allahabad-211002, India*

*E-mail: ldsyadav@hotmail.com*

- I. General Information:** All commercially available reagents were used without further purification unless otherwise specified by a reference. Solvent was purified by the usual methods and stored over molecular sieves. All reactions were performed using oven-dried glassware under a nitrogen atmosphere. Organic solutions were concentrated using a Buchi rotary evaporator. Column chromatography was carried out over silica gel (Merck 100–200 mesh) and TLC was performed using silica gel GF254 (Merck) plates. Melting point (m.p.) was determined by open glass tube. <sup>1</sup>H (400 MHz), <sup>13</sup>C (100 MHz) NMR spectra were recorded on a Bruker AVII spectrometer in CDCl<sub>3</sub> using TMS as internal reference. All chemical shifts are reported in δ/ppm and coupling constants (*J*) in Hertz (Hz). MS (EI) spectra were recorded double focusing mass spectrometer.
- II. General procedure for the synthesis of 2-alkylbenzo-1,3-azoles:** Charged a round bottom flask with aryl isothiocyanate/isocyanate **1** (1.0 mmol), alkane **2** (1.0 mL), DTBP (4.0 equiv.), Cu(OAc)<sub>2</sub> (10 mol%) and CH<sub>3</sub>CN (3 mL). The contents were stirred at 120 °C under a nitrogen atmosphere for 6-12 h. After the completion of reaction (as indicated by TLC), it was quenched with water (10 mL) and extracted with ethyl acetate (3 × 10 mL). The organic phase was dried over anhydrous magnesium sulfate and concentrated under reduced pressure to yield the crude product, which was purified by silica gel column chromatography using a mixture of EtOAc-Hexane (1:9) to give the pure product **3** in high yields (Table 2). The structure of the products was confirmed by comparison of <sup>1</sup>H, <sup>13</sup>C NMR and MS spectral data with those reported in the literature.<sup>1</sup>

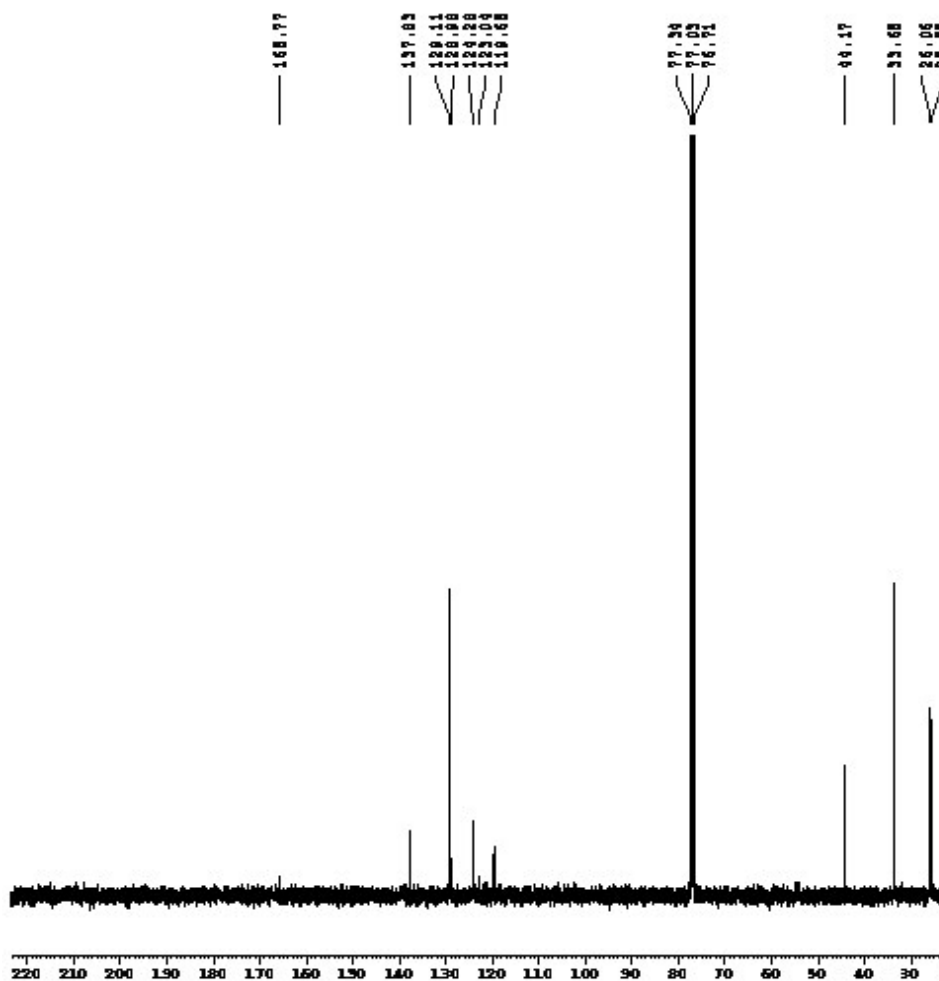
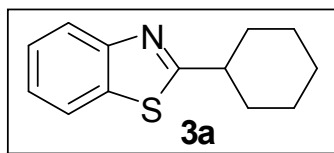
### III. Copies of $^1\text{H}$ and $^{13}\text{C}$ NMR spectra.

Compound 3a.  $^1\text{H}$  NMR Spectrum ( $\text{CDCl}_3$ ).



avtar saifpu@yahoo.co.in

Compound 3a.  $^{13}\text{C}$  NMR Spectrum ( $\text{CDCl}_3$ ).



BRUKER  
 AVANCE II 400 NMR  
 Spectrometer  
 SAIF  
 Panjab University  
 Chandigarh

Current Data Parameters  
 NAME Sep16-2014  
 EXPNO 240  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20140916  
 Time 16.42  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 512  
 DS 4  
 SSB 29761.904 Hz  
 FIDRES 0.454131 Hz  
 AQ 1.1010546 sec  
 RG 2050  
 DF 16.800 msec  
 DE 6.00 msec  
 TE 297.9 K  
 H1 2.0000000 sec  
 H11 0.0300000 sec  
 DELTA 1.6999996 sec  
 TD0 1

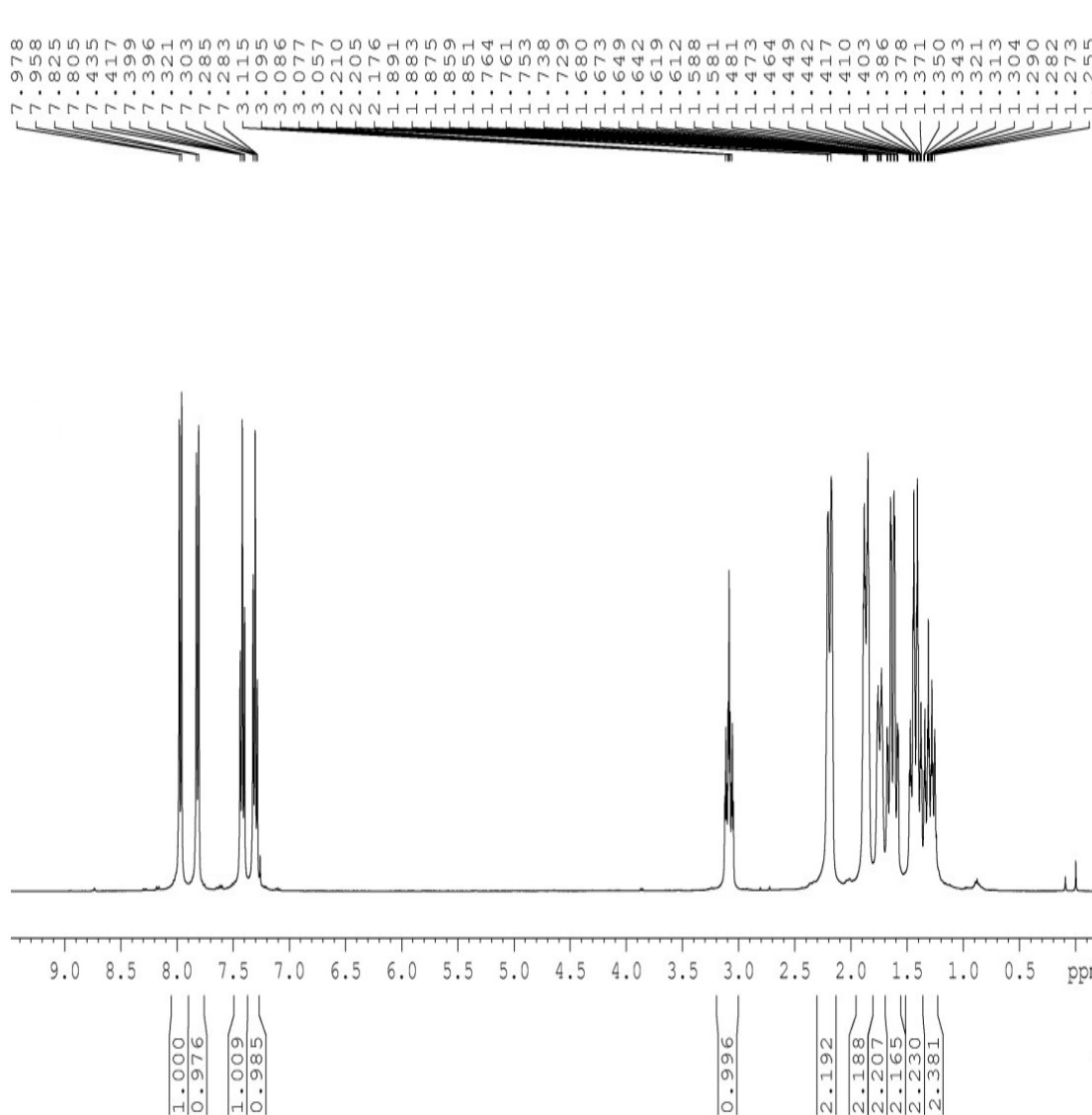
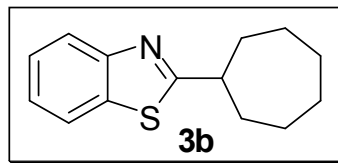
==== CHANNEL F1 =====  
 NUC1 13C  
 P1 9.60 msec  
 PL1 -2.00 dB  
 SFO1 100.6226296 MHz

==== CHANNEL F2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 60.00 msec  
 PL2 -3.00 dB  
 PL12 14.31 dB  
 PL13 18.00 dB  
 SFO2 400.1316005 MHz

F2 - Processing parameters  
 SI 32768  
 SF 100.6127690 MHz  
 GM EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

avtar.saifou@yahoo.co.in

Compound 3b. <sup>1</sup>H NMR Spectrum (CDCl<sub>3</sub>).



**BRUKER**  
**AVANCE II 400 NMR**  
**Spectrometer**  
**SAIF**  
**Panjab University**  
**Chandigarh**

Current Data Parameters  
**NAME** Jul16-2014  
**EXPNO** 20  
**PROCNO** 1

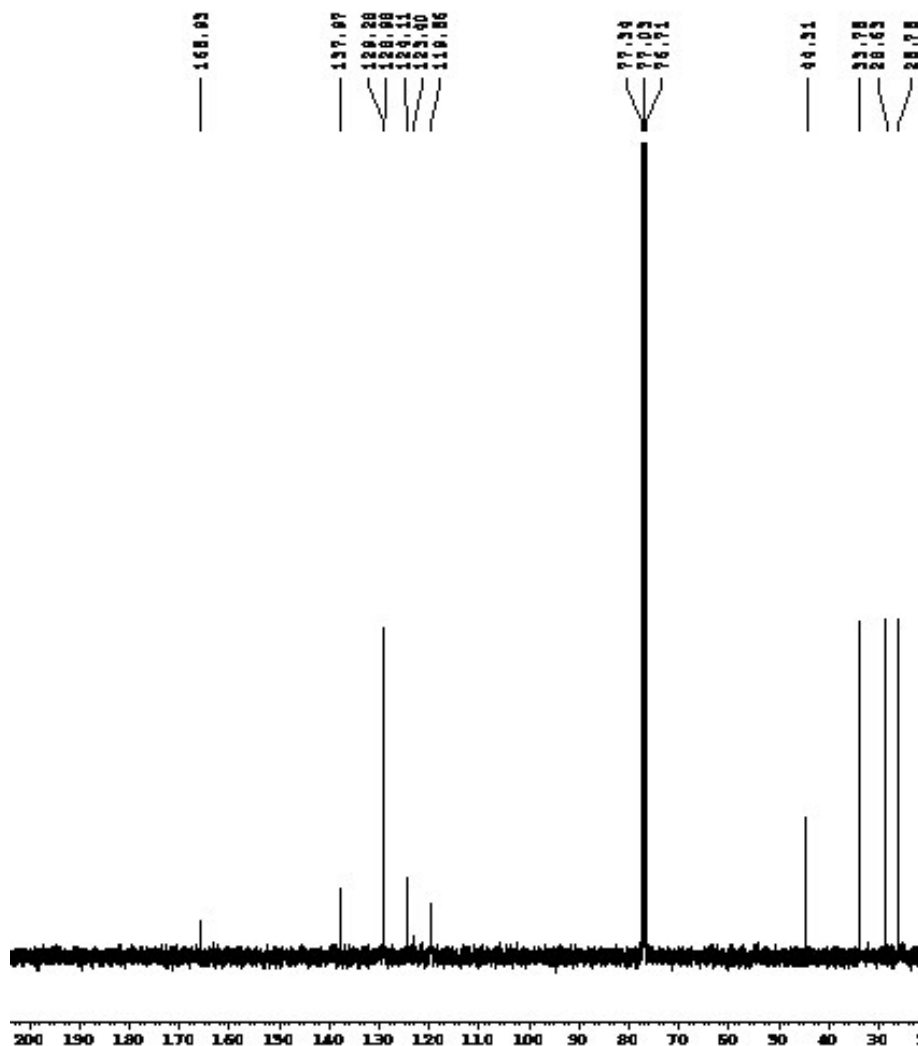
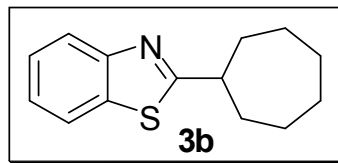
F2 - Acquisition Parameters  
**Date\_** 20140711  
**Time** 11.02  
**INSTRUM** spect  
**PROBHD** 5 mm PABBO BB-  
**PULPROG** zg30  
**TD** 65536  
**SOLVENT** CDCl3  
**NS** 8  
**DS** 2  
**SWH** 12019.230 Hz  
**FIDRES** 0.168339 Hz  
**AQ** 2.7263477 sec  
**RG** 267  
**DF** 41.600 sec  
**DE** 6.00 sec  
**TE** 295.7 K  
**D1** 1.00000000 sec  
**TD0** 1

==== CHANNEL f1 =====  
**NUC1** 1H  
**F1** 10.90 use  
**FI1** -2.00 dB  
**SFO1** 400.1324710 MHz

F2 - Processing parameters  
**SI** 32768  
**SP** 400.1300104 MHz  
**WDW** EM  
**SSB** 0  
**LB** 0.30 Hz  
**GB** 0  
**PC** 1.00

avtar saifpu@yahoo.co.in

Compound 3b.  $^{13}\text{C}$  NMR Spectrum ( $\text{CDCl}_3$ ).



**BROKER**  
**AVANCE II 400 NMR**  
**Spectrometer**  
**SAIF**  
**Panjab University**  
**Chandigarh**

Current Data Parameters  
 NAME Sep18-2014  
 EXPNO 240  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20140718  
 Time\_ 15.42  
 INSTRUM spect  
 PROBRW 5 mm PABBO BB-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 512  
 DS 4  
 SSB 29761.904 Hz  
 FIDRES 0.434131 Hz  
 AQ 1.1010548 sec  
 EC 2050  
 DM 15.800 msec  
 DE 6.00 msec  
 TE 297.9 K  
 DL 2.00000000 sec  
 ALL 0.03000000 sec  
 DELTA 1.89999998 sec  
 TDO 1

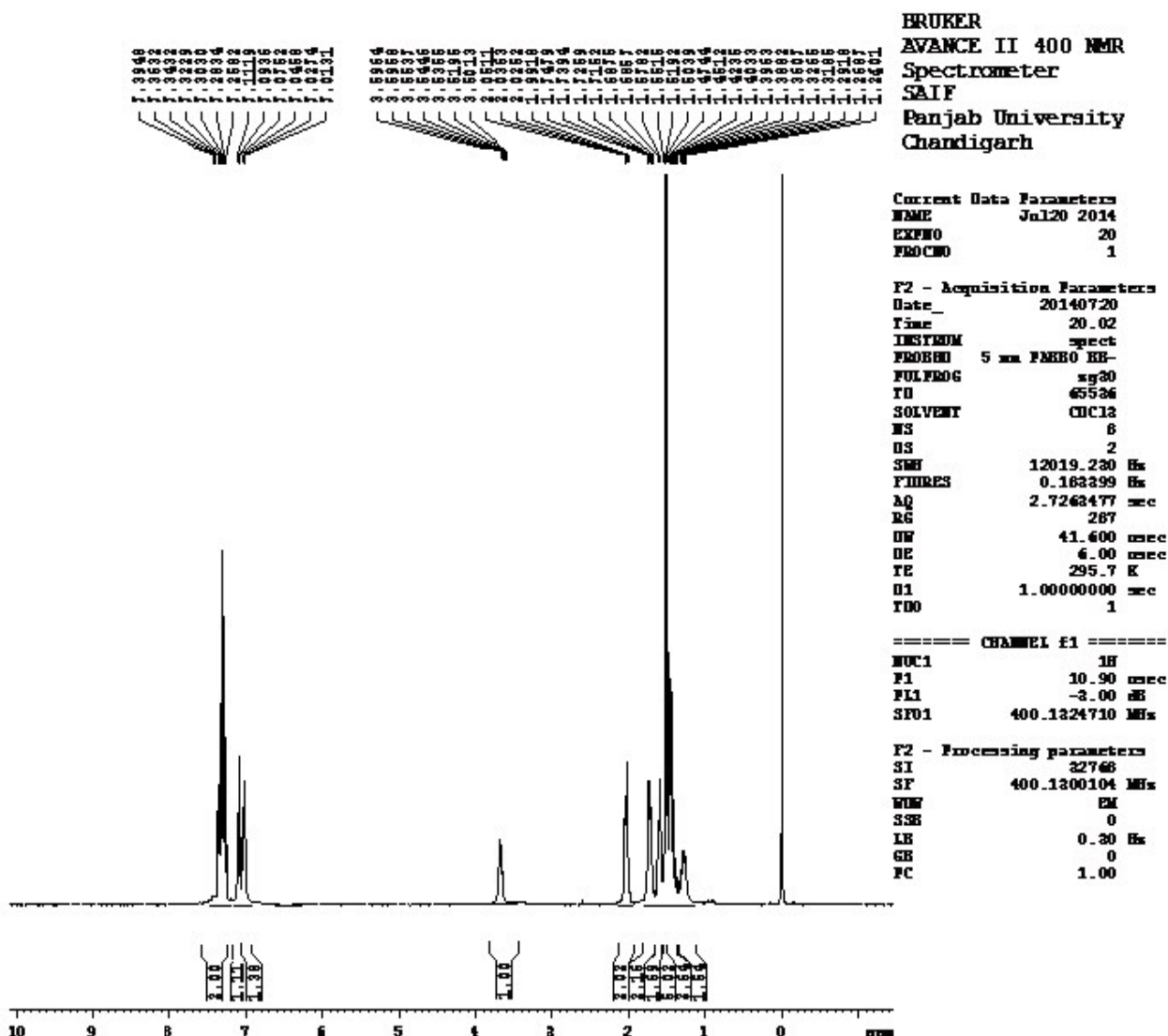
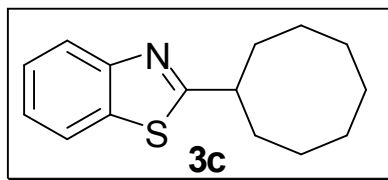
CHANNEL F1  
 NUC1 13C  
 P1 9.50 msec  
 PL1 -2.00 dB  
 SFO1 100.6228298 MHz

