

Facile Synthesis of 2-azaspiro[3.4]octane

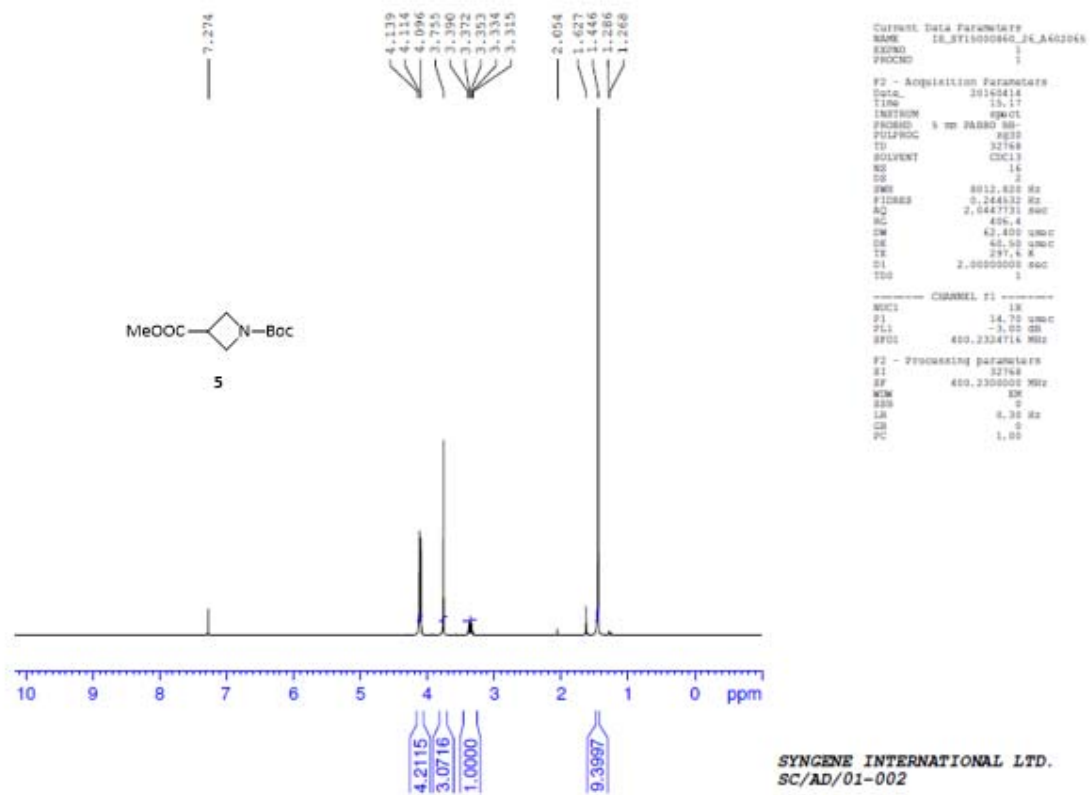
Subbiah Ramesh,^[a] Ramadas Balakumar,^{*[a]} John R. Rizzo^[b] and Tony Y. Zhang^[b]

^[a] Syngene International Ltd., Biocon Park, Plot No. 2 & 3, Bommasandra IV Phase, Jigani Link Road, Bangalore 560 099, India

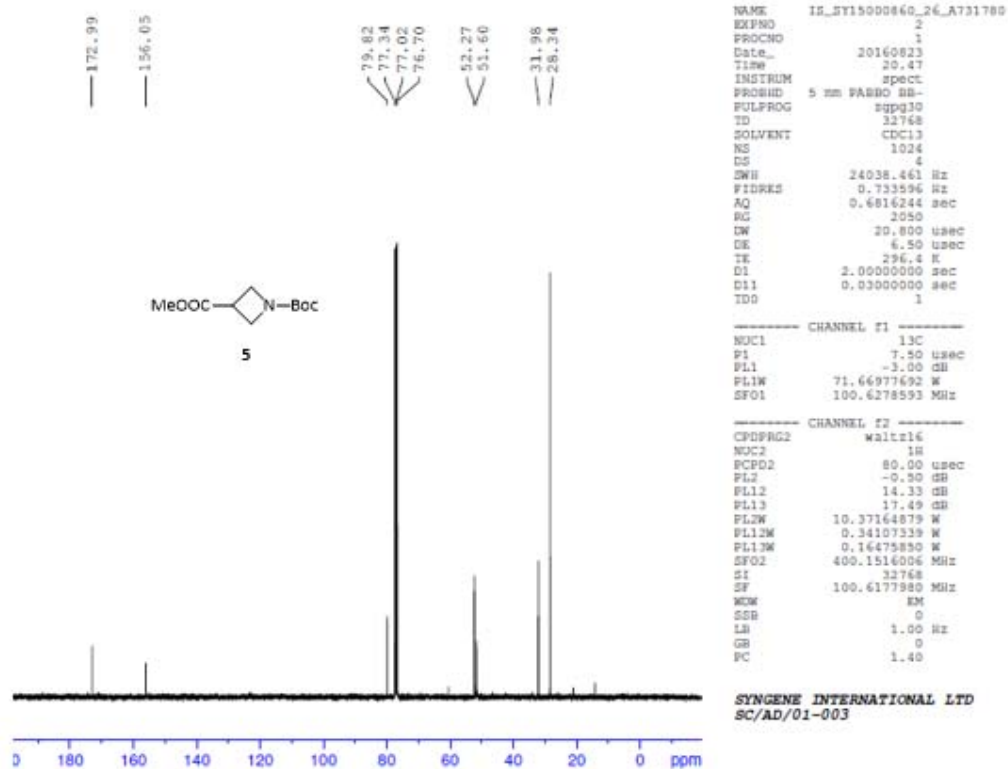
^[b] Small Molecule Design and Development, Lilly Research Laboratories, Eli Lilly and Company, Indianapolis, Indiana 46285, USA.