CHANNEL F2  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 80.00 msec  
 PL2 -3.00 dB  
 PL12 14.31 dB  
 PL13 18.00 dB  
 SFO2 400.1316005 MHz

F2 - Processing parameters  
 SI 32768  
 SF 100.6127690 MHz  
 DS 4  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 EC 1.40

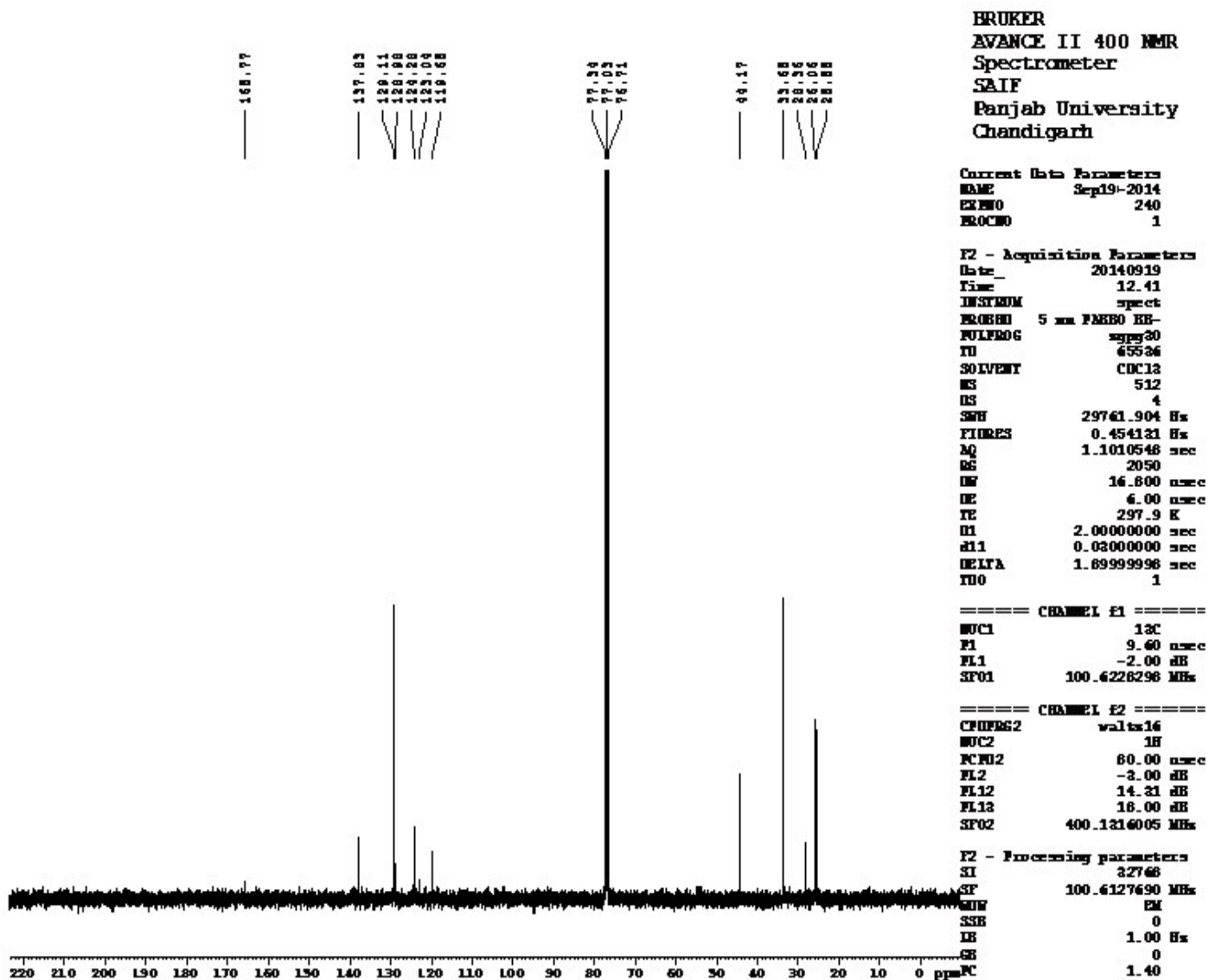
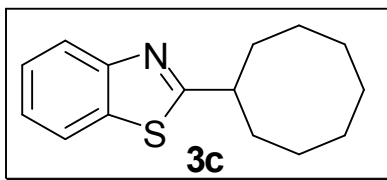
avtar saifon@yahoo.co.in

Compound 3c. <sup>1</sup>H NMR Spectrum (CDCl<sub>3</sub>).



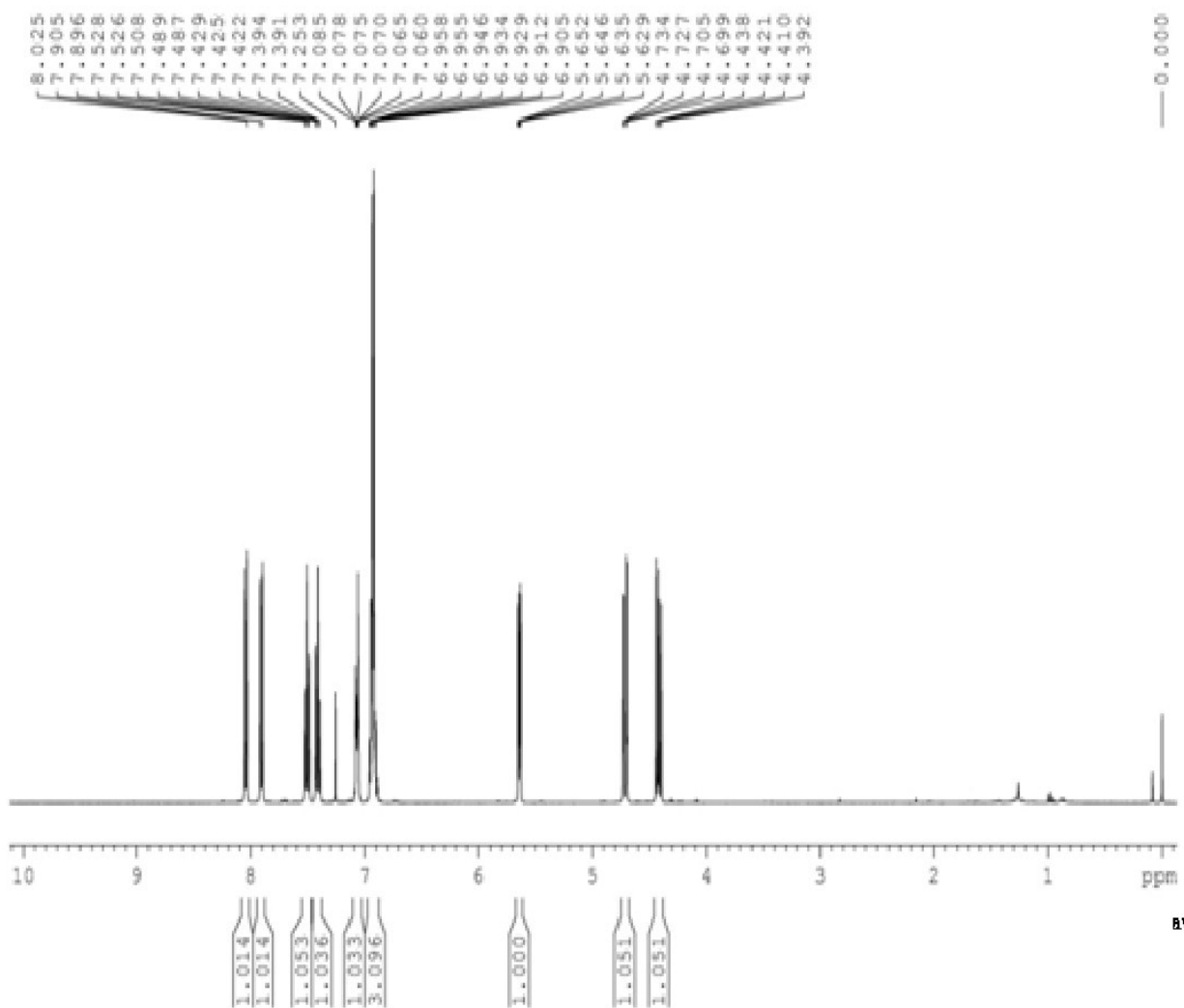
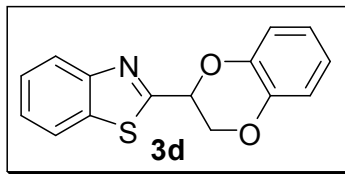
avtar saifpu@yahoo.co.in

Compound 3c. <sup>13</sup>C NMR Spectrum (CDCl<sub>3</sub>).



avtar saifpu@yahoo.co.in

Compound 3d. <sup>1</sup>H NMR Spectrum (CDCl<sub>3</sub>).



BRUKER  
 AVANCE II 400  
 Spectrometer  
 SAIF  
 Panjab University  
 Chandigarh

Current Data Parameters  
 NAME Jul12  
 EXPR0  
 PROC00

F2 - Acquisition Parameters  
 Date\_ 2012  
 Time  
 INSTRUM  
 PROBR0 5 mm TMRB  
 PULPROG  
 ID  
 SOLVENT  
 NS  
 DS  
 SWH 12013  
 FIDRES 0.1  
 AQ 2.72  
 RG  
 DF  
 DE  
 IE  
 D1 1.000  
 TDO

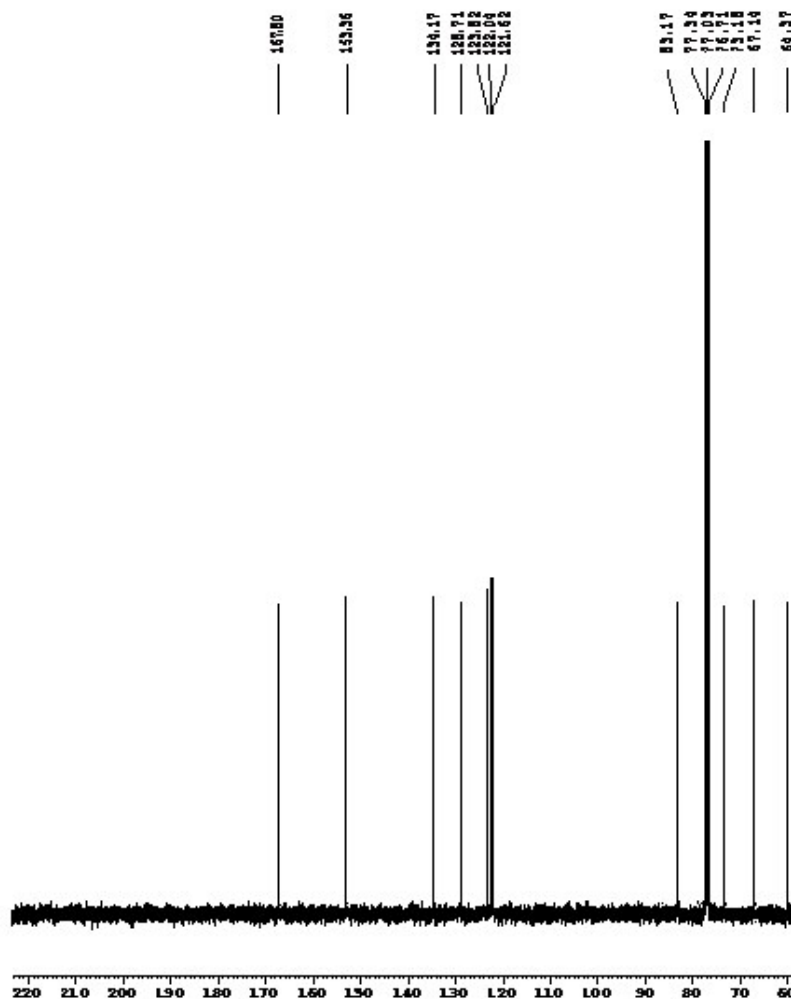
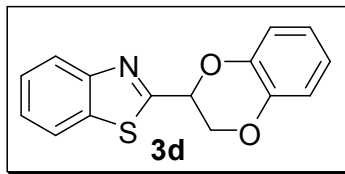
----- CHANNEL f1  
 NUC1  
 P1  
 PL1  
 SFO1 400.13

F2 - Processing parameters  
 SI  
 SF 400.13  
 WDW  
 SSB  
 LB  
 GB  
 EC

avtar saifpu@yahoo.co.



Compound 3d. <sup>13</sup>C NMR Spectrum (CDCl<sub>3</sub>).



BRUKER  
 AVANCE II 400 NMR  
 Spectrometer  
 SAIF  
 Panjab University  
 Chandigarh

Current Data Parameters  
 NAME Sep12-2014  
 EXNO 240  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20140912  
 Time 12.19  
 INSTRUM spect  
 PROBHD 5 mm PBEBO BB-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 512  
 DS 4  
 SSB 29741.904 Hz  
 FIDRES 0.454121 Hz  
 AQ 1.1010548 sec  
 RG 2050  
 DW 16.600 msec  
 DE 6.00 msec  
 TE 297.9 K  
 D1 2.0000000 sec  
 d11 0.0200000 sec  
 DELTA 1.6999998 sec  
 TD0 1

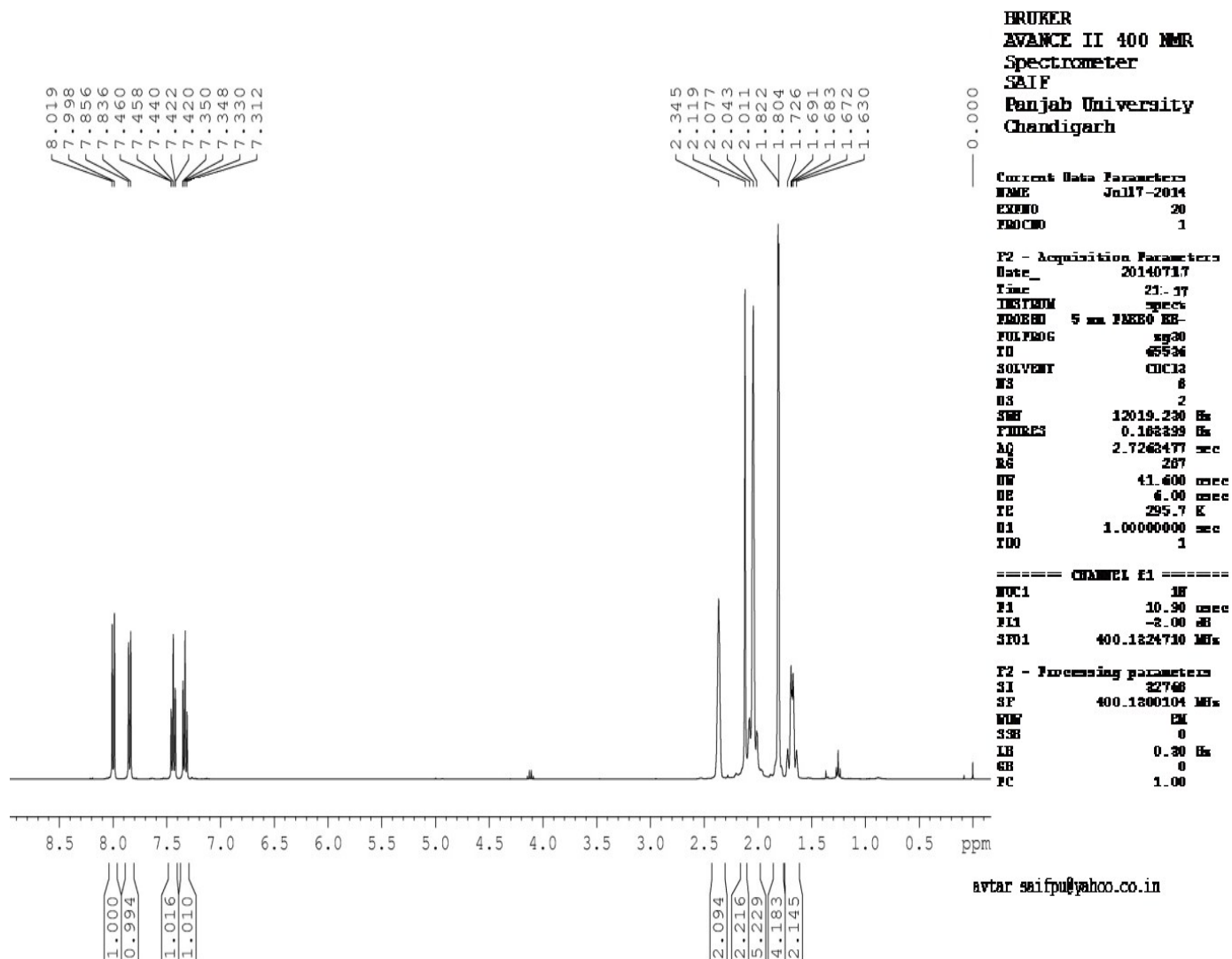
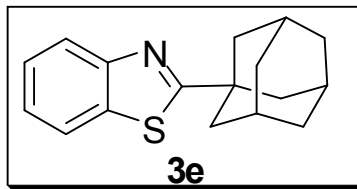
==== CHANNEL F1 =====  
 NUC1 13C  
 P1 9.60 msec  
 PL1 -2.00 dB  
 SF01 100.6228298 MHz

==== CHANNEL F2 =====  
 CHPROG2 waltz16  
 NUC2 1H  
 PCPD2 60.00 msec  
 PL2 -3.00 dB  
 PL12 14.31 dB  
 PL13 18.00 dB  
 SF02 400.1216005 MHz