Supporting Information

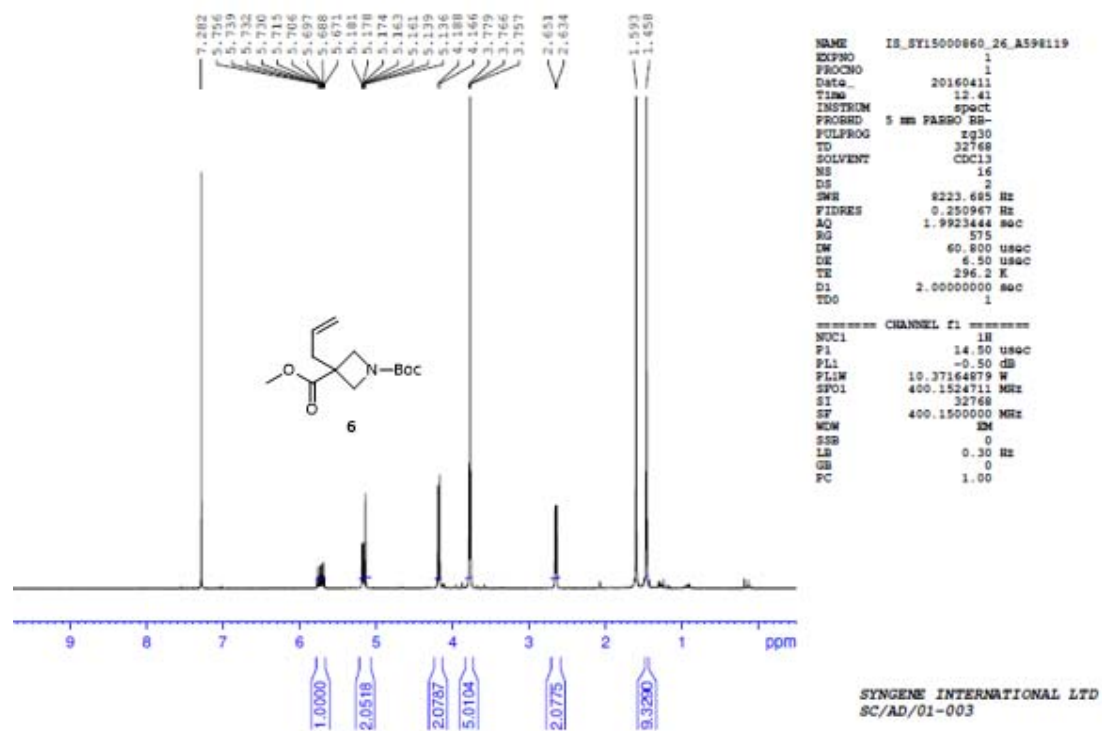
¹HNMR spectrum of Compound 5



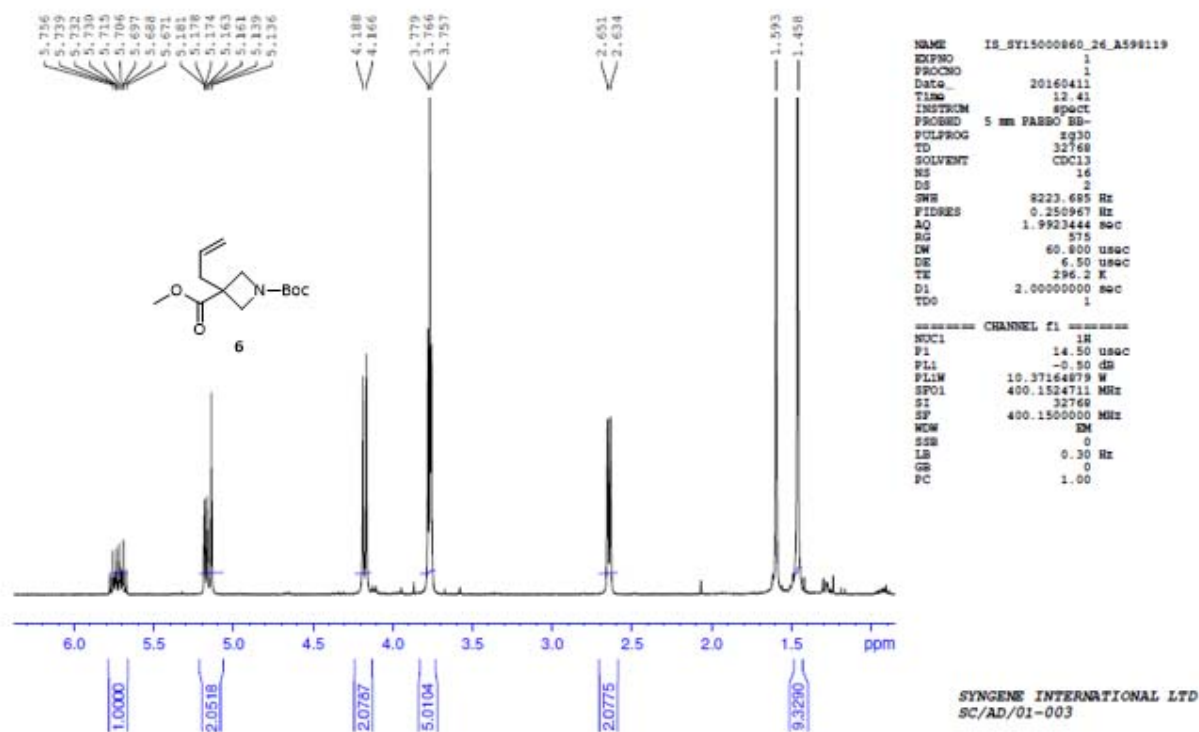
¹³C NMR Spectrum of Compound 5



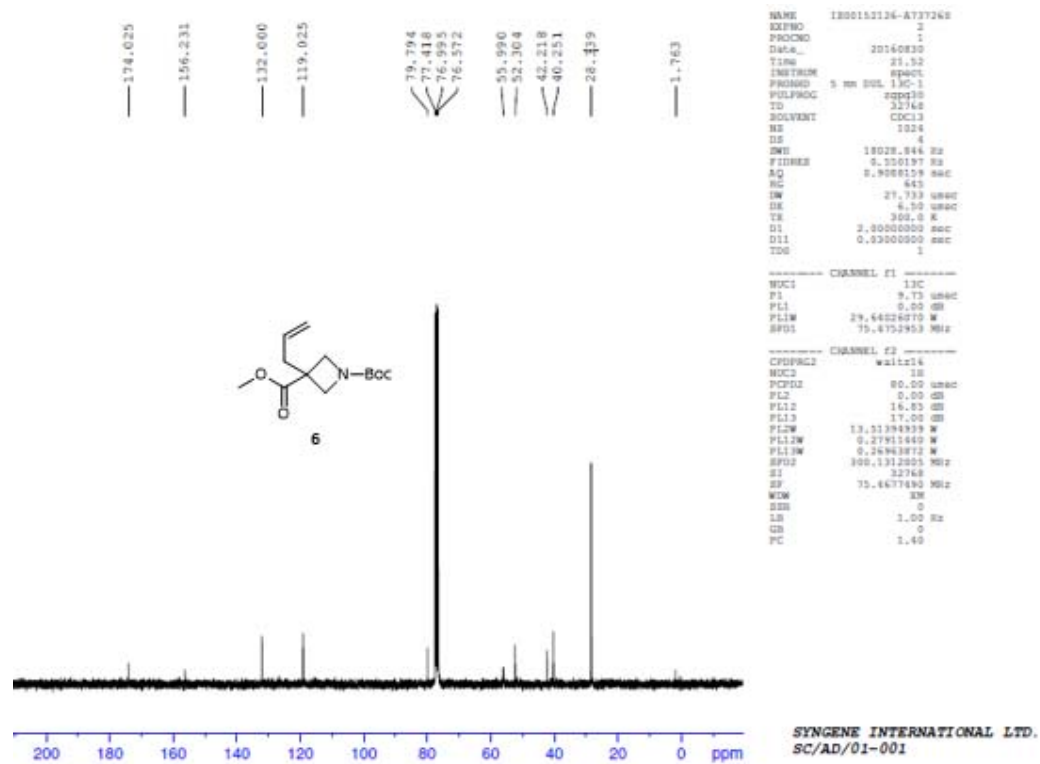
¹HNMR Spectrum of Compound 6



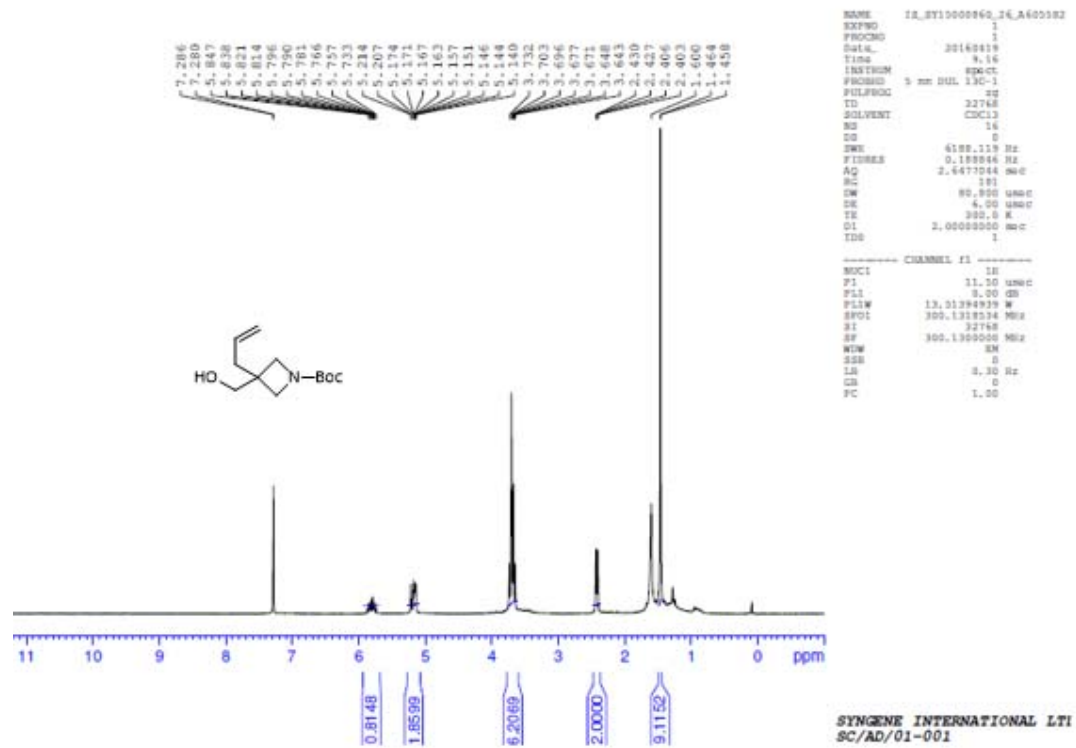
¹HNMR Spectrum (expansion) of Compound 6