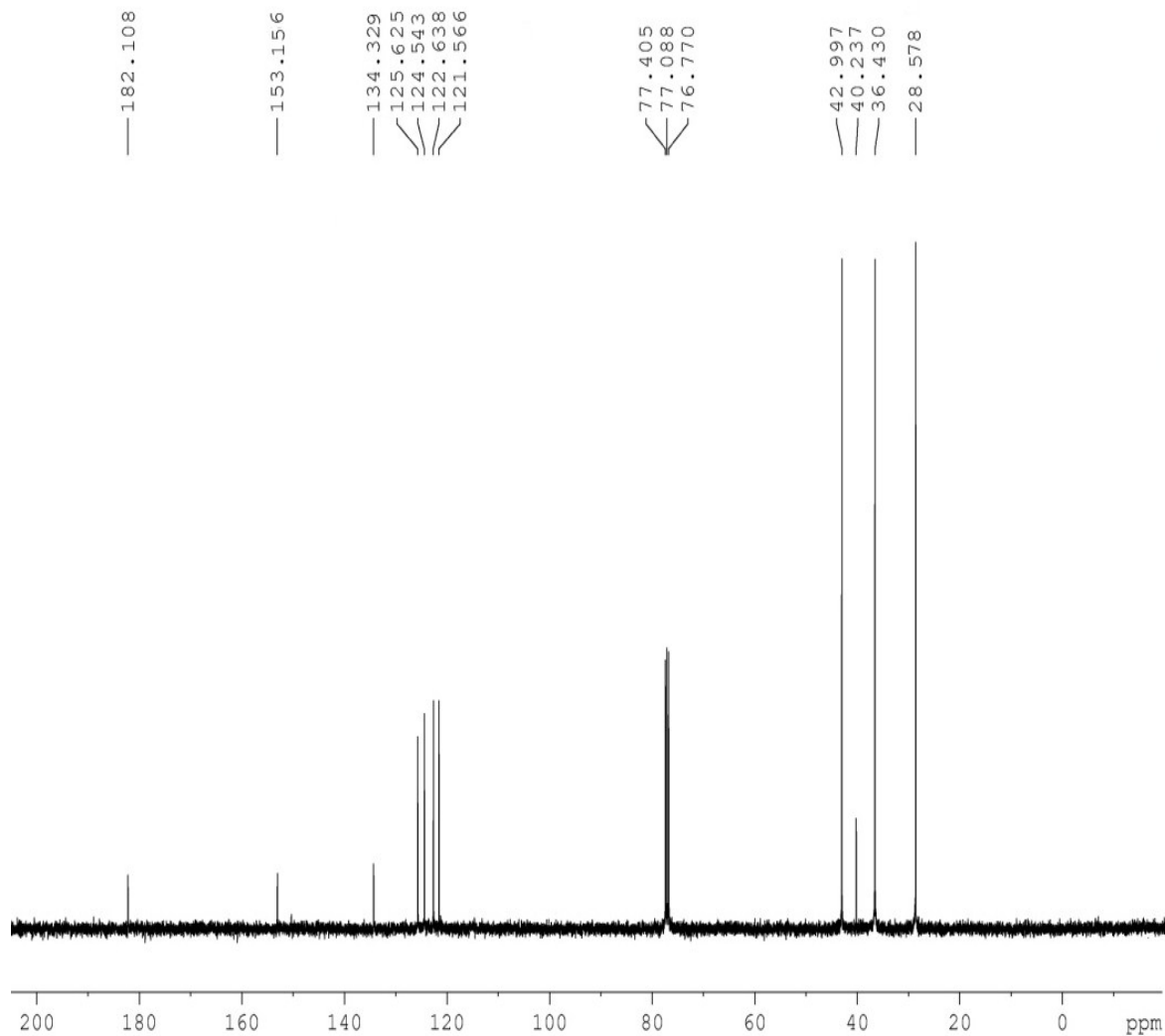
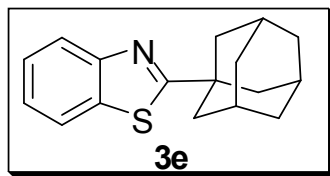
F2 - Processing parameters  
 SI 32768  
 SF 100.6127690 MHz  
 SSB EM  
 LB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

avtar saifpu@yahoo.co.in

Compound 3e. <sup>1</sup>H NMR Spectrum (CDCl<sub>3</sub>).



Compound 3e.  $^{13}\text{C}$  NMR Spectrum ( $\text{CDCl}_3$ ).



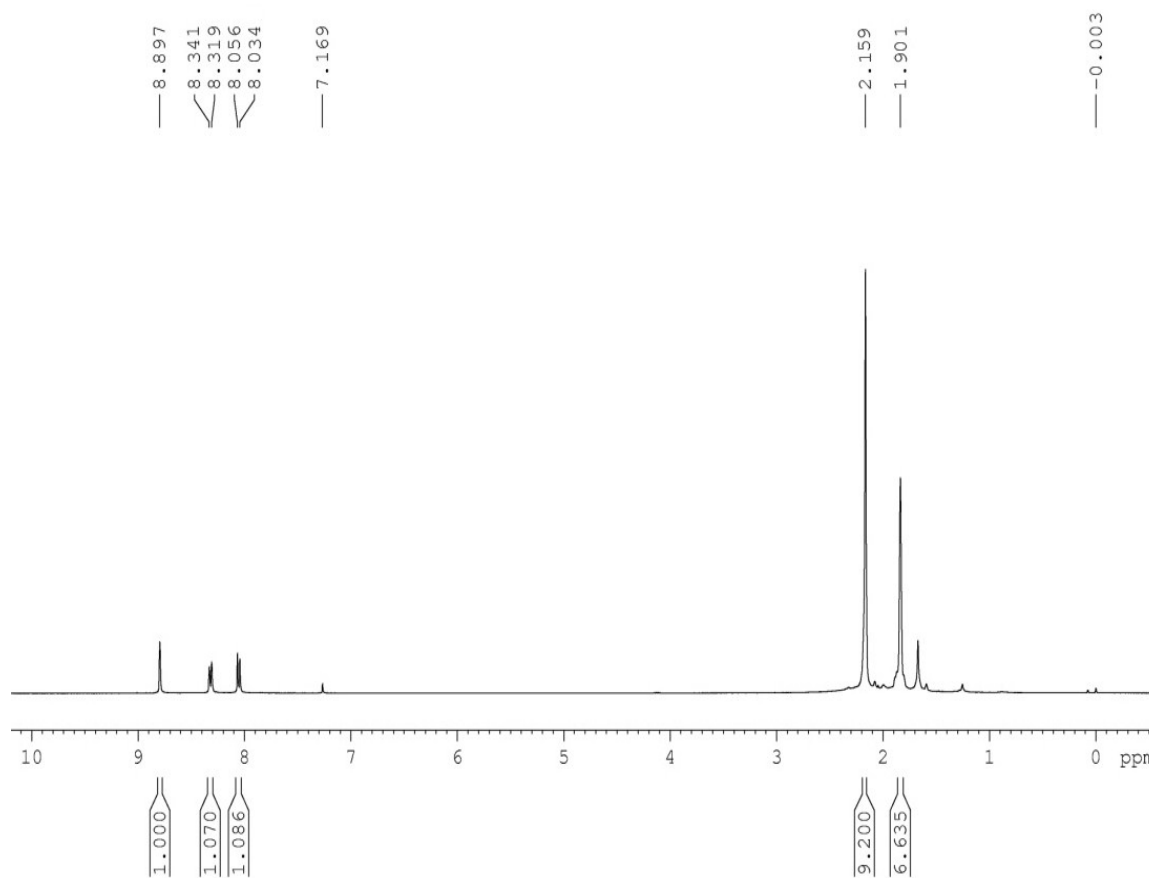
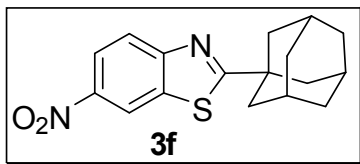
**BRUKER**  
**AVANCE II 400**  
**Spectrometer**  
**SAIF**  
**Panjab Univer**  
**Chandigarh**

Current Data Parameters  
**NAME** Sep16-  
**EXPNO**  
**PROCNO**  
**F2 - Acquisition Parameters**  
**Date\_** 2014  
**Time** 2  
**INSTRUM** zgpg30  
**PROBHD** 5 mm BBOBO  
**PULPROG** zgpg30  
**TD** 65536  
**SOLVENT** CDCl3  
**MS**  
**DS**  
**SWH** 29761.2  
**FIDRES** 0.45  
**AQ** 1.101  
**RG**  
**DE** 1.6  
**DB**  
**TE** 298.2  
**OL** 2.00000  
**ILL** 0.03000  
**DELTA** 1.89999  
**TD0**

**CHANNEL F1**  
**NUC1**  
**F1**  
**P1**  
**SP01** 100.622  
**CHANNEL F2**  
**NAME F2**  
**NUC2**  
**F2**  
**P2**  
**F1 F2**  
**F1 F2**  
**F1 F2**  
**F1 F2**  
**SP02** 400.131  
**F2 - Processing parameters**  
**SI**  
**SF** 100.612  
**GB**  
**GB**  
**GB**  
**GB**

avtar saifonvahoo.

Compound 3f. <sup>1</sup>H NMR Spectrum (CDCl<sub>3</sub>).



BRUKER  
 AVANCE II 400 NMR  
 Spectrometer  
 SAIF  
 Panjab University  
 Chandigarh

Current Data Parameters  
 NAME Jnl12-2014  
 EXNO 20  
 PROCNO 1

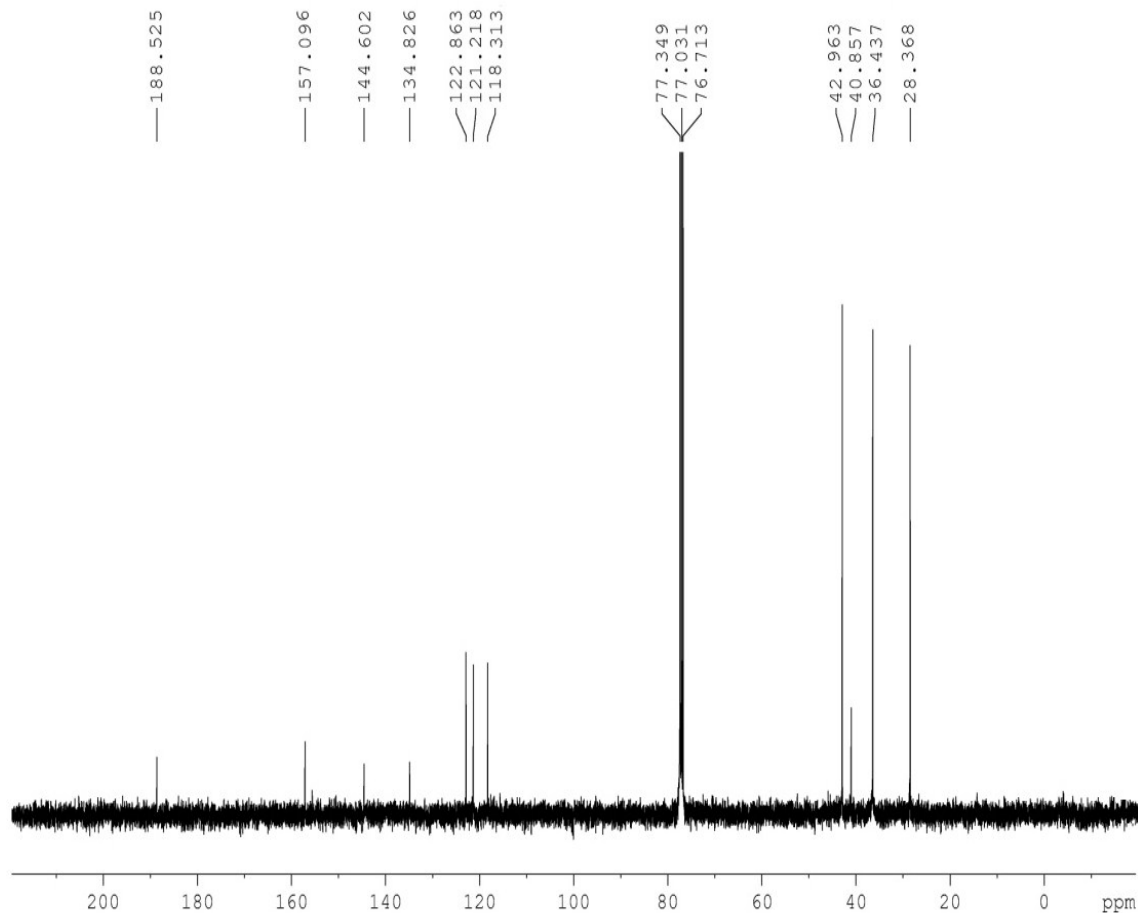
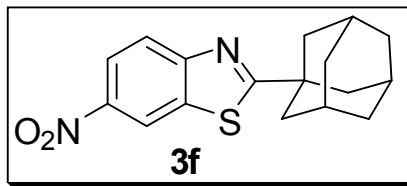
F2 - Acquisition Parameters  
 Date\_ 20140712  
 Time 21.14  
 INSTRUM spect  
 F2PROG 5 nu FREQ0 EE-  
 PULPROG zg30  
 ID 45534  
 SOLVENT CDCl3  
 NS 8  
 DS 2  
 SSB 12019.230 Hz  
 FIDRES 0.168389 Hz  
 AQ 2.7263477 sec  
 RG 287  
 HF 41.600 msec  
 HE 6.00 msec  
 FE 295.7 K  
 D1 1.0000000 sec  
 TD0 1

===== CHANNEL F1 =====  
 NUC1 1H  
 P1 10.90 msec  
 PL1 -2.00 dB  
 SFO1 400.1224710 MHz

F2 - Processing parameters  
 SI 32768  
 SF 400.1200104 MHz  
 WDF 0  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

avtar saifpu@yahoo.co.in

Compound 3f. <sup>13</sup>C NMR Spectrum (CDCl<sub>3</sub>).



**BROKER**  
**AVANCE II 400 MHz**  
**Spectrometer**  
**SAIF**  
**Panjab University**  
**Chandigarh**

Current Data Parameters  
**NAME** Sep18-2014  
**EXPNO** 240  
**PROCNO** 1

**F2 - Acquisition Parameters**  
**Date\_** 20140718  
**Time** 15.40  
**INSTRUM** spect  
**PROBHD** 5 mm QNP90 BBO  
**PULPROG** zgpg30  
**TD** 65536  
**SOLVENT** CDCl3  
**NS** 512  
**DS** 4  
**SWH** 29761.904  
**FIDRES** 0.434131  
**AQ** 1.1010546  
**RG** 2096  
**OR** 15.800  
**DE** 5.00  
**TE** 297.3  
**D1** 2.00000000  
**d11** 0.03000000  
**DELTA** 1.83333333  
**TD0** 1

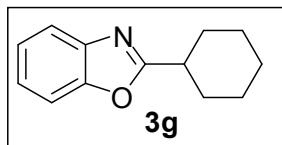
**CHANNEL F1**  
**NUC1** 13C  
**FL** 9.90  
**PL1** -2.00  
**RF01** 100.6228230

**CHANNEL F2**  
**CPDPRG2** waltz16  
**NUC2** 1H  
**PCPD2** 80.00  
**PL2** -3.00  
**PL12** 14.30  
**PL13** 18.00  
**RF02** 400.1316000

**F2 - Processing parameters**  
**SI** 32768  
**SF** 100.6127690  
**WDW** EM  
**SSB** 0  
**LB** 1.00  
**GB** 0  
**PC** 1.40

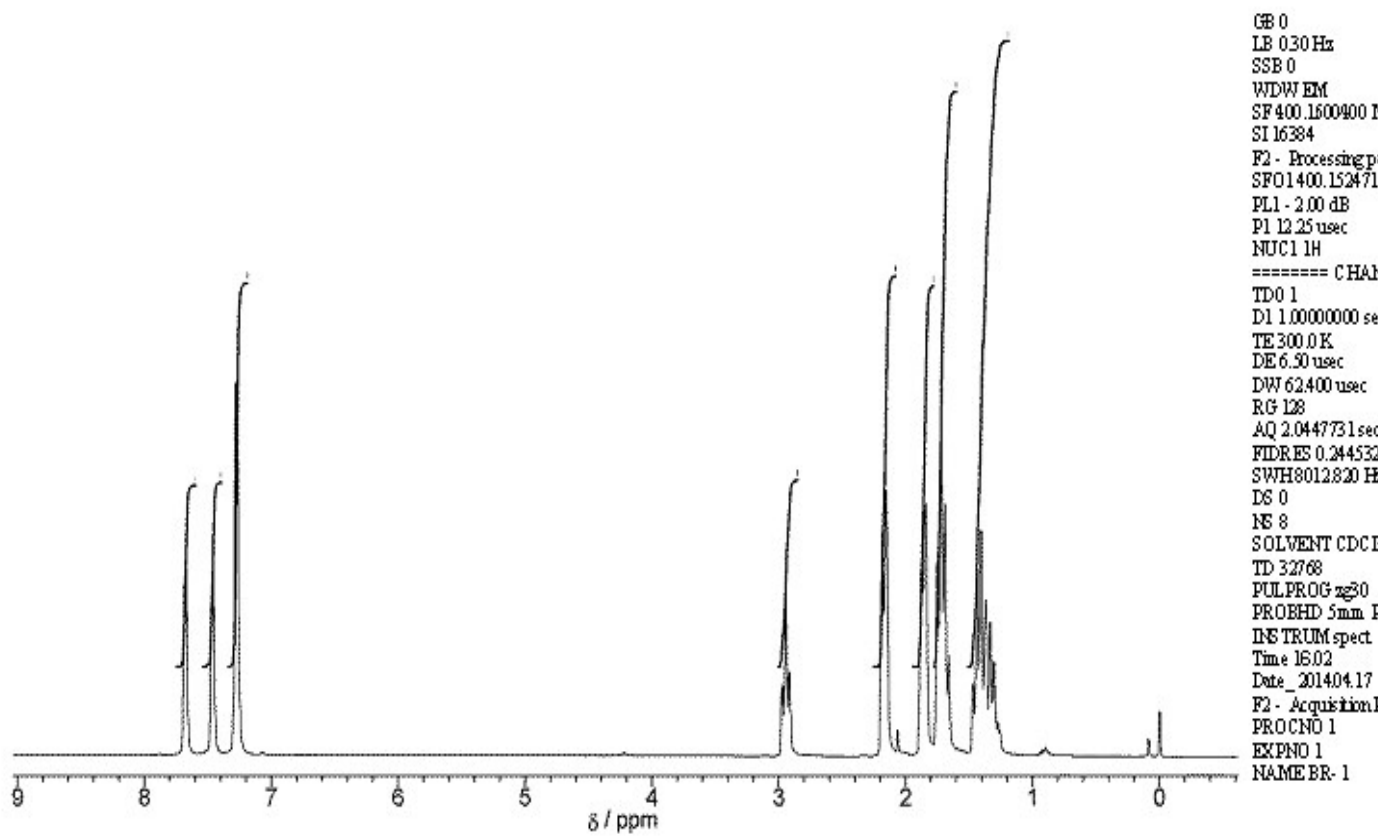
avtar saifon@yahoo.co.

**Compound 3g.**  $^1\text{H}$  NMR Spectrum ( $\text{CDCl}_3$ ).

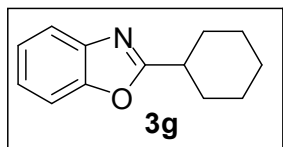


CHEMBIOTEK, A TCG Lifesciences Enterprise

BR-1 IN  $\text{CDCl}_3$  TCGLS/ARD/NMR02/K02

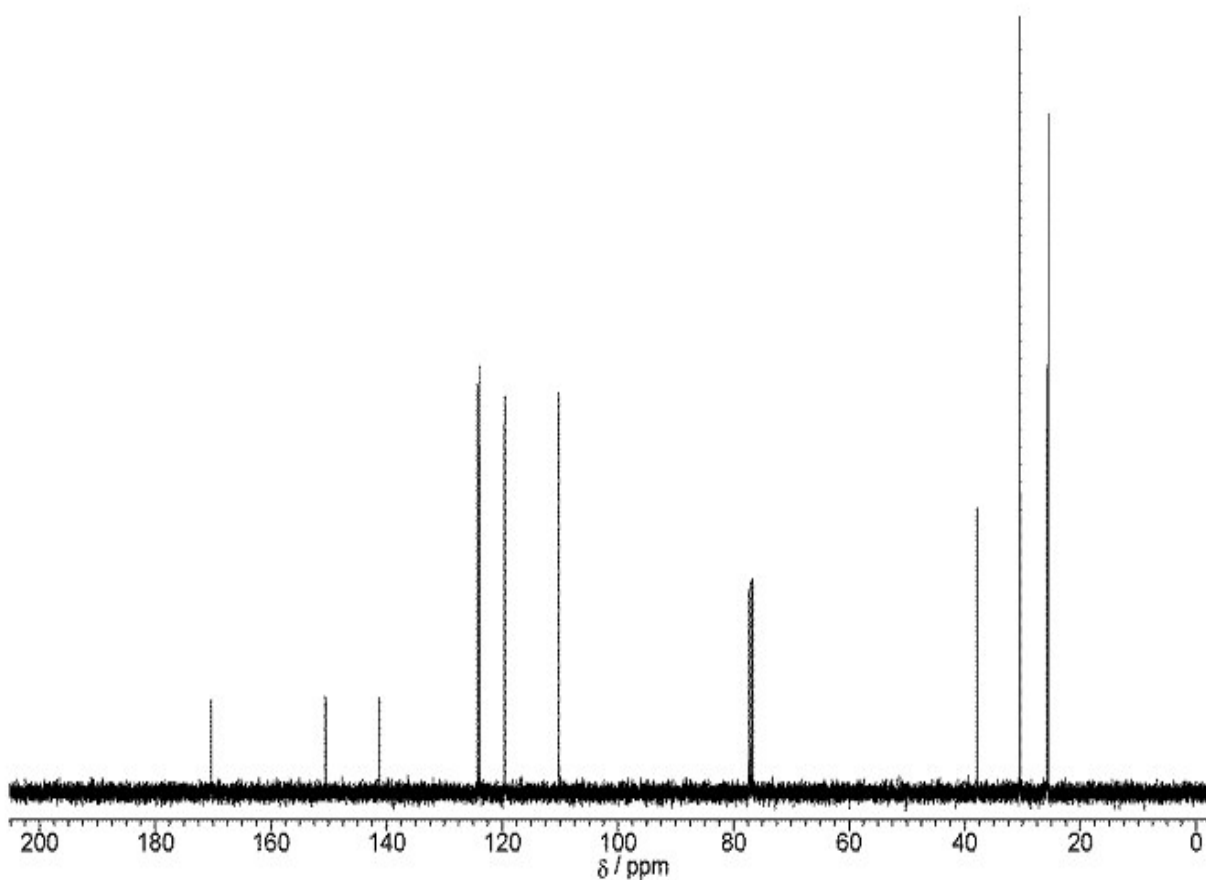


Compound 3g. <sup>13</sup>C NMR Spectrum (CDCl<sub>3</sub>).



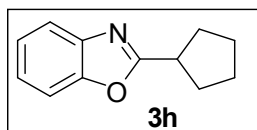
CHEMBIOTEK, A TCG Lifesciences Enterprise

BR- 1 IN CDCl<sub>3</sub> TCGLS/ARD/NMR02/K02



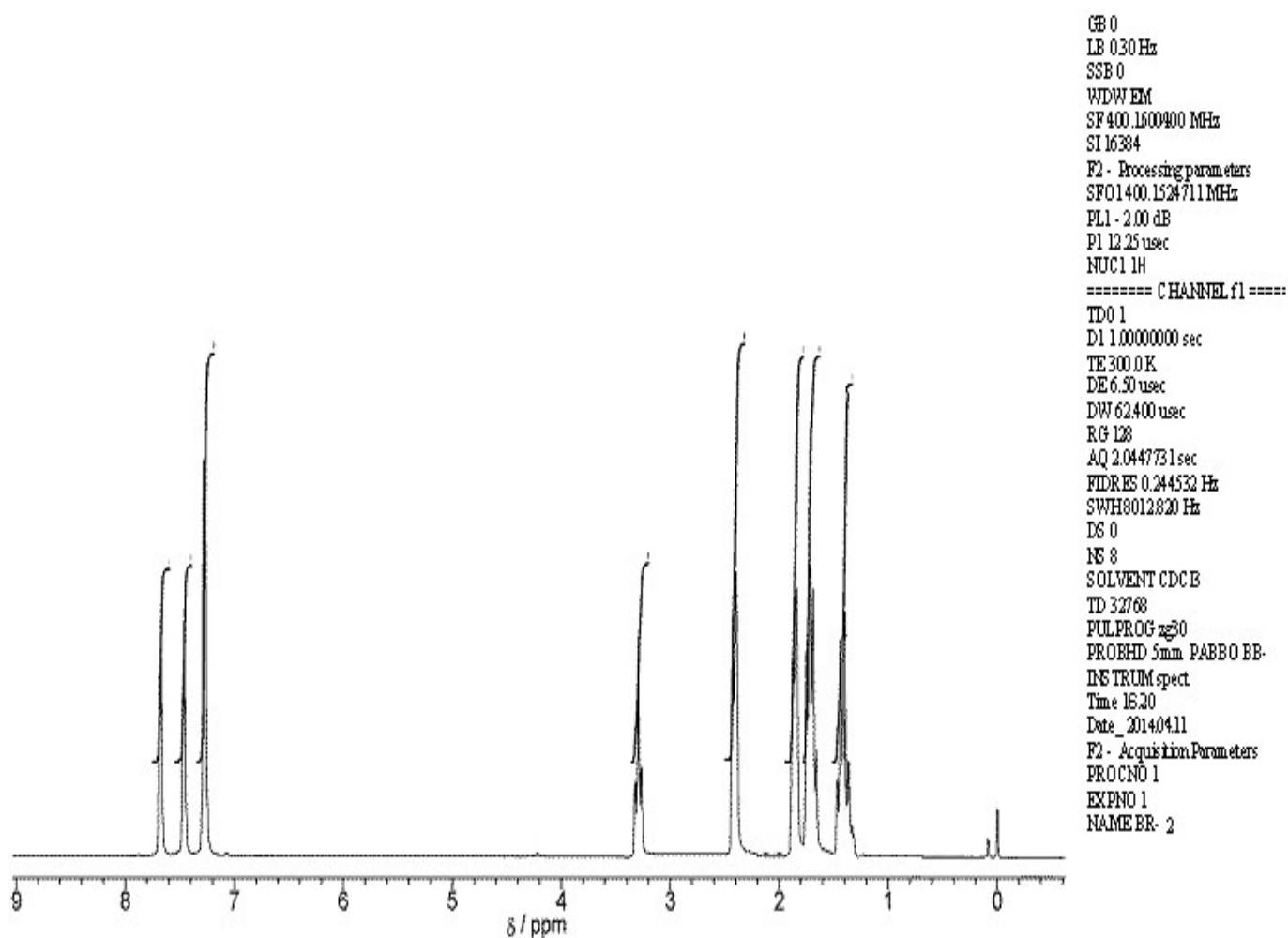
CE 0  
LE 0.30 Hz  
SE 0  
WDW/EM  
SF100.6178028 MHz  
SI131072  
F2 - Processing parameters  
SFO2400.1520008 MHz  
EL2 - 2.00 dB  
EL314.30 dB  
EL1214.30 dB  
PCPD80.00 wsc  
NUC2 1H  
CPDPRG vshel6  
===== CHANNEL f2 =====  
SFO1100.6278398 MHz  
EL1 - 2.50 dB  
FI 800 wsc  
NUC1 13C  
===== CHANNEL f1 =====  
ID01  
FID1A1.8999998 ssc  
d11.0300000 ssc  
I1 2.0000000 ssc  
TE 300.0 K  
IE 6.00 wsc  
DQ 20.800 wsc  
RG 2050  
AQ1.3431988 ssc  
FIDRES0.344798 Hz  
SFOH240384.61 Hz  
IS 2  
NS 244  
SOLVENT CDCl3  
ID 0334  
PULPROG zgpg  
PROCBD 1 mm PABCORP  
INSTRUM spect  
Time 12.23  
Date\_ 2014.04.17  
F2 - Acquisition Parameters  
PROCNO1  
EXNO 40  
NAME BR-1

Compound 3h. <sup>1</sup>H NMR Spectrum (CDCl<sub>3</sub>).



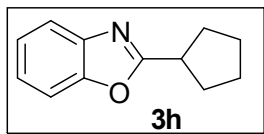
CHEMBIOTEK, A TCG Lifesciences Enterprise

BR- 2 IN CDCl<sub>3</sub> TCGLS/ARD/NMR02/K02



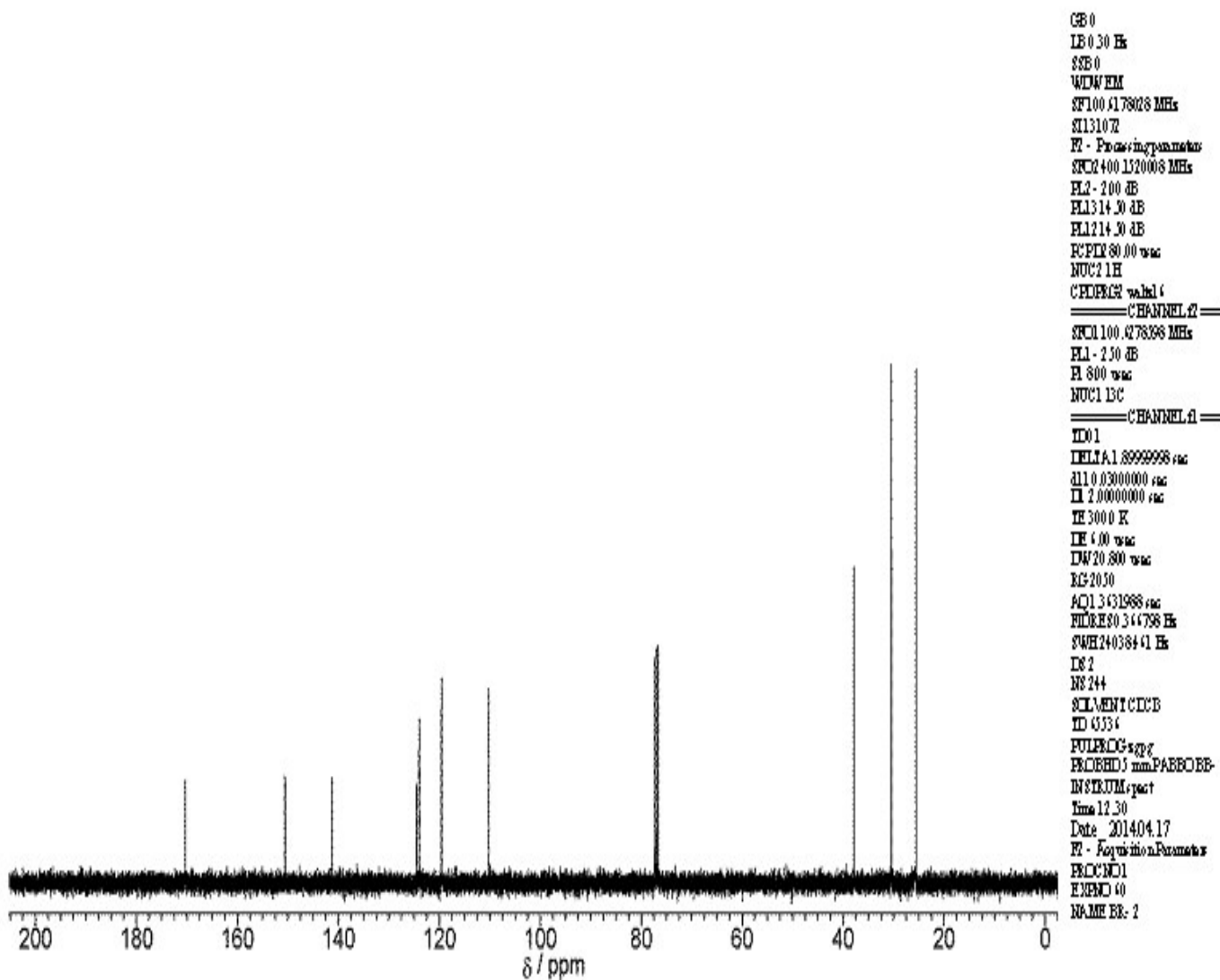


Compound 3h. <sup>13</sup>C NMR Spectrum (CDCl<sub>3</sub>).

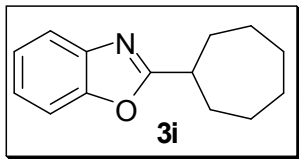


CHEMBIOTEK, A TCG Lifesciences Enterprise

BR-2 IN CDCl<sub>3</sub> TCGLS/ARD/NMR02/K02

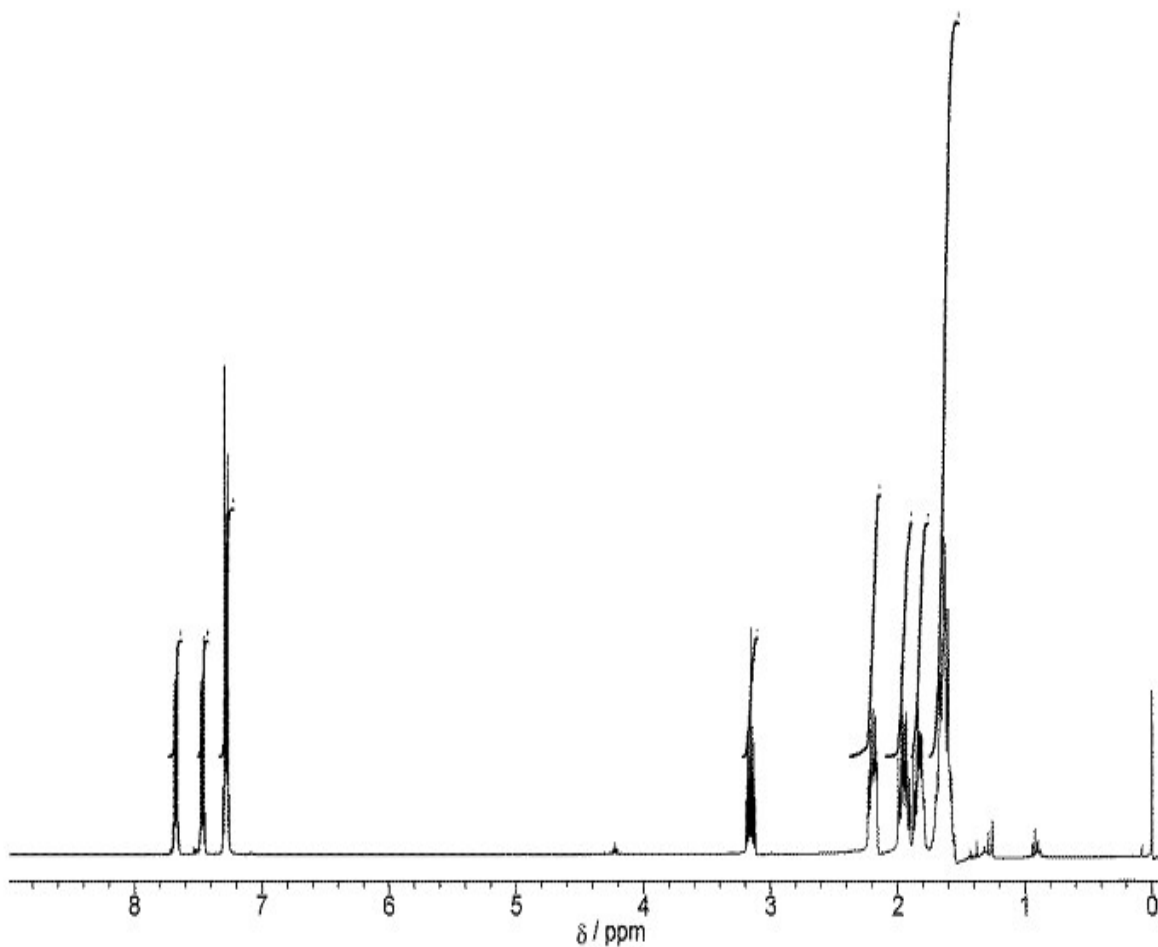


Compound 3i. <sup>1</sup>H NMR Spectrum (CDCl<sub>3</sub>).