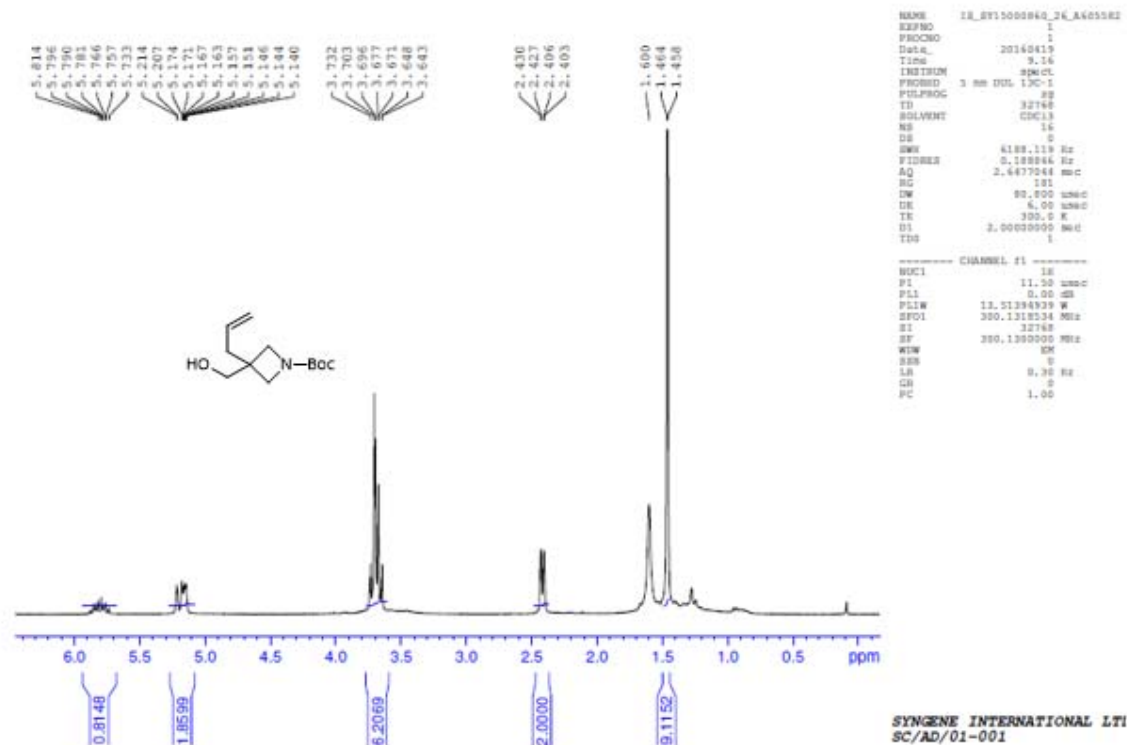
¹³C NMR Spectrum of Compound 6



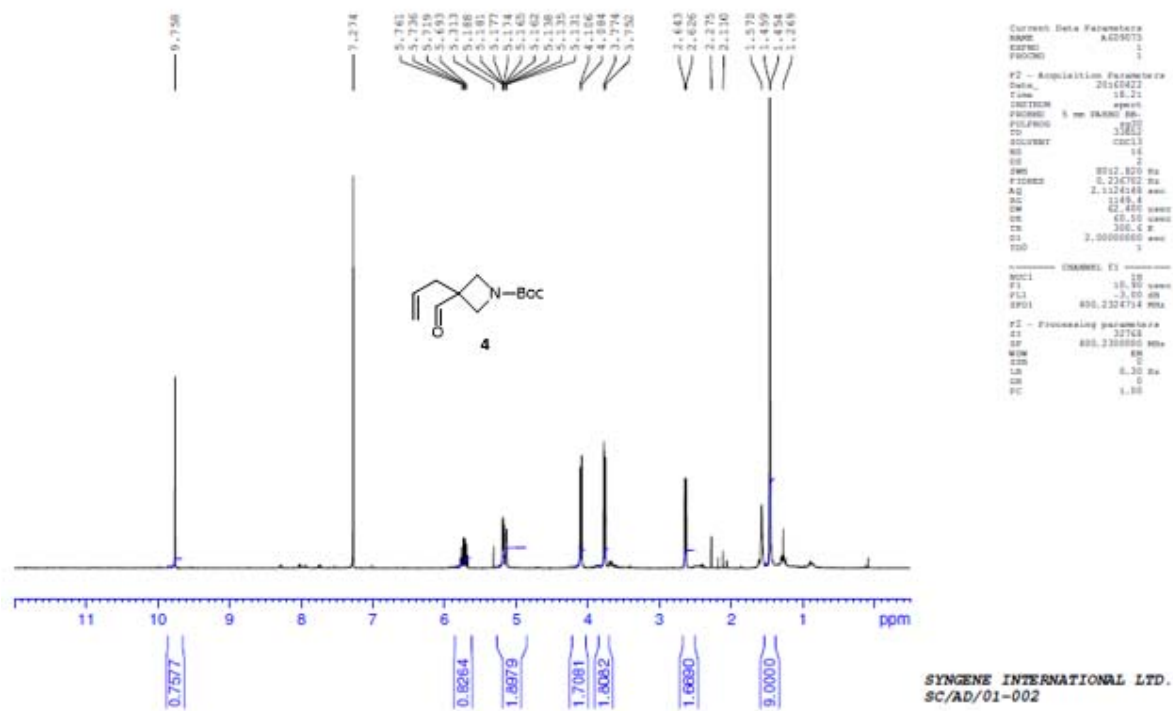
¹HNMR Spectrum of tert-butyl 3-allyl-3-(hydroxymethyl)azetidine-1-carboxylate



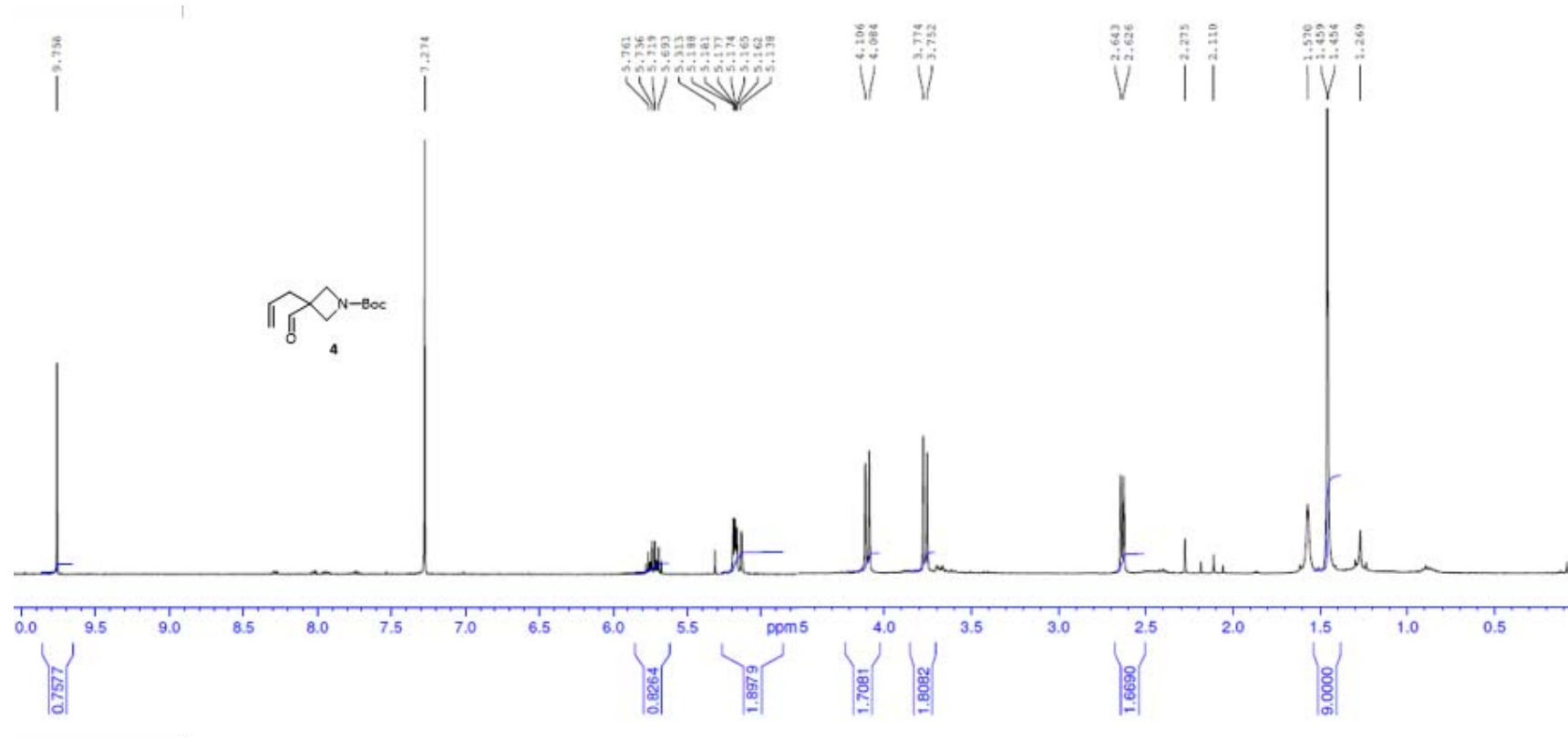
¹HNMR Spectrum (expansion) of tert-butyl 3-allyl-3-(hydroxymethyl)azetidine-1-carboxylate