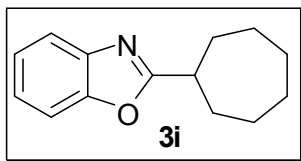
CHEMBIOTEK, A TCG Lifesciences Enterprise

BR- AIN CDC13 TCGLS/ARD/NMR02/K02



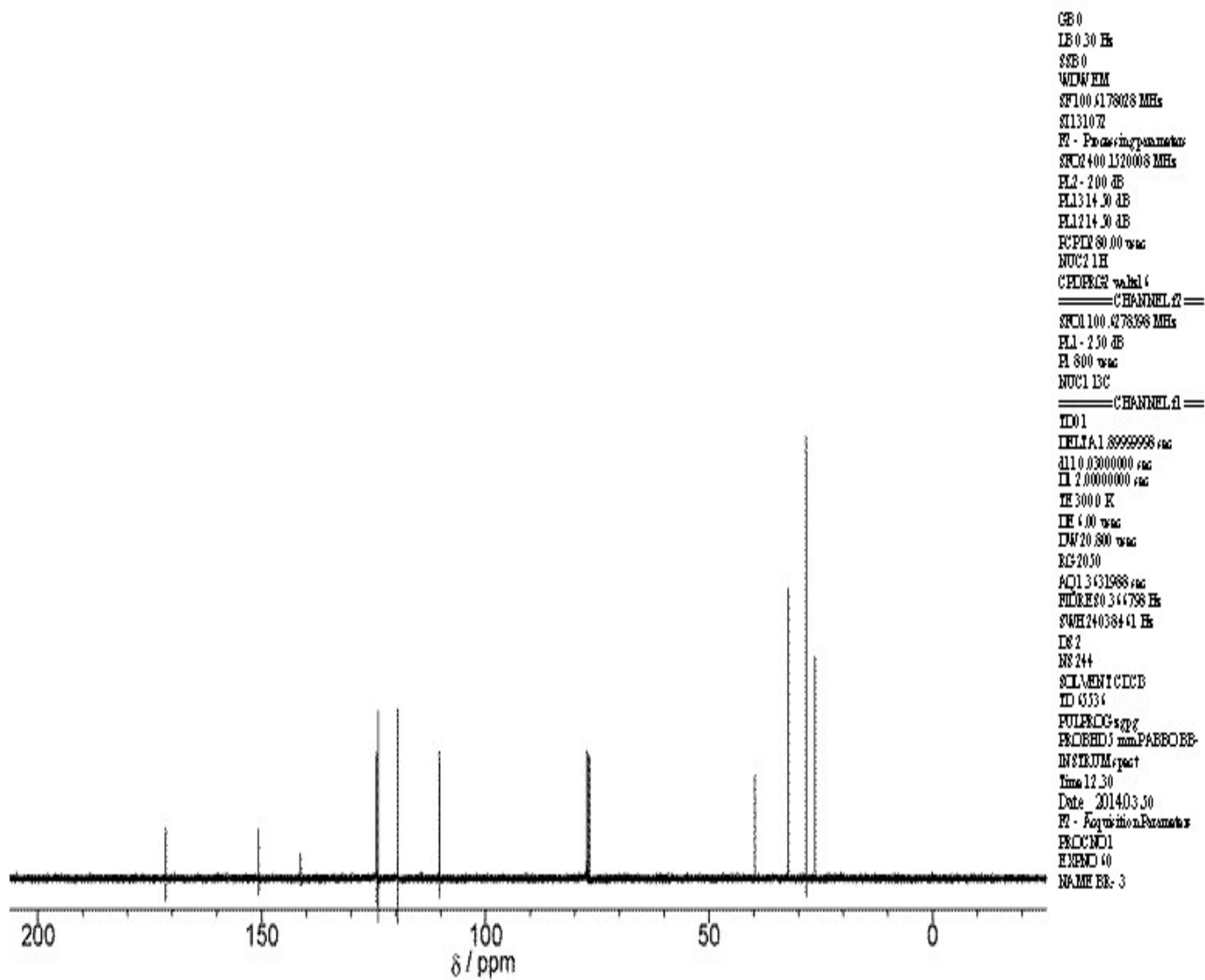
GB 0  
LB 030Hz  
SSB 0  
WDW EM  
SF 400.1509400 MHz  
SI 16384  
F2 - Processing parameters  
SFO1400.1524711 MHz  
PL1 - 2.00 dB  
PI 12.25 usec  
NUC1 1H  
===== CHANNEL f1 =====  
TD0 1  
D1 1.0000000 sec  
TE 300.0 K  
DE 6.30 usec  
DW 62.400 usec  
RG 128  
AQ 2.0447731 sec  
FIDRES 0.244532 Hz  
SWH8012820 Hz  
DS 0  
NS 8  
SOLVENT CDCl3  
TD 32768  
PULPROG zg30  
PROBHD 5mm PABBO BB-  
INSTRUM spect  
Time 20.20  
Date 20140821  
F2 - Acquisition Parameters  
PROCNO 1  
EXPNO 1  
NAME BR- A

Compound 3i. <sup>13</sup>C NMR Spectrum (CDCl<sub>3</sub>).

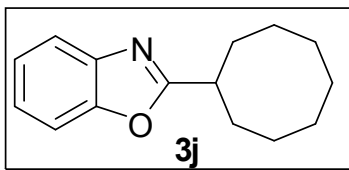


CHEMBIOTEK, A TCG Lifesciences Enterprise

BR-3 IN CDCl<sub>3</sub> TCGLS/ARD/NMR02/K02

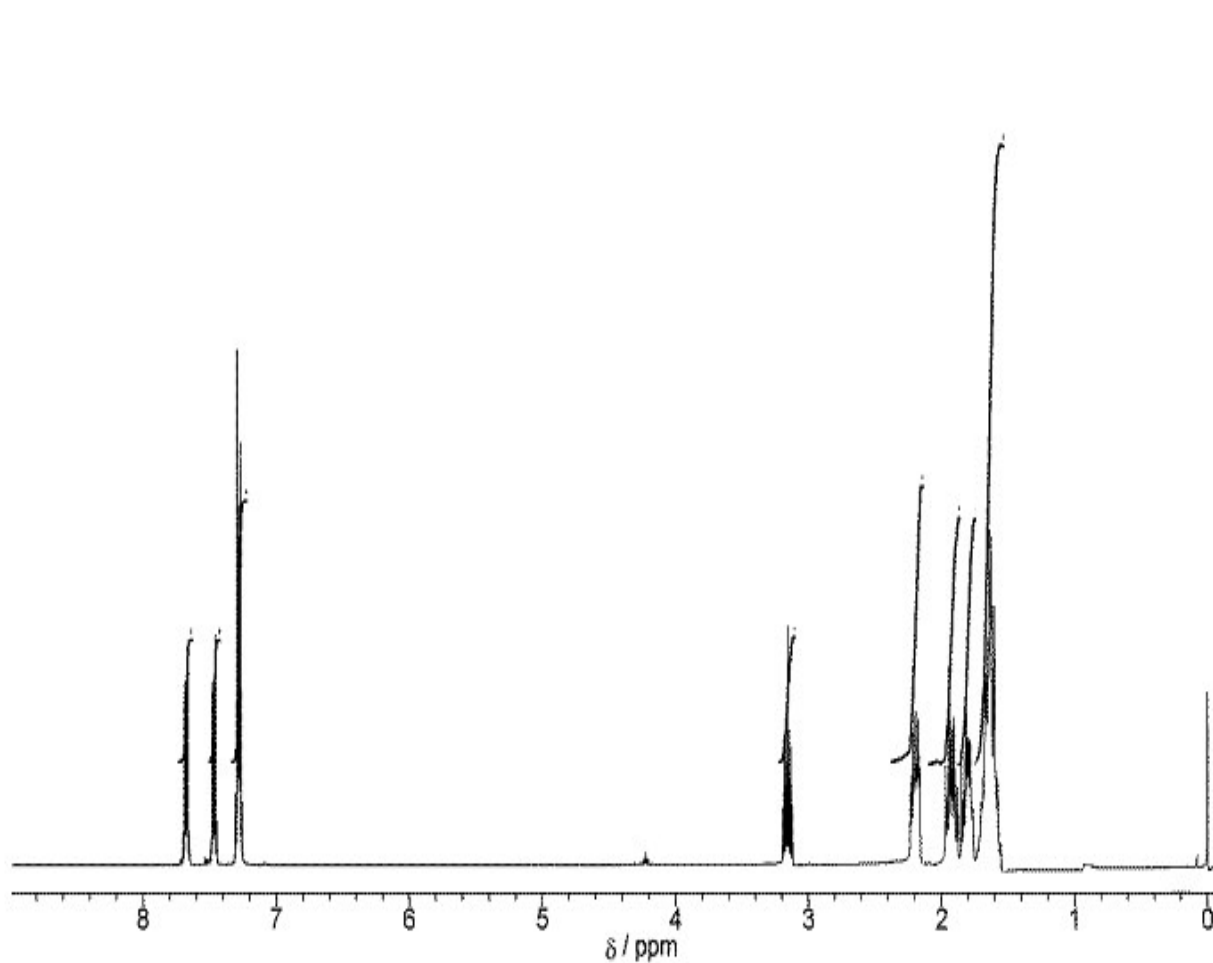


Compound 3j. <sup>1</sup>H NMR Spectrum (CDCl<sub>3</sub>).

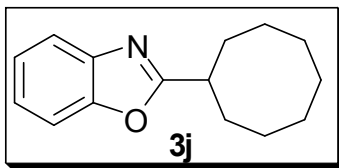


CHEMBIOTEK, A TCG Lifesciences Enterprise

BR-G.IN CDCl3 TCGLS/ARD/NMR02/K02

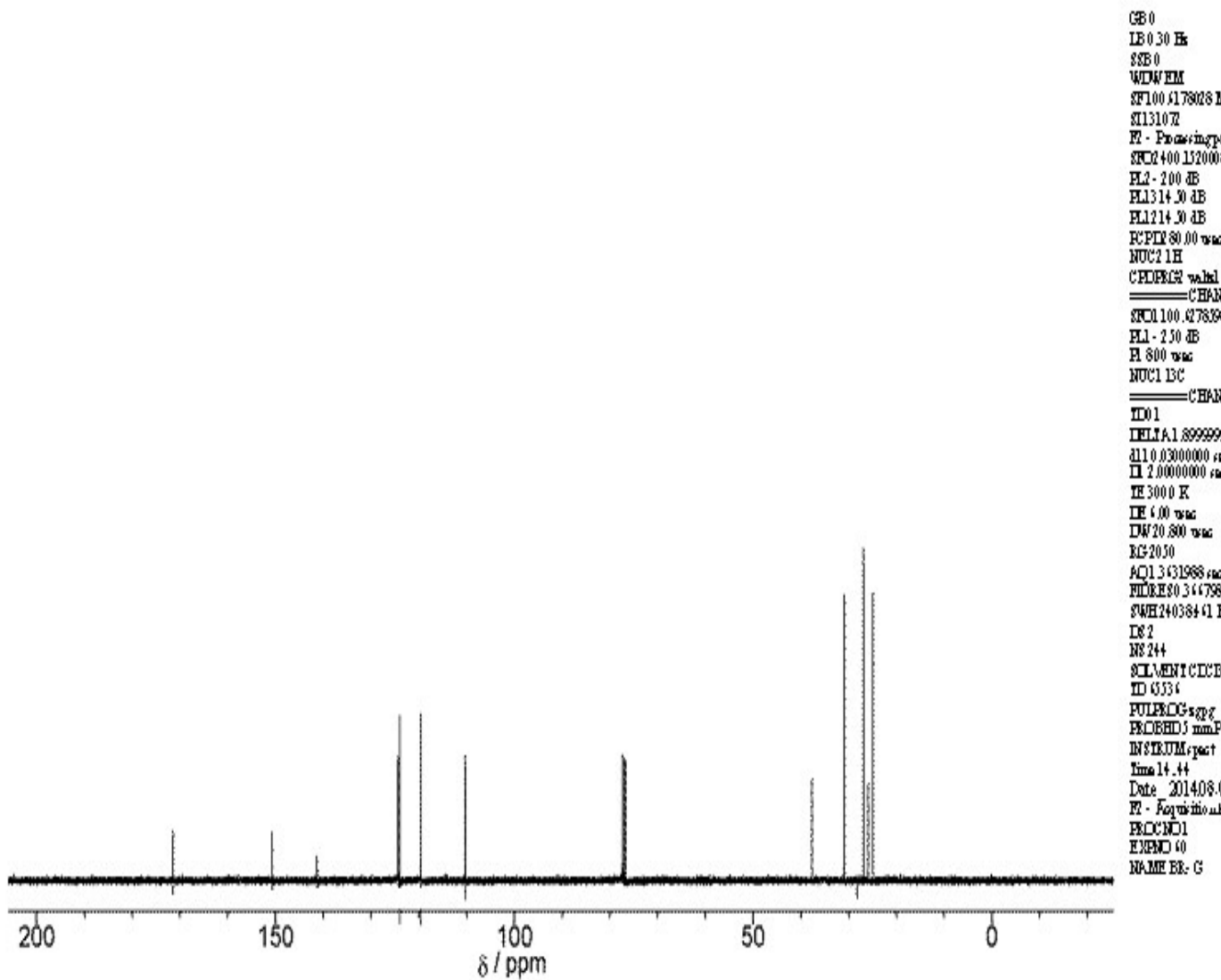


Compound 3j. <sup>13</sup>C NMR Spectrum (CDCl<sub>3</sub>).

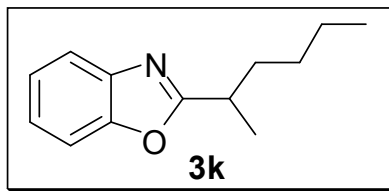


CHEMBIOTEK, A TCG Lifesciences Enterprise

BR-GIN CDCl<sub>3</sub> TCGLS/ARD/NMR02/K02

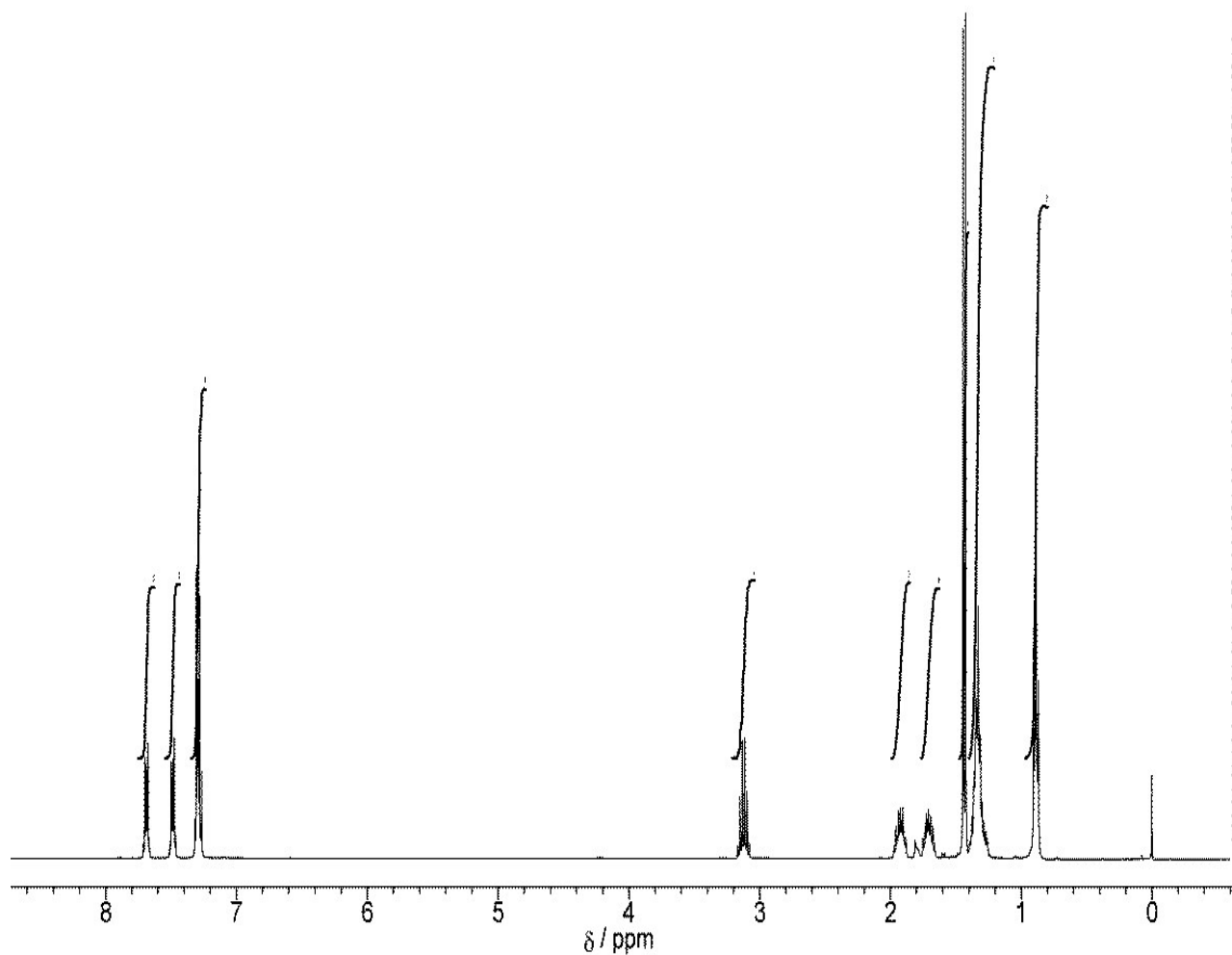


Compound 3k. <sup>1</sup>H NMR Spectrum (CDCl<sub>3</sub>).



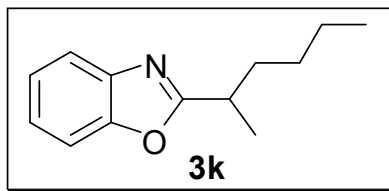
CHEMBIOTEK, A TCG Lifesciences Enterprise

BR- BIN CDCl3 TCGLS/ARD/NMR02/K02



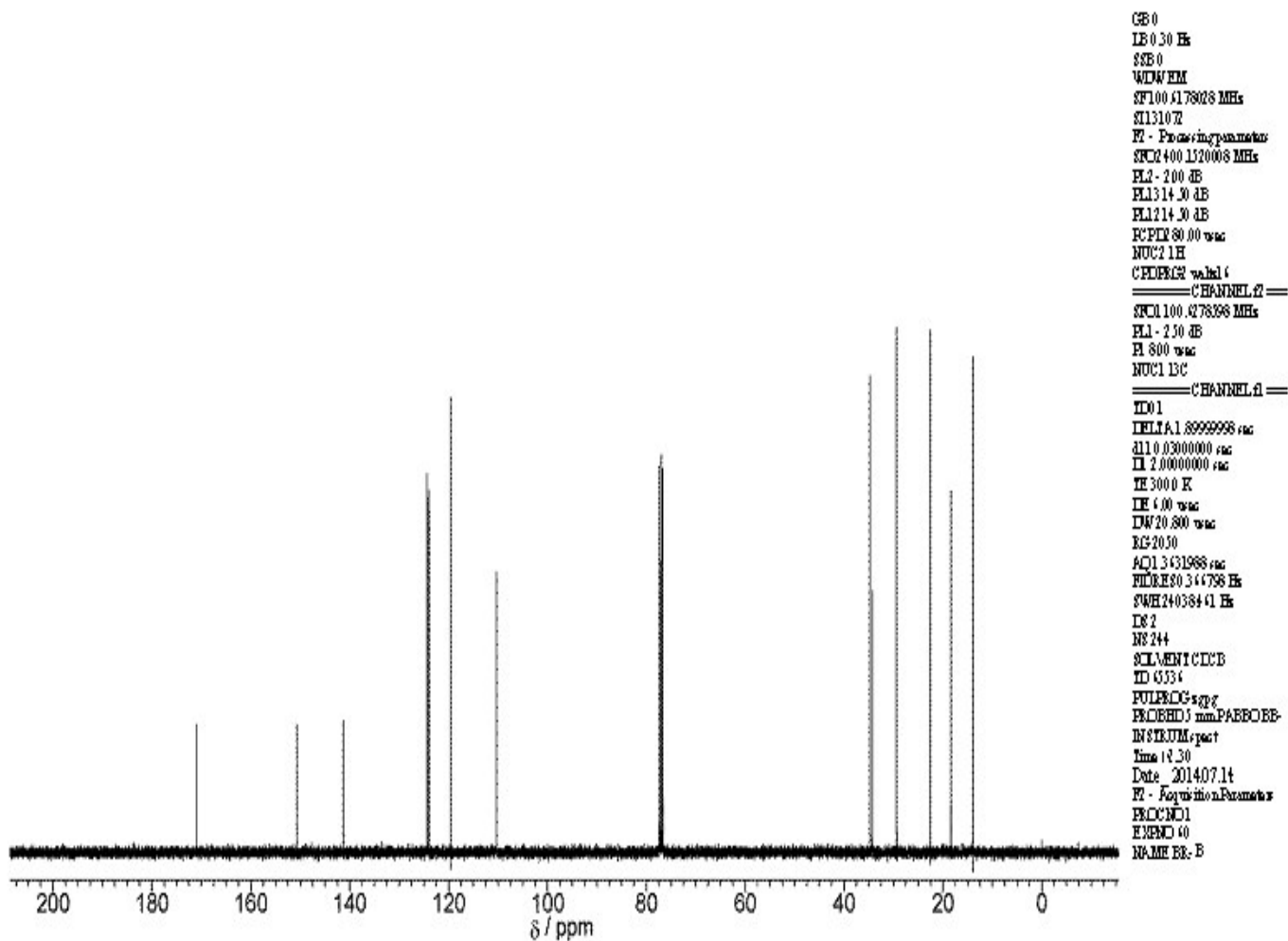
GB 0  
LB 0.30 Hz  
SSB 0  
WDW EM  
SF 400.1600400 MHz  
SI 16384  
F2 - Processing parameters  
SFO 1400.1524711 MHz  
PL1 - 2.00 dB  
P1 12.25 usec  
NUC1 1H  
===== CHANNEL f1 =  
TD 0 1  
D1 1.0000000 sec  
TE 300.0 K  
DE 6.50 usec  
DW 62.400 usec  
RG 128  
AQ 2.0447731 sec  
FIDRES 0.244532 Hz  
SFW 8012.820 Hz  
DS 0  
NS 8  
SOLVENT CDCl3  
TD 32768  
PULPROG zg30  
PROBHD 5mm PABBO B1  
INSTRUM spect  
Time 24.14  
Date\_ 20140824  
F2 - Acquisition Parameters  
PROCNO 1  
EXPNO 1  
NAME BR- B

Compound 3k. <sup>13</sup>C NMR Spectrum (CDCl<sub>3</sub>).