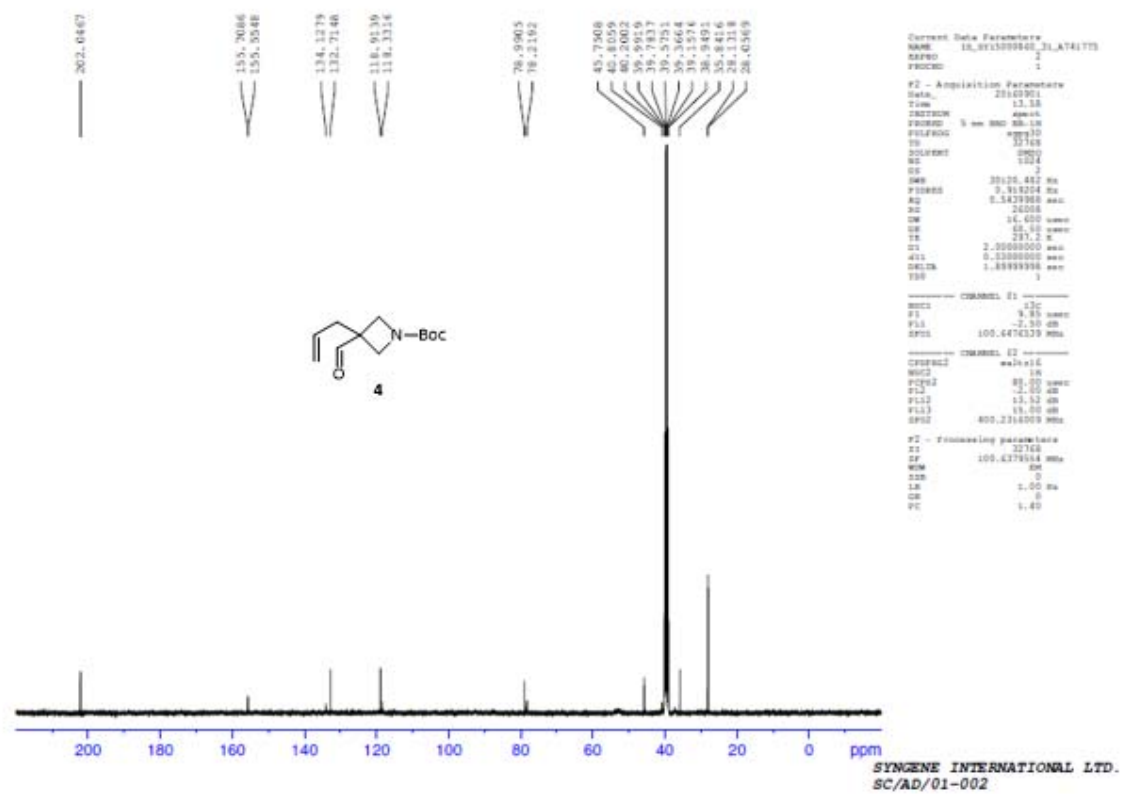
¹HNMR Spectrum of Compound 4



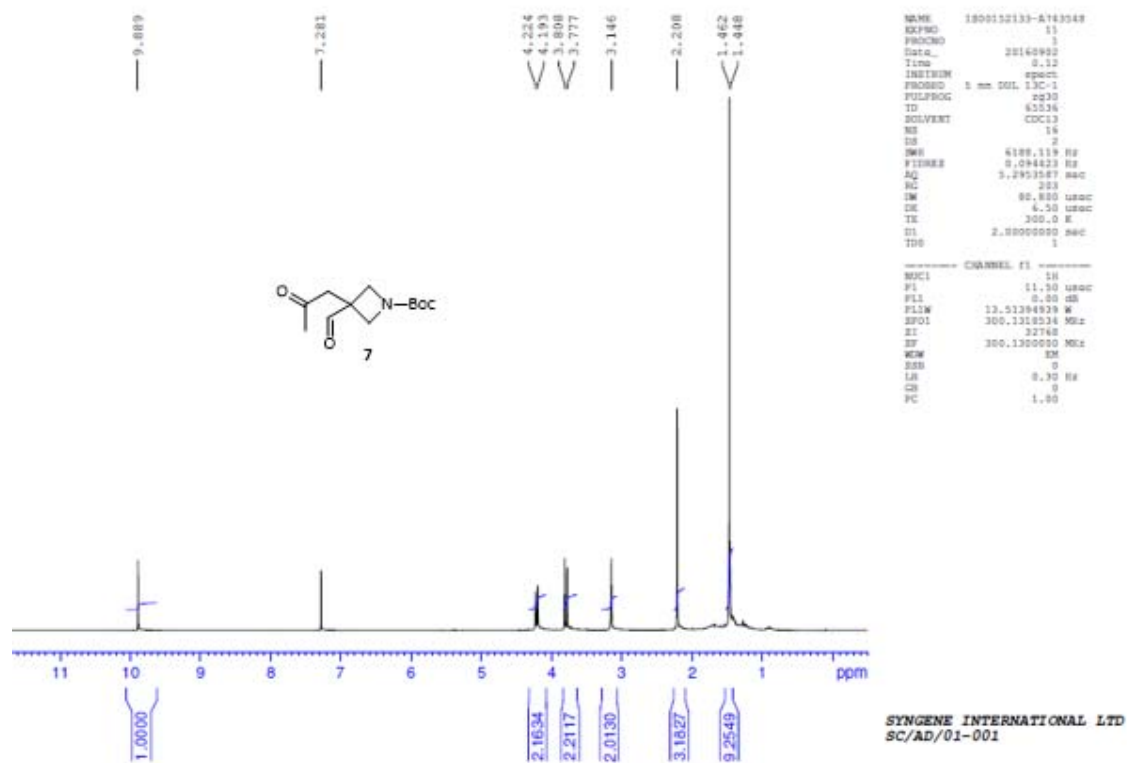
¹HNMR Spectrum (expansion) of Compound 4



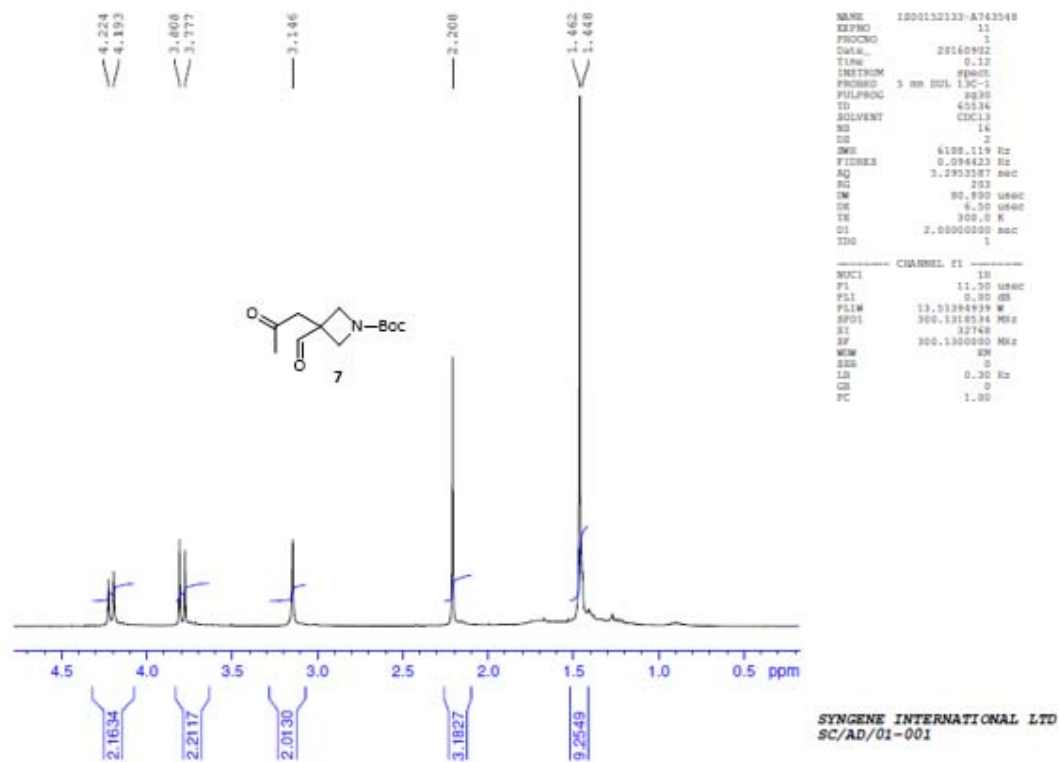
¹³C NMR Spectrum of Compound 4



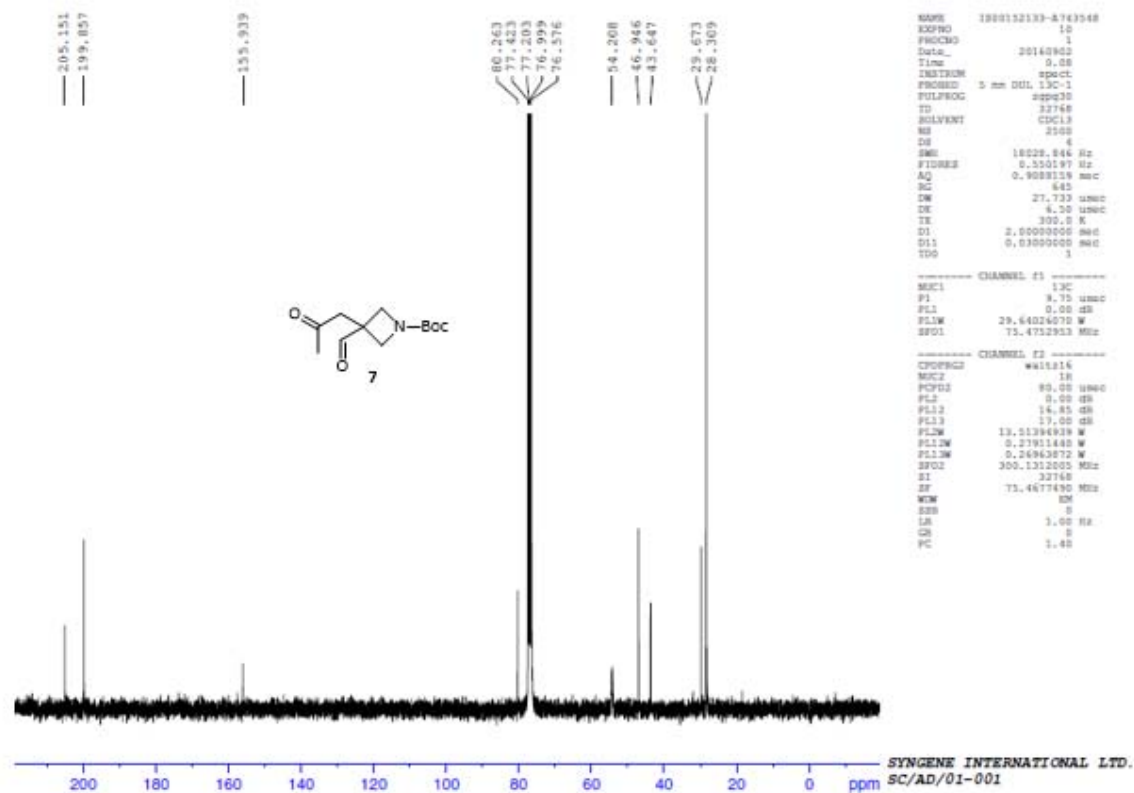
¹HNMR Spectrum of Compound 7



¹HNMR Spectrum (expansion) of Compound 7



¹³C NMR Spectrum of Compound 7



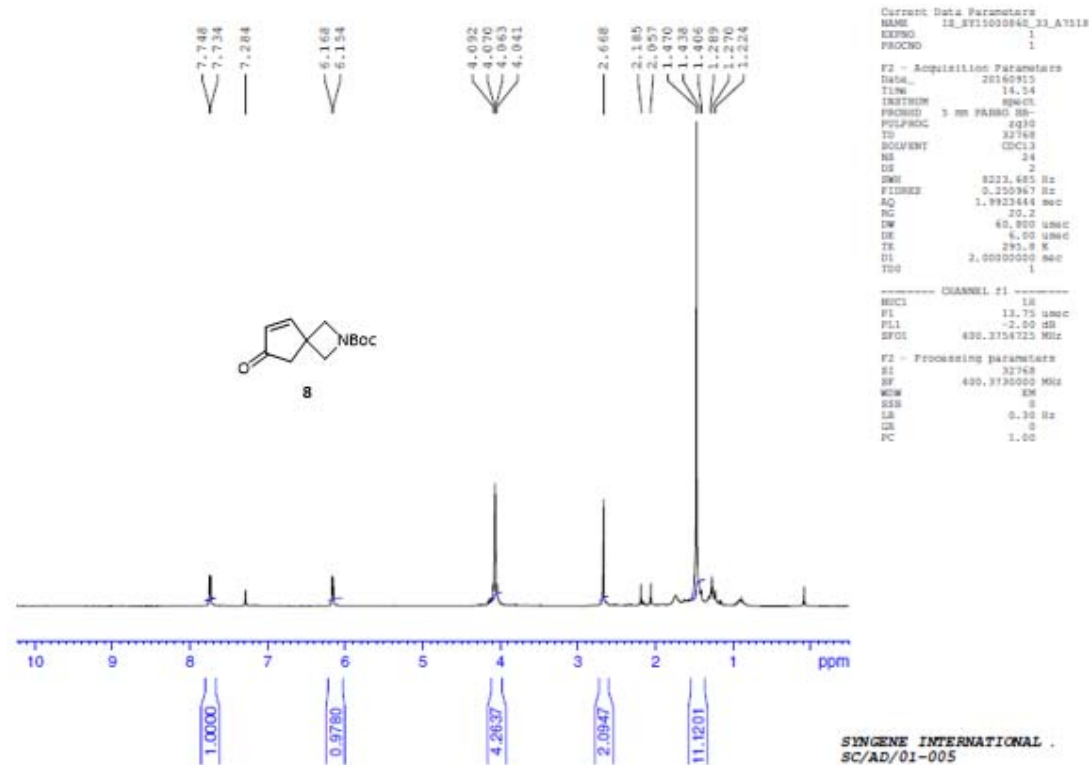
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PROCNO    1
Data_     20140802
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INSTRUM    spect
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PULPROG    zgpg30
TD         32768
SOLVENT    CDCl3
NS         2500
DS         4
SWH         18028.846 Hz
FIDRES     0.150197 Hz
AQ         0.9088138 sec
RG         640
DW         27.732 usec
DE         4.50 usec
TE         300.2 K
D1         2.00000000 sec
d11        0.03000000 sec
TD0        1