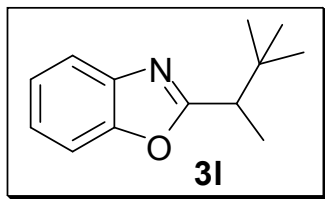


CHEMBIOTEK, A TCG Lifesciences Enterprise

BR- BIN CDC13 TCGLS/ARD/NMR02/K02

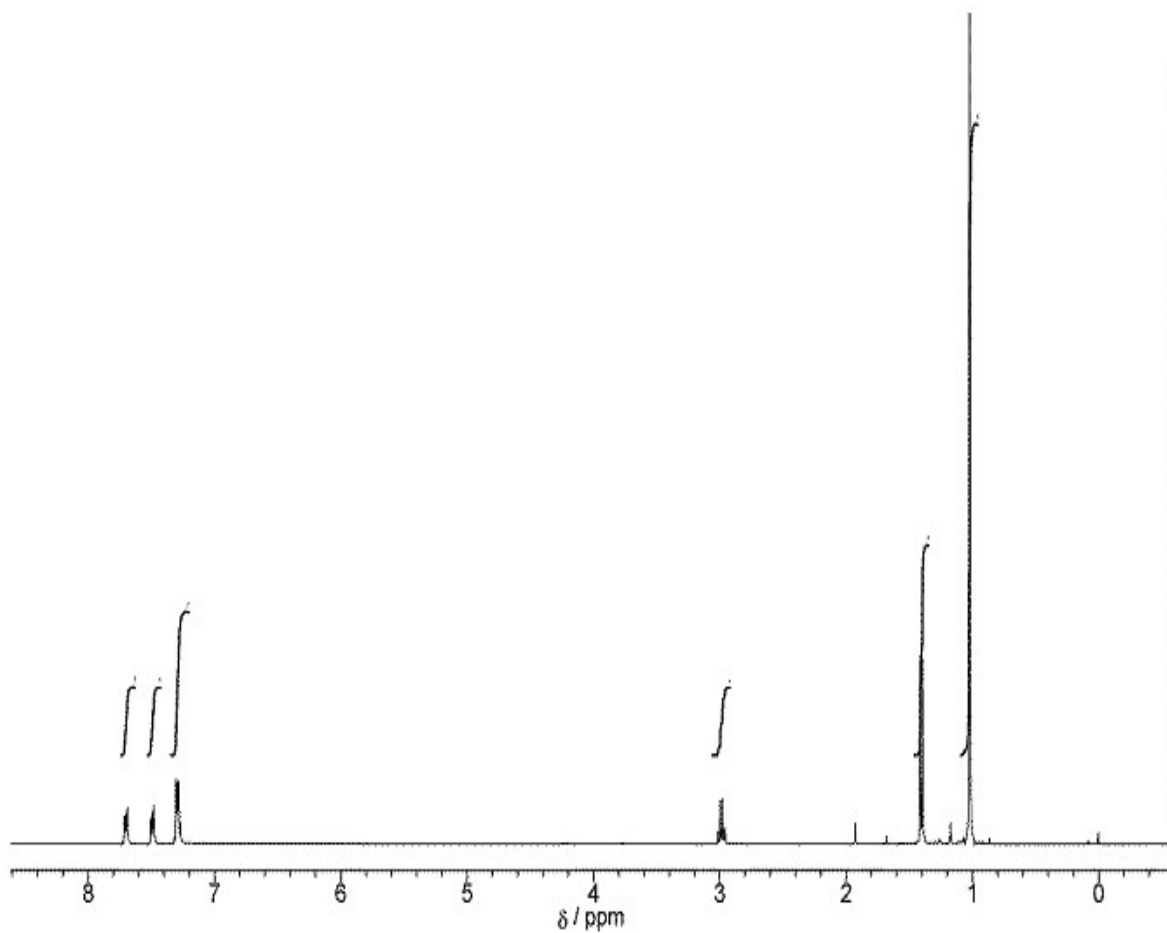


Compound 31. <sup>1</sup>H NMR Spectrum (CDCl<sub>3</sub>).



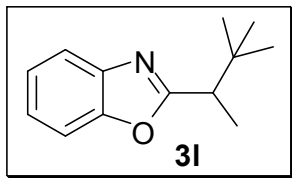
CHEMBIOTEK, A TCG Lifesciences Enterprise

BR-CIN CDCl3 TCGLS/ARD/NMR02/K02



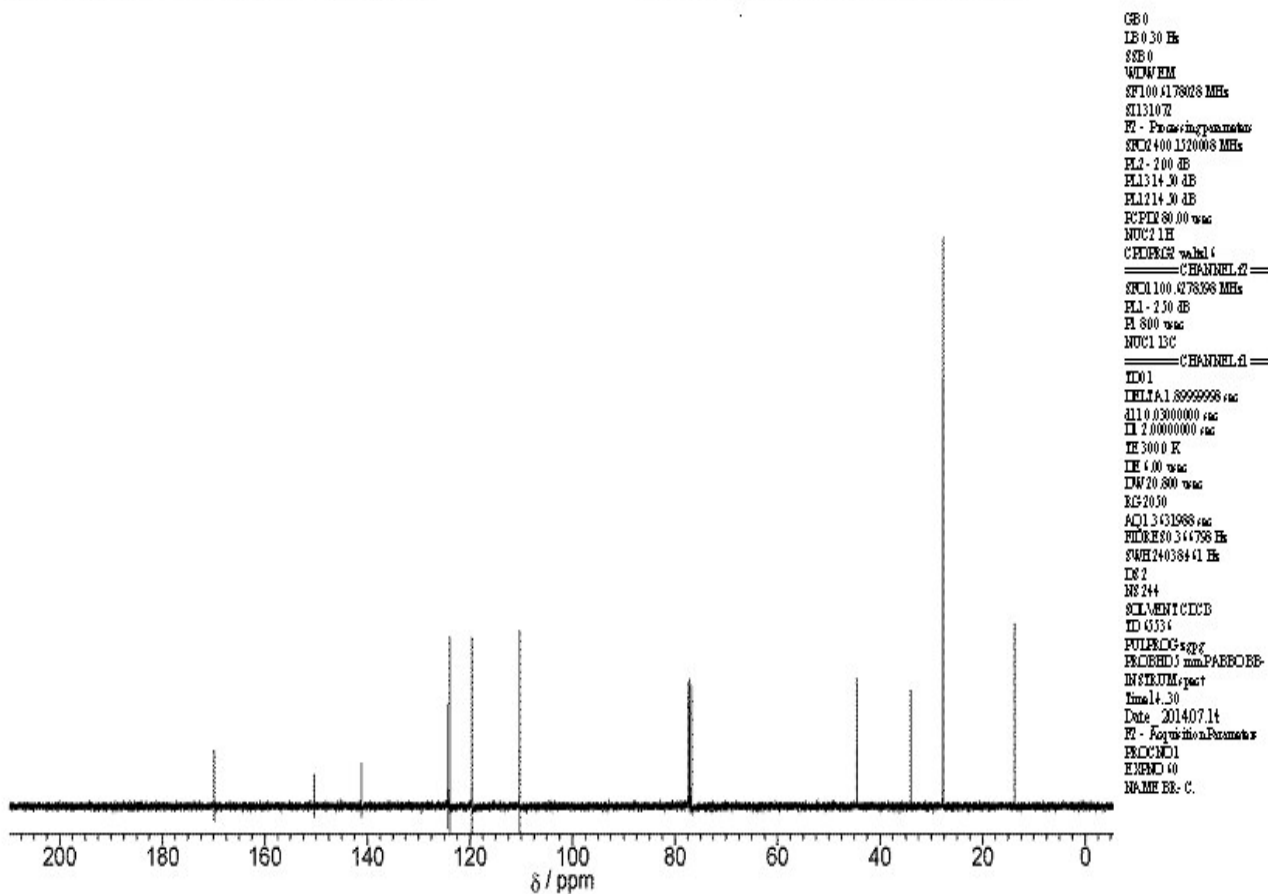


Compound 31. <sup>13</sup>C NMR Spectrum (CDCl<sub>3</sub>).

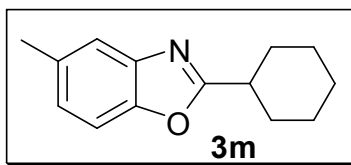


CHEMBIOTEK, A TCG Lifesciences Enterprise

BR-CIN CDC13 TCGLS/ARD/NMR02/K02

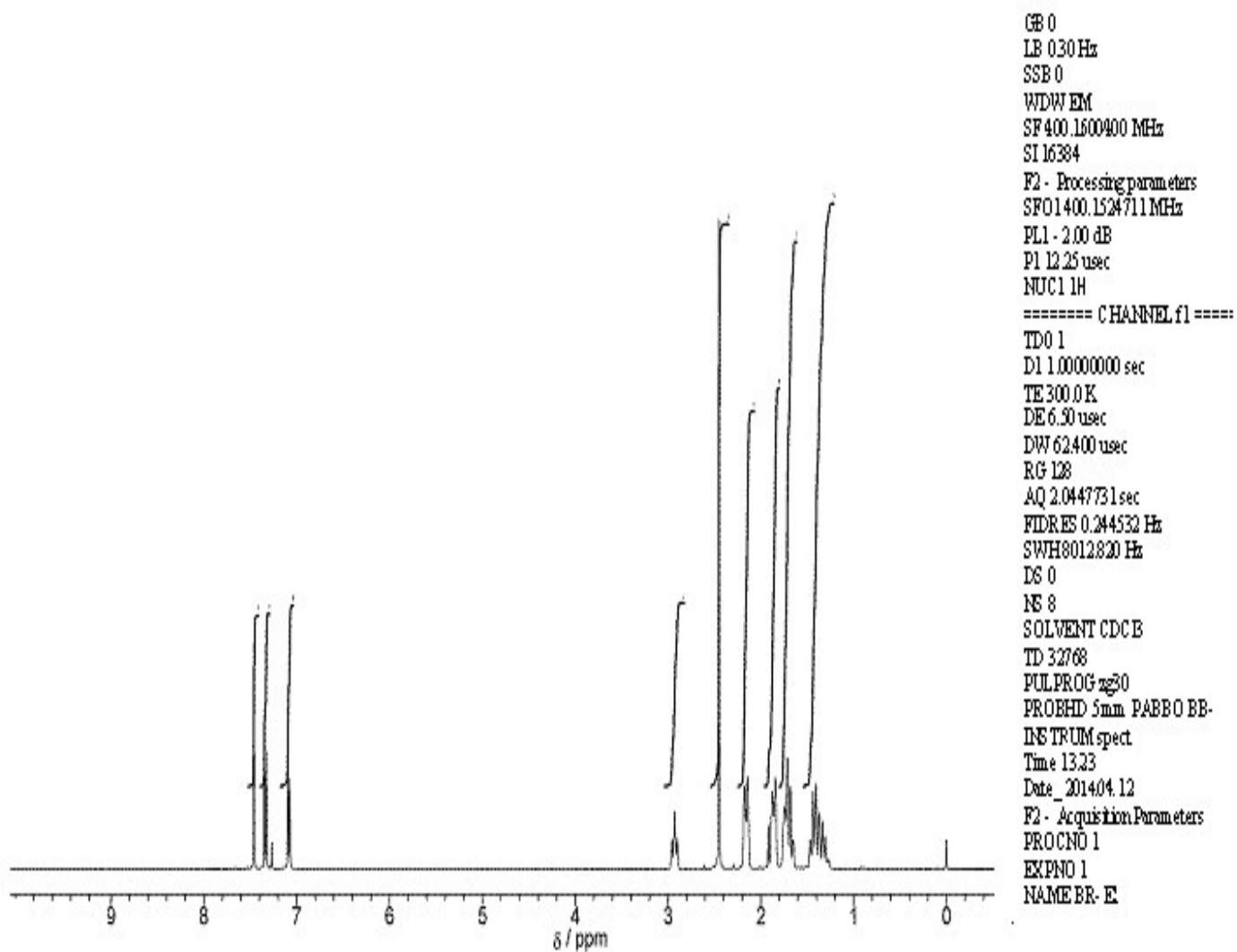


Compound 3m. <sup>1</sup>H NMR Spectrum (CDCl<sub>3</sub>).

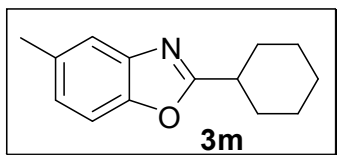


CHEMBIOTEK, A TCG Lifesciences Enterprise

BR-E IN CDCl<sub>3</sub> TCGLS/ARD/NMR02/K02

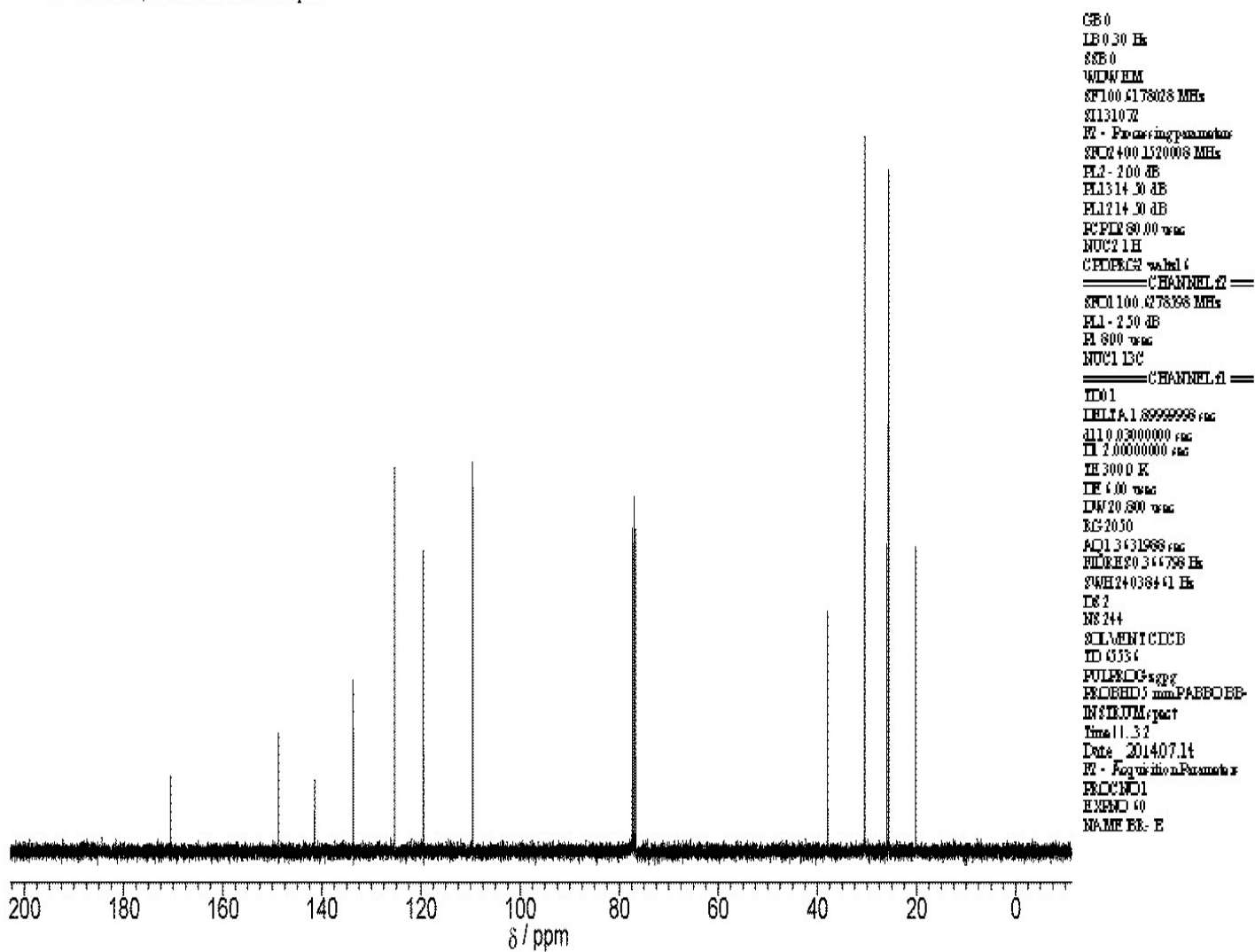


Compound 3m. <sup>13</sup>C NMR Spectrum (CDCl<sub>3</sub>).