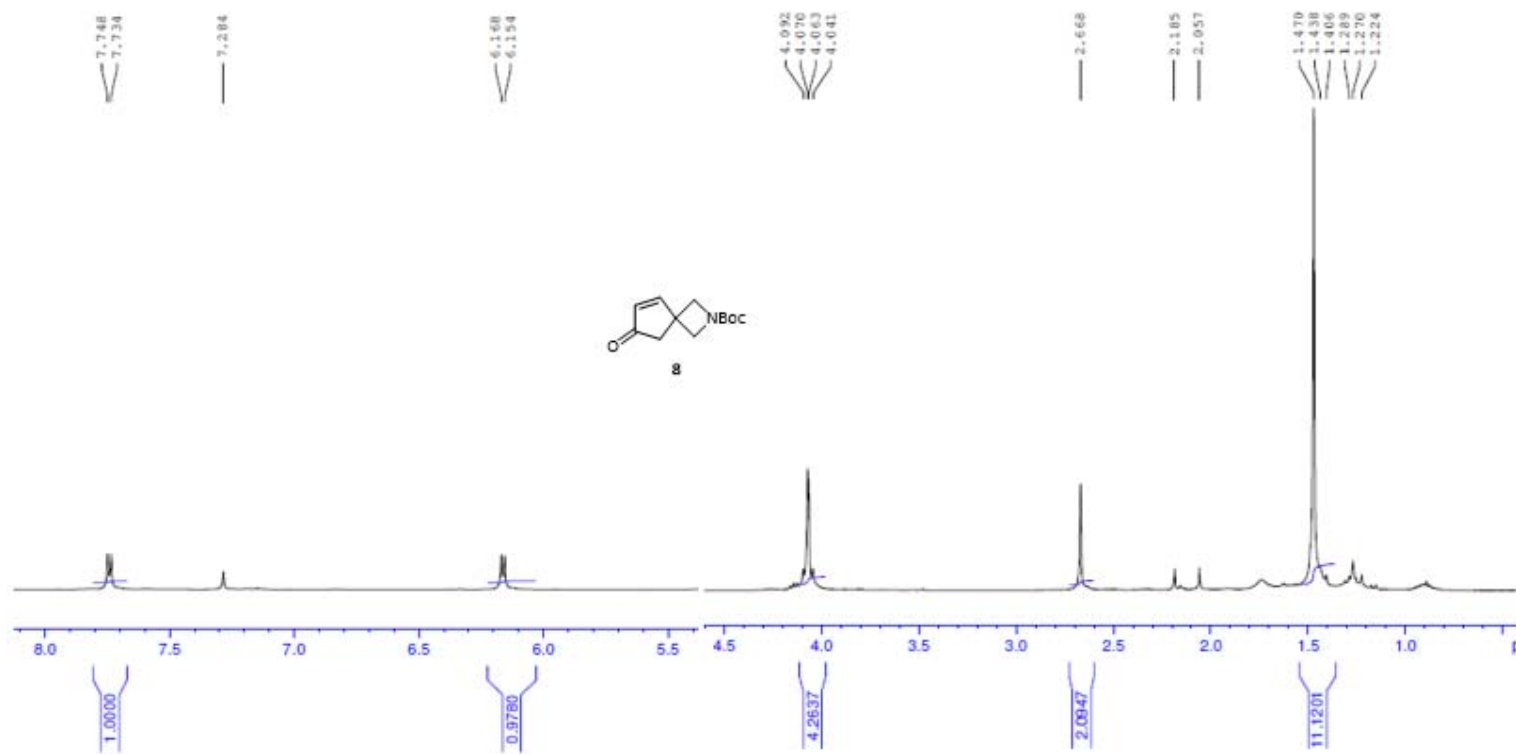
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NUC1       13C
P1         8.70 usec
PL1        0.00 dB
PL1W       29.64024570 W
SFO1       75.4752953 MHz

----- CHANNEL f2 -----
CPDPRG2    waltz16
NUC2       1H
PCPD2      80.00 usec
PL2        0.00 dB
PL12       16.85 dB
PL13       17.00 dB
PL1W       13.51394939 W
PL12W      0.27951480 W
PL13W      0.26963072 W
SFO2       300.1352605 MHz
SI         32768
SF         75.4677490 MHz
WCM        RM
SFO        0
LA         1.00 Hz
GB         0
PC         1.40
    
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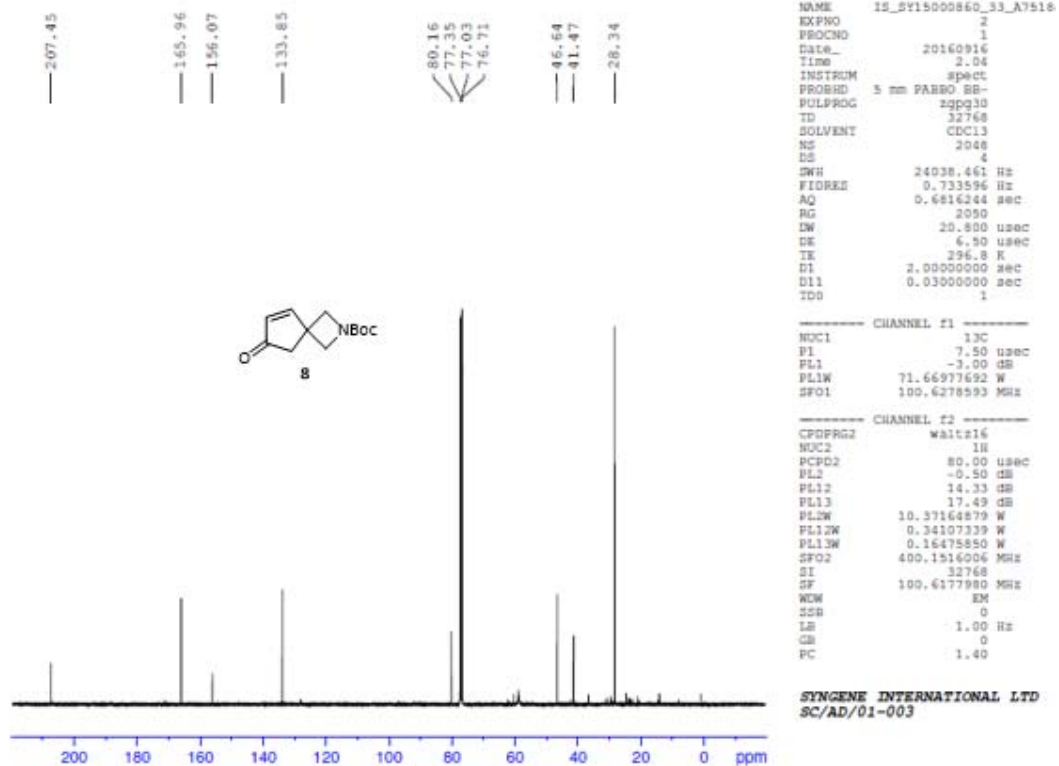
¹HNMR Spectrum of Compound 8



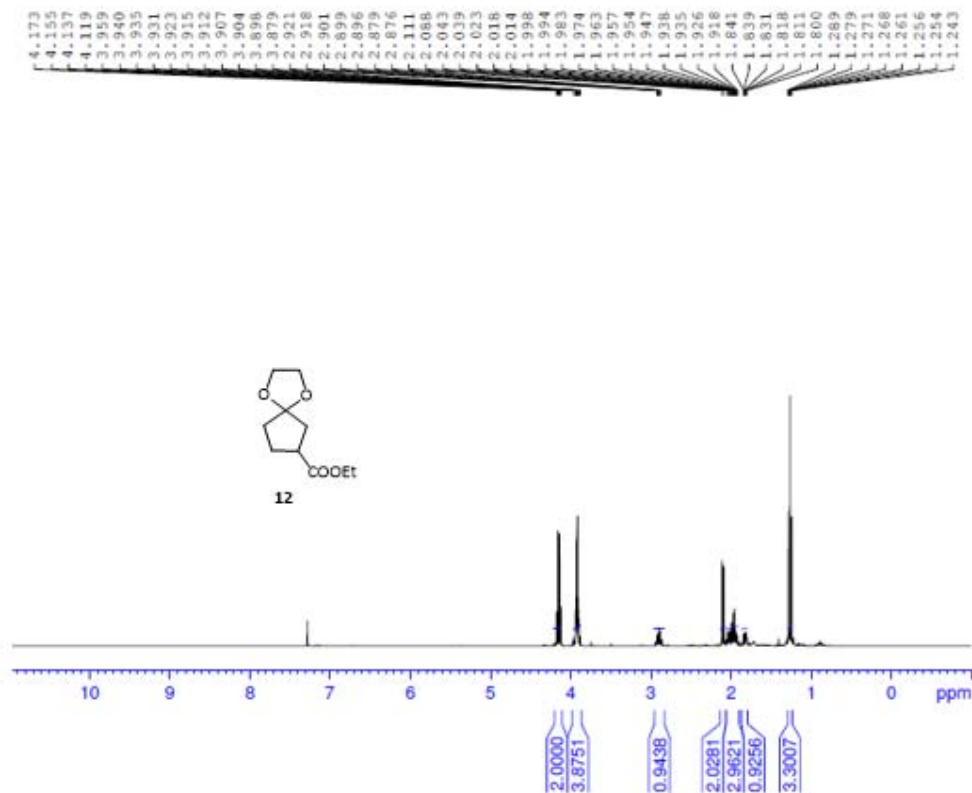
¹HNMR Spectrum (expansion) of Compound 8



¹³C NMR Spectrum of Compound 8



¹H NMR Spectrum of Compound 12

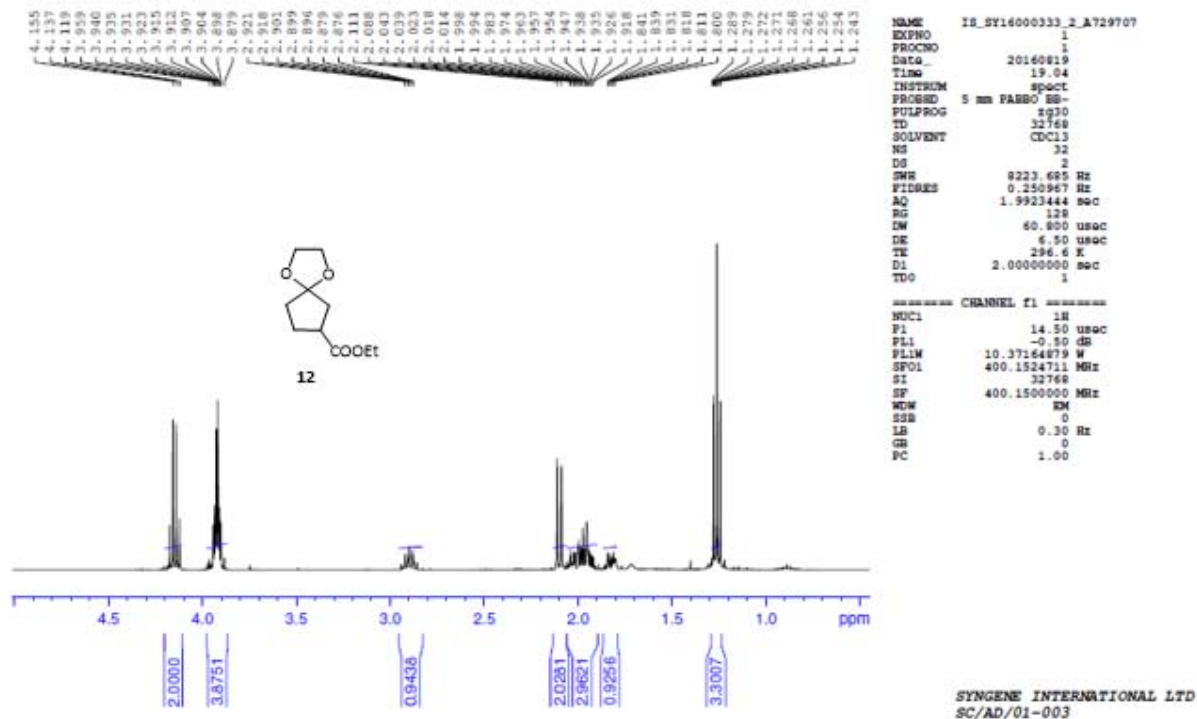


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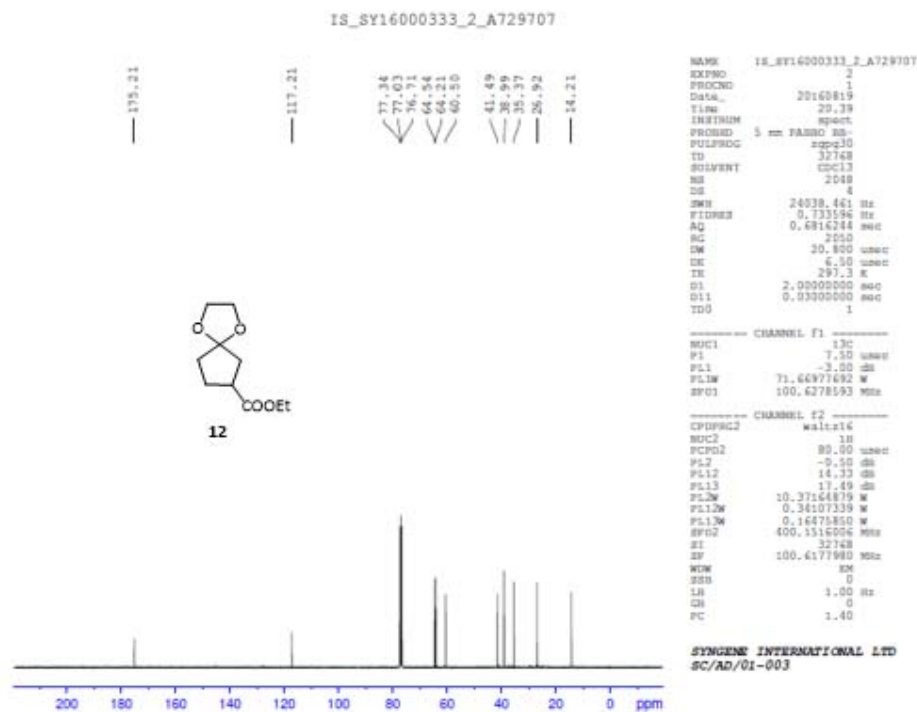
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EXPNO     1
PROCNO    1
Date_     20160819
Time      19.04
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD        32768
SOLVENT   CDCl3
NS        32
DS        2
SWH        8223.685 Hz
FIDRES     0.250967 Hz
AQ         1.9923444 sec
RG         128
DW         60.800 usec
DE         6.50 usec
TE         296.6 K
D1         2.00000000 sec
D10        1
===== CHANNEL f1 =====
NUC1       1H
P1         14.50 usec
PL1        -0.50 dB
PL1W       10.37164879 W
SFO1       400.1524711 MHz
SI         32768
SF         400.1500000 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
  
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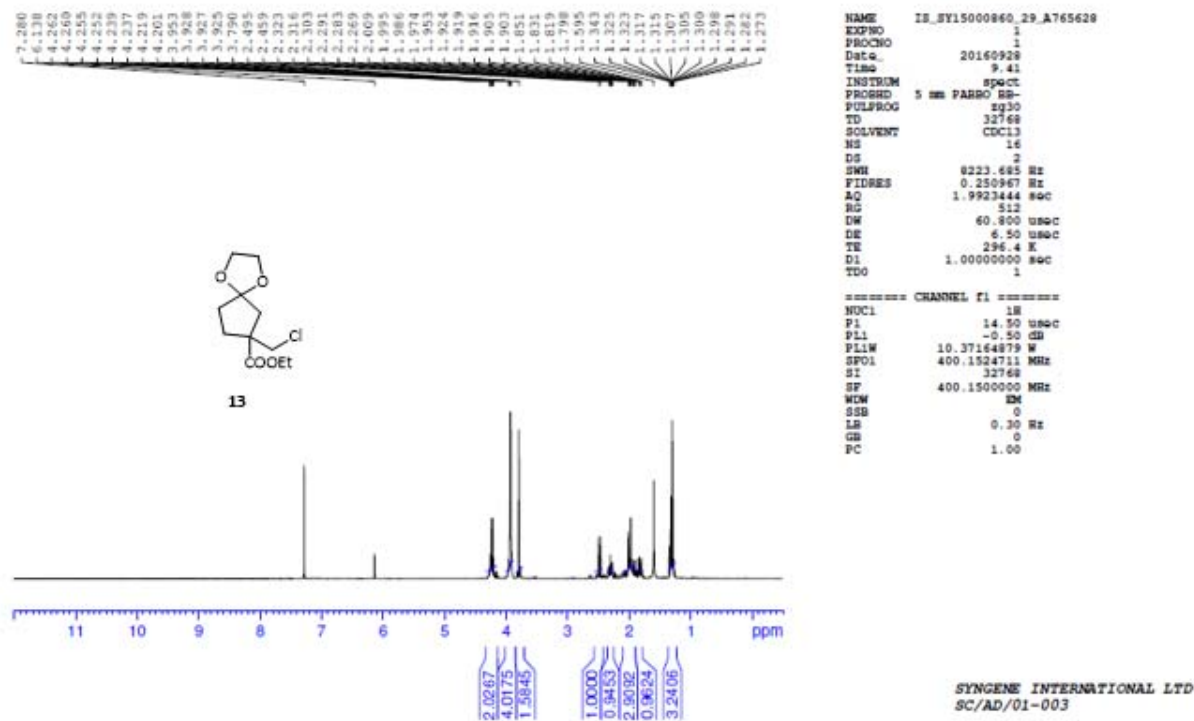
¹HNMR Spectrum (expansion) of Compound 12