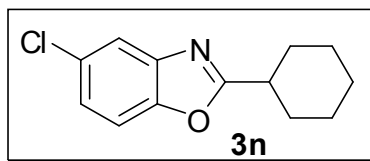


CHEMBIOTEK, A TCC Lifesciences Enterprise

BR-EIN CDCl<sub>3</sub>TCGLS/ARD/NMR02/K02

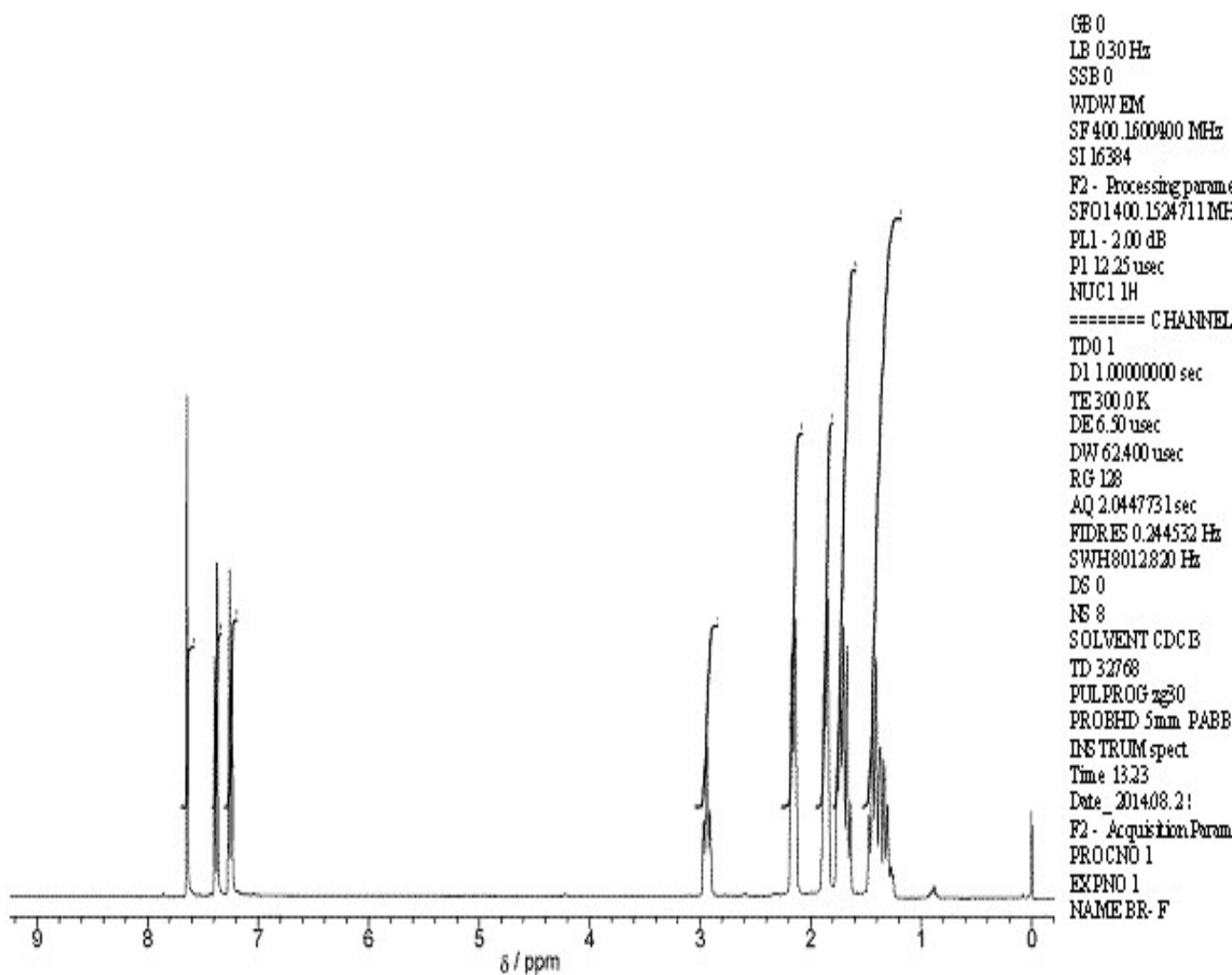


Compound 3n. <sup>1</sup>H NMR Spectrum (CDCl<sub>3</sub>).

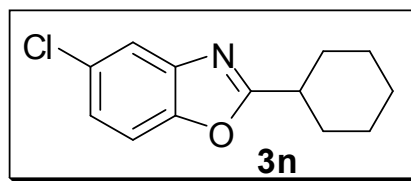


CHEMBIOTEK, A TCG Lifesciences Enterprise

BR-F IN CDCl3 TCGLS/ARD/NMR02/K02

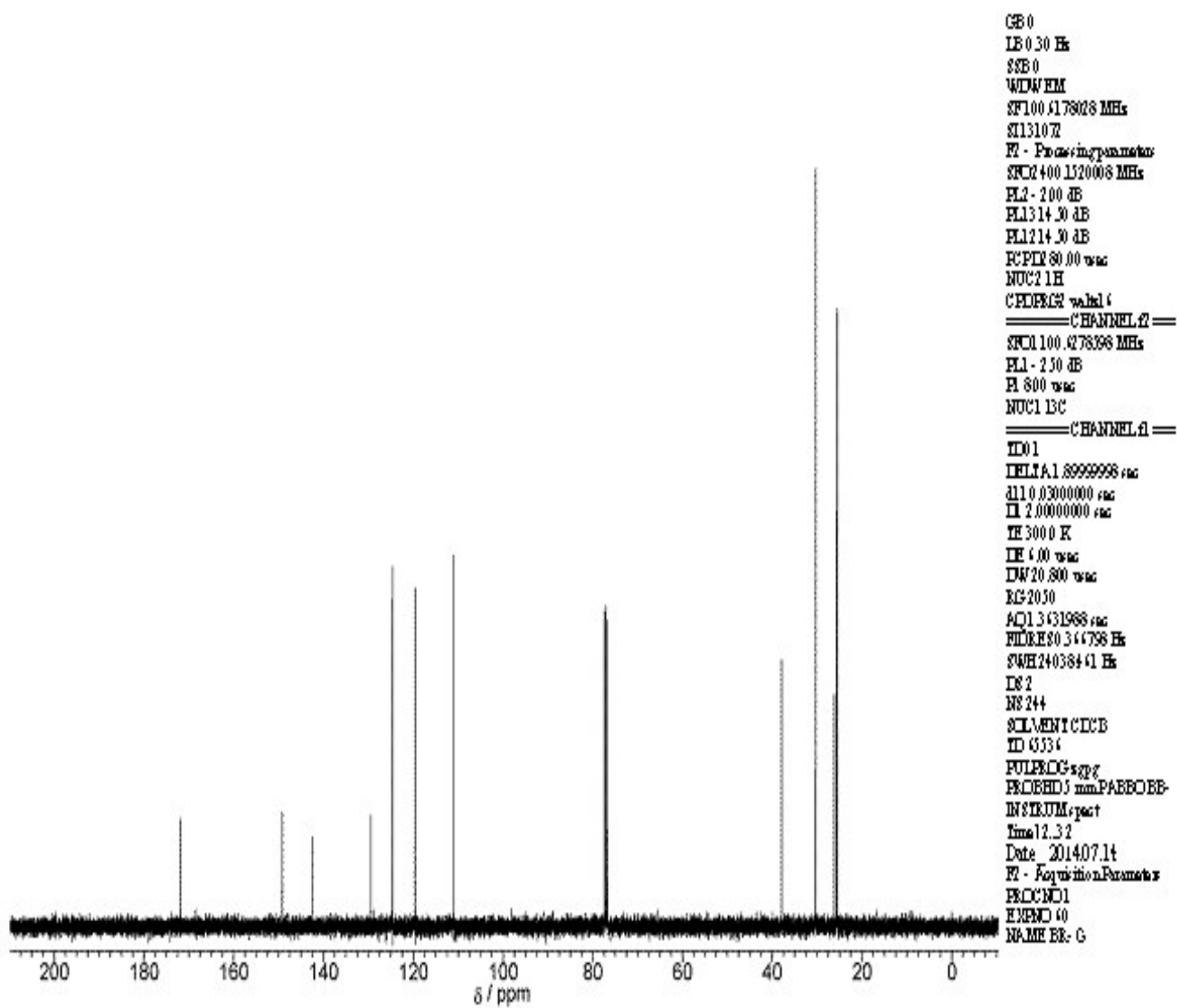


Compound 3n. <sup>13</sup>C NMR Spectrum (CDCl<sub>3</sub>).

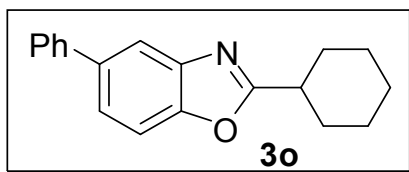


CHEMBIOTEK, A TCG Lifesciences Enterprise

BR-GIN CDCl3 TCGLS/ARD/NMR02/K02

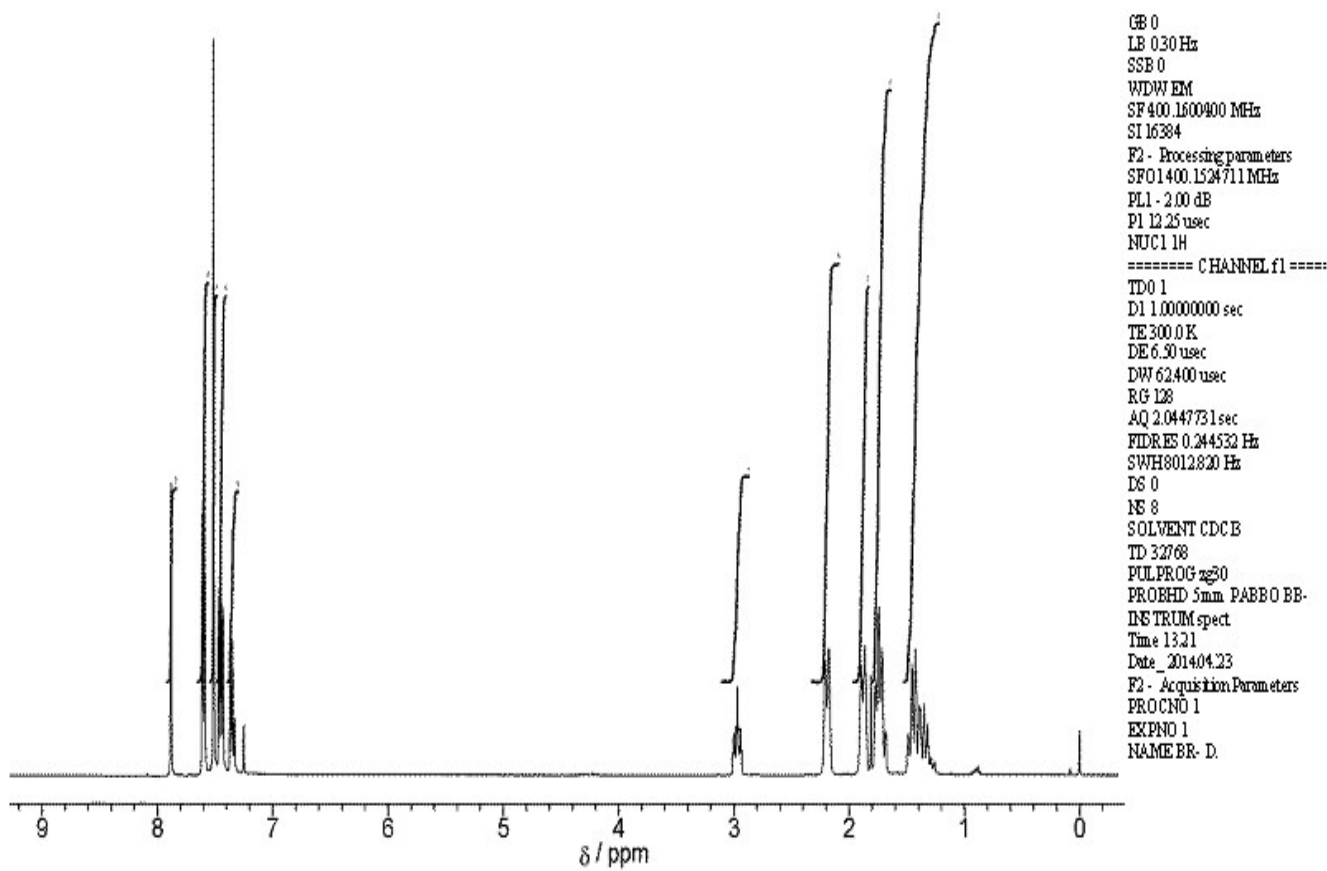


Compound 3o. <sup>1</sup>H NMR Spectrum (CDCl<sub>3</sub>).

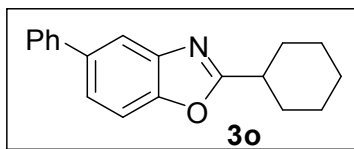


CHEMBIOTEK, A TCG Lifesciences Enterprise

BR-DIN CDCl<sub>3</sub> TCGLS/ARD/NMR02/K02

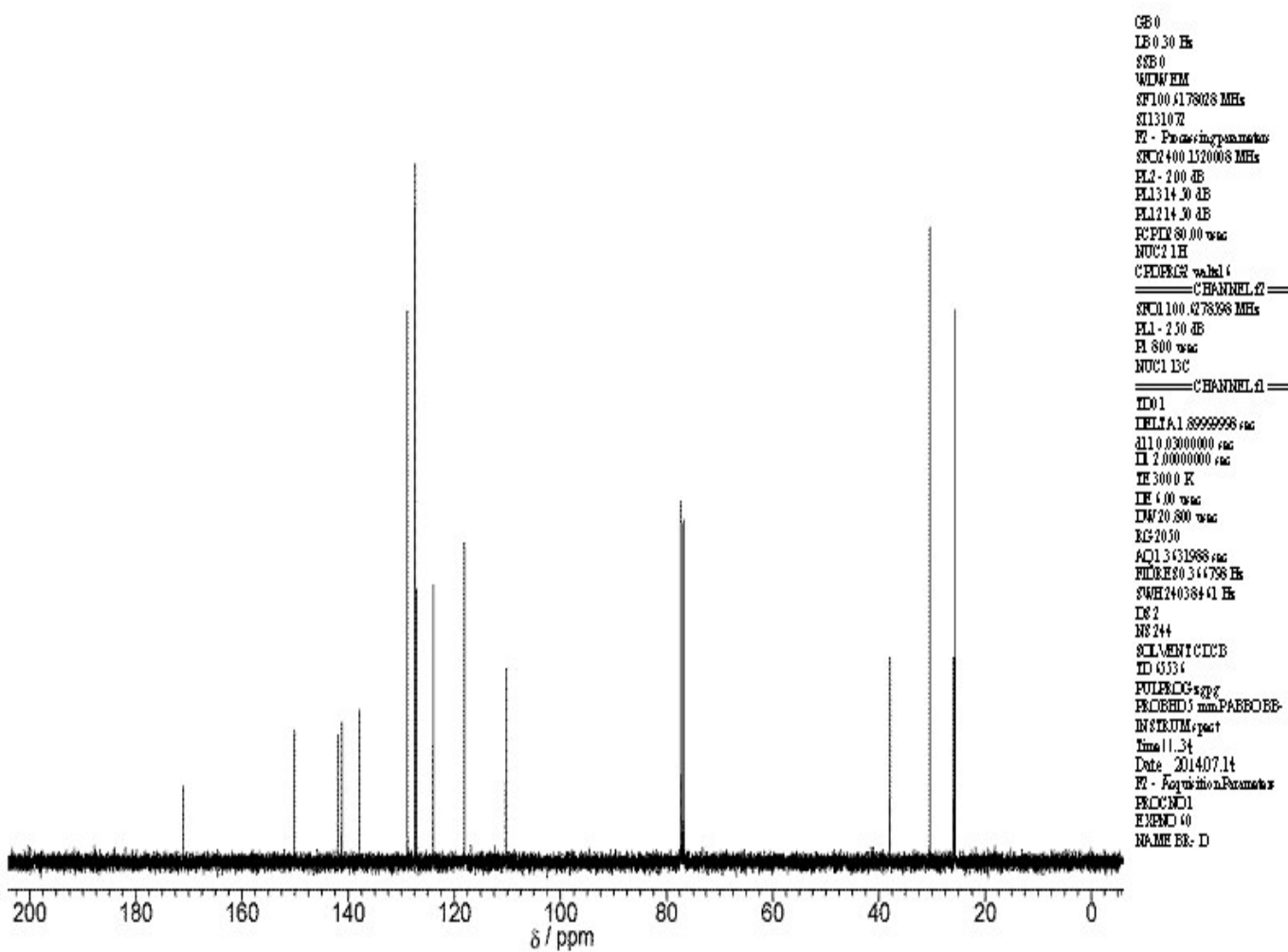


Compound 3o. <sup>13</sup>C NMR Spectrum (CDCl<sub>3</sub>).

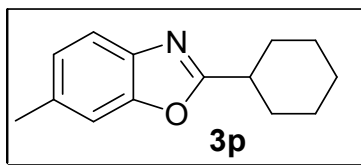


CHEMBIOTEK, A TCG Lifesciences Enterprise

BR-DIN CDCl<sub>3</sub> TCGLS/ARD/NMR02/K02

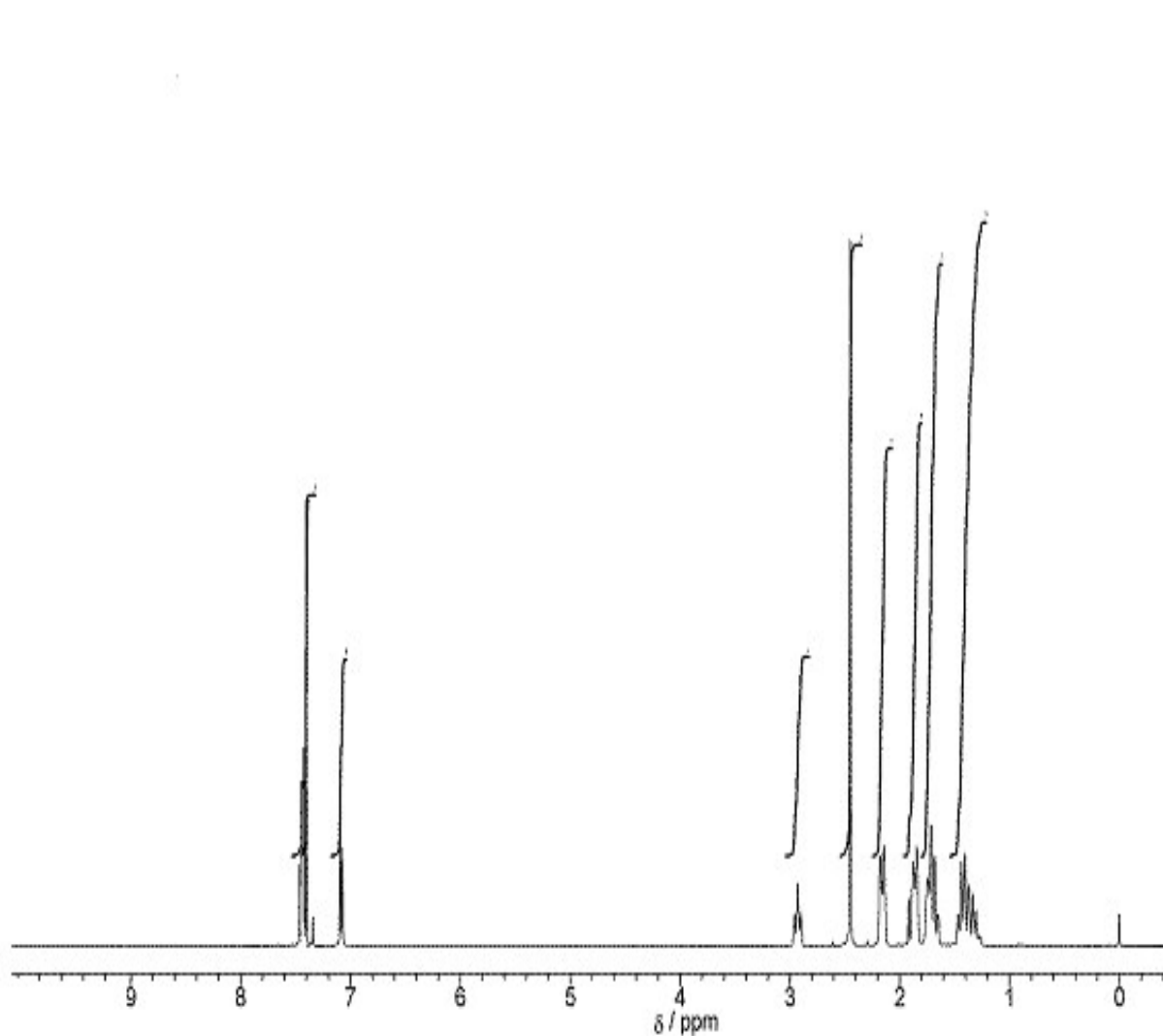


Compound 3p. <sup>1</sup>H NMR Spectrum (CDCl<sub>3</sub>).



CHEMBIOTEK, A TCG Lifesciences Enterprise

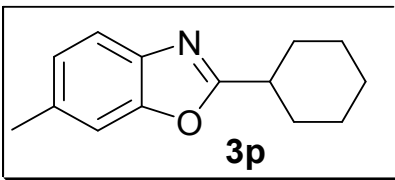
BR-MIN CDCl3 TCGLS/ARD/NMR02/K02



GB 0  
LB 0.30 Hz  
SSB 0  
WDW EM  
SF 400.1600400 MHz  
SI 16384  
F2 - Processing parameters  
SF 01400.1524711 MHz  
PL1 - 2.00 dB  
P1 12.25 usec  
NUC1 1H  
===== CHANNEL f  
TD 0 1  
D1 1.00000000 sec  
TE 300.0 K  
DE 6.50 usec  
DW 62.400 usec  
RG 128  
AQ 2.0447731 sec  
FIDRES 0.244532 Hz  
SWH8012.820 Hz  
DS 0  
NS 8  
SOLVENT CDCl3  
TD 32768  
PULPROG zg30  
PROBHD 5mm PABBO  
INSTRUM spect  
Time 13.11  
Date\_ 2014.12.13  
F2 - Acquisition Parameters  
PROCNO 1  
EXPNO 1  
NAME BR-M

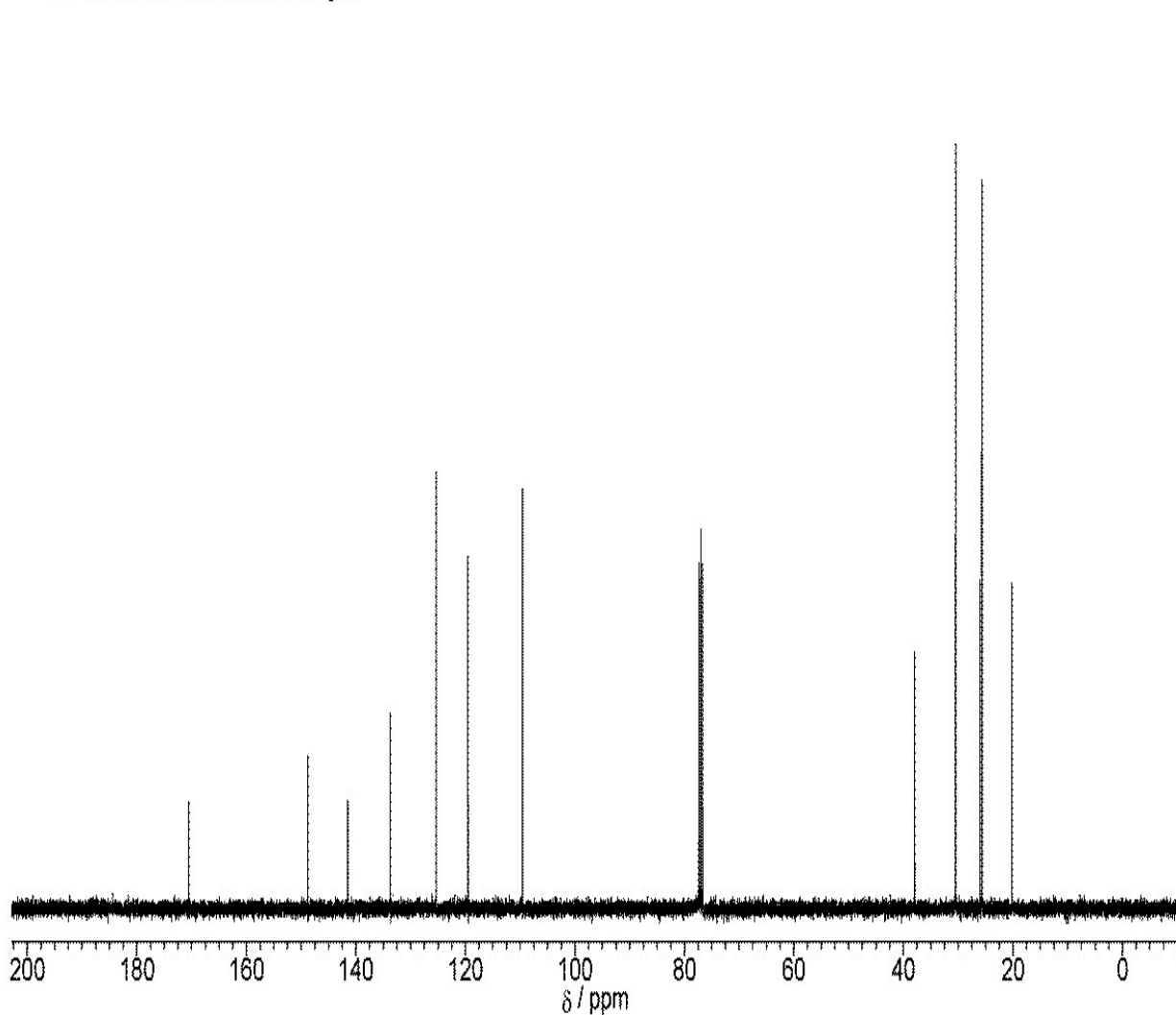


Compound 3p.  $^{13}\text{C}$  NMR Spectrum ( $\text{CDCl}_3$ ).



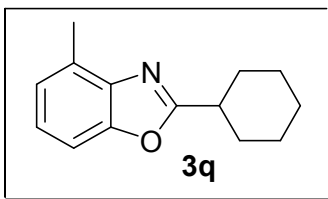
CHEMBIOTEK, A TCG Lifesciences Enterprise

BR-MIN CDCl3 TCG LS/ARD/NMR02/K02



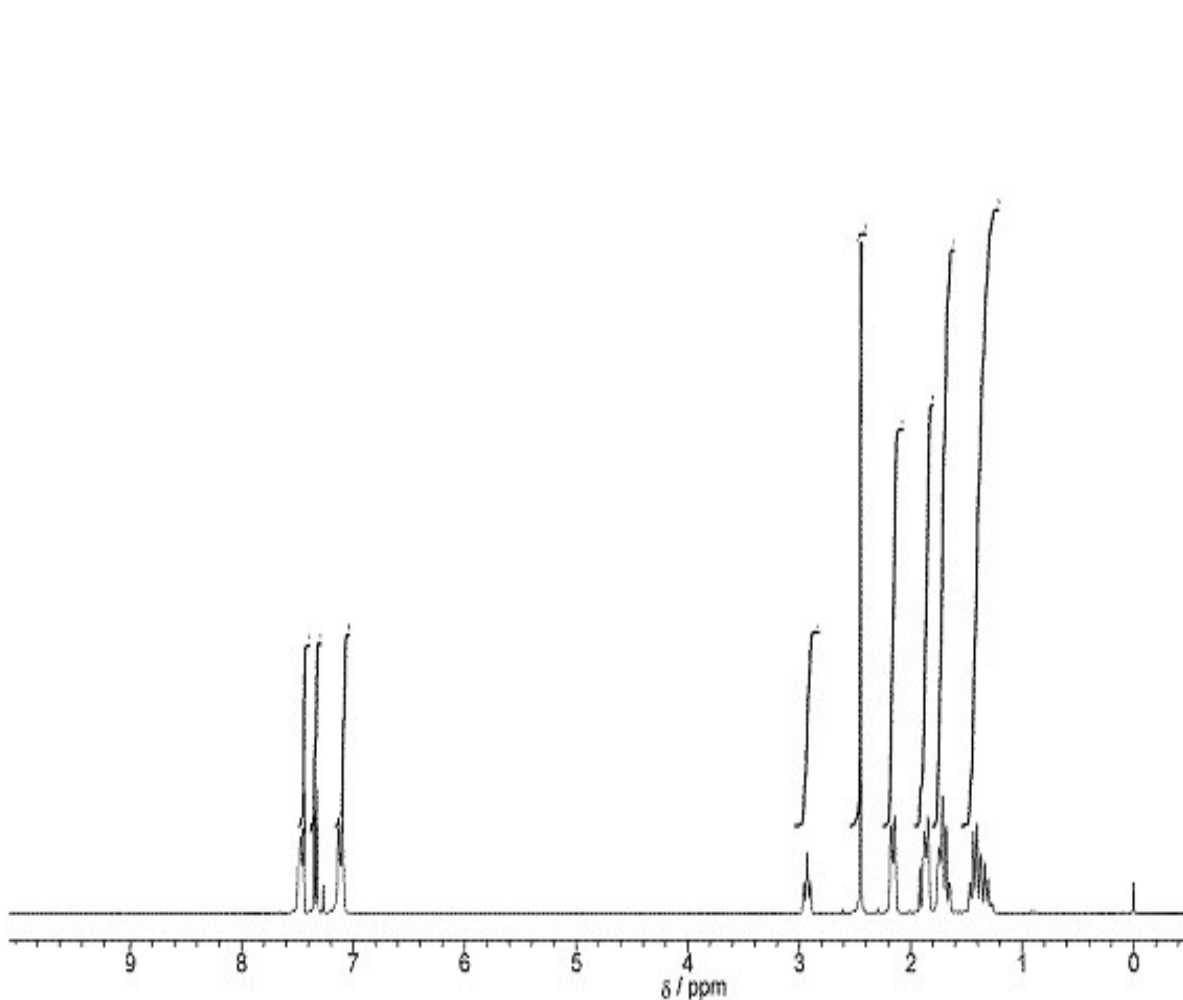
CS0  
LB 0.30 Hz  
SSE 0  
WDW HEM  
SF100 4178028 MHz  
SI13107  
F2 - Processing parameter  
SFO2 400.1520068 MHz  
FL2 - 2.00 dB  
EL1514.30 dB  
EL1214.30 dB  
PCPD 60.00 usec  
NUC2 1H  
CPDPRG2 waltz16  
===== CHANNEL f2 =====  
SFO1 100.6278968 MHz  
FL1 - 2.50 dB  
EL 600 usec  
NUC1 13C  
===== CHANNEL f1 =====  
TD01  
LHFLA 1.89999998 usec  
dL1 0.00000000 usec  
TL 2.00000000 usec  
TE 300.0 K  
DE 6.00 usec  
DM 20.800 usec  
RG 2050  
AQ1 3 431988 usec  
FIDRES 0.3 4798 Hz  
SFOH2 40384.41 Hz  
DS 2  
NS 244  
SCLAMN1C1CB  
TD 4334  
PULPROG sggg  
PCPDPRG2 mmPABPOB  
INSTRUM spect  
Time 11.52  
Date\_ 201412.13  
F2 - Acquisition Parameters  
PROCNO 1  
EXNO 40  
NAME BR-M

Compound 3q. <sup>1</sup>H NMR Spectrum (CDCl<sub>3</sub>).



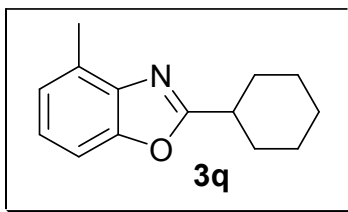
CHEMBIOTEK, A TCG Lifesciences Enterprise

BR-0: IN CDCl<sub>3</sub> TCGLS/ARD/NMR02/K02



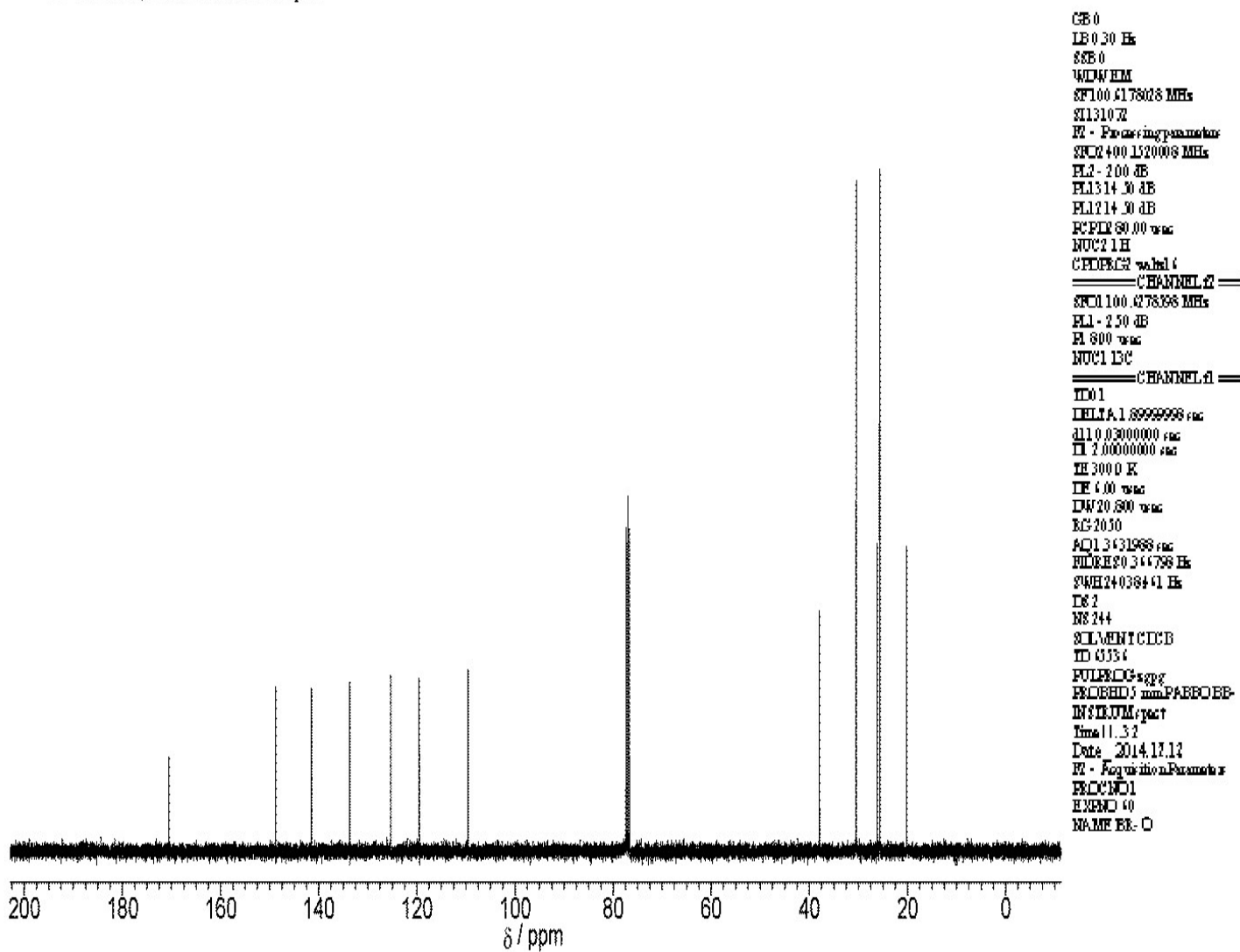
GB 0  
LB 0.30 Hz  
SSB 0  
WDW EM  
SF 400.1500400 MHz  
SI 16384  
F2 - Processing parameter  
SFO 1400.1524711 MHz  
PL1 - 2.00 dB  
P1 12.25 usec  
NUC1 1H  
===== CHANNEL f1  
TD0 1  
D1 1.00000000 sec  
TE 300.0 K  
DE 6.50 usec  
DW 62.400 usec  
RG 128  
AQ 2.0447731 sec  
FIDRES 0.244532 Hz  
SWH 8012.820 Hz  
DS 0  
NS 8  
SOLVENT CDCl<sub>3</sub>  
TD 32768  
PULPROG zg30  
PROBHD 5mm PABBO1  
INSTRUM spect  
Time 13.20  
Date 2014.12.12  
F2 - Acquisition Parameter  
PROCNO 1  
EXPNO 1  
NAME BR-0

Compound 3q. <sup>13</sup>C NMR Spectrum (CDCl<sub>3</sub>).



CHEMBIOTEK, A TCG Lifesciences Enterprise

BR-QIN CDCl3 TCGLS/ARD/NMR02/K02



#### IV. References:

1. W.-M. Zhao, X.-L. Chen, J.-W. Yuan, L.-B. Qu, L.-K. Duana and Y.-F. Zhao, *Chem. Commun.*, 2014, **50**, 2018.
2. P. Ren, I. Salihu, R. Scopelliti and X. L. Hu, *Org. Lett.* 2012, **14**, 1748.
3. P. Y. Xin, H. Y. Niu, G. R. Qu, R. F. Ding and H. M. Guo, *Chem. Commun.*, 2012, **48**, 6717.
4. T. Yao, K. Hirano, T. Satoh and M. Miura, *Angew. Chem., Int. Ed.*, 2012, **51**, 775.
5. R. Xia, H.-Y. Niu, G.-R. Qu and H.-M. Guo, *Org. Lett.*, 2012, **14**, 5546.
6. G. A. Molander, V. Colombel and V. A. Braz, *Org. Lett.*, 2011, **13**, 1852.