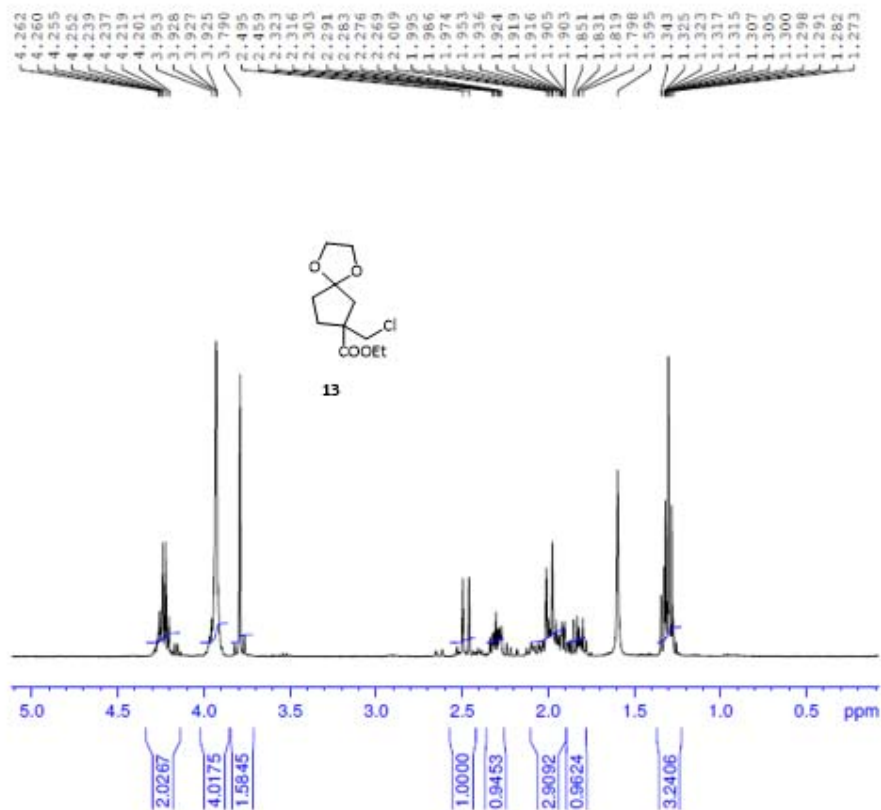
¹³C NMR Spectrum of Compound 12



¹HNMR Spectrum of Compound 13



¹HNMR Spectrum (expansion) of Compound 13

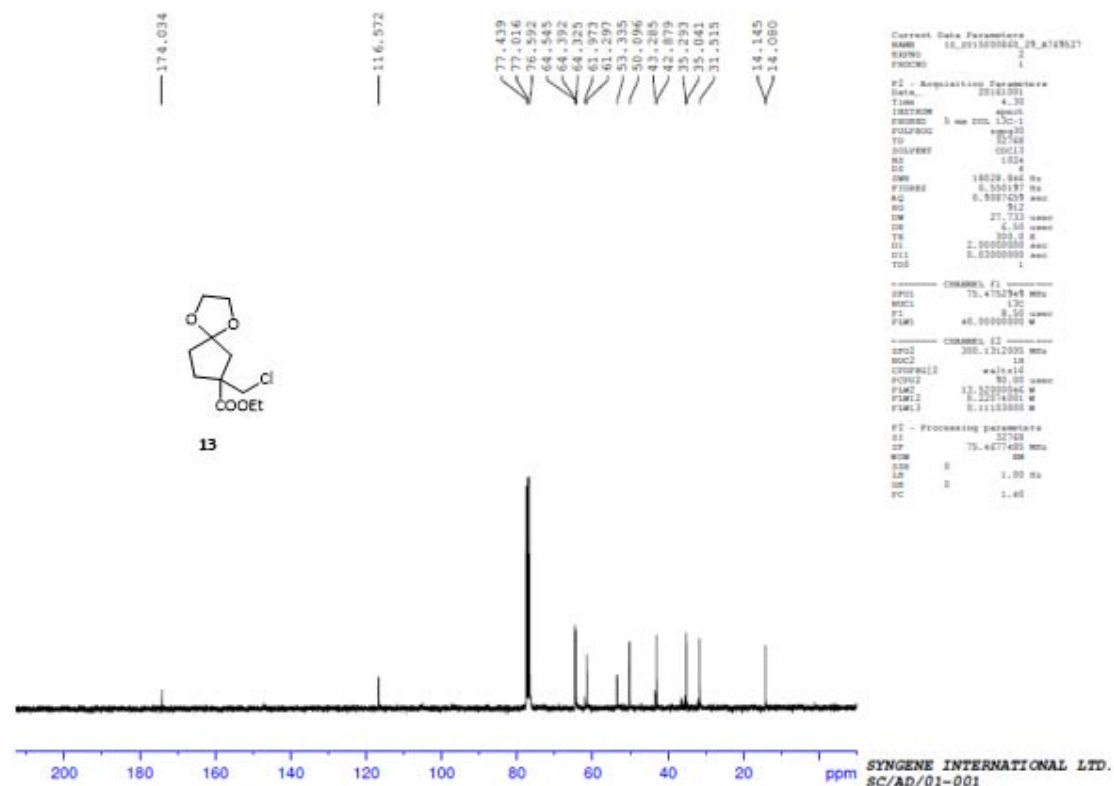


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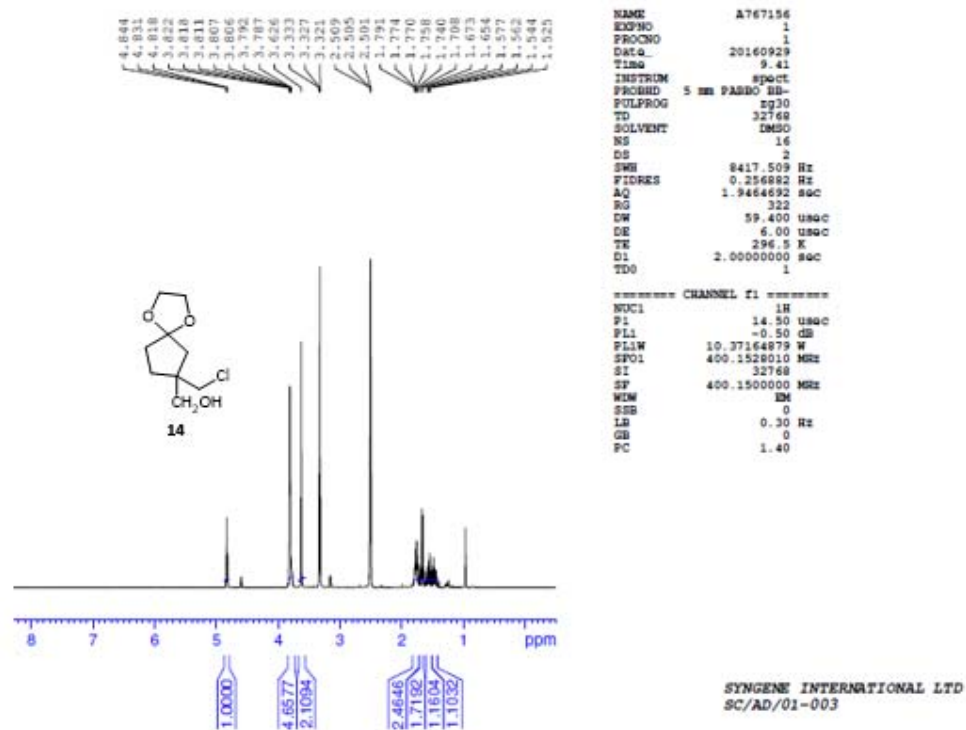
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EXPNO     1
PROCNO    1
Date_     20160928
Time      9.41
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD        32768
SOLVENT   CDCl3
NS        16
DS        2
SWH       8223.685 Hz
FIDRES    0.250967 Hz
AQ        1.9923444 sec
RG        512
DW        60.800 usec
DE        6.50 usec
TE        296.4 K
D1        1.00000000 sec
TDO       1
===== CHANNEL f1 =====
NUC1      1H
P1        14.50 usec
PL1       -0.50 dB
PL1W      10.37164879 W
SFO1      400.1524711 MHz
SI        32768
SF        400.1500000 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
    
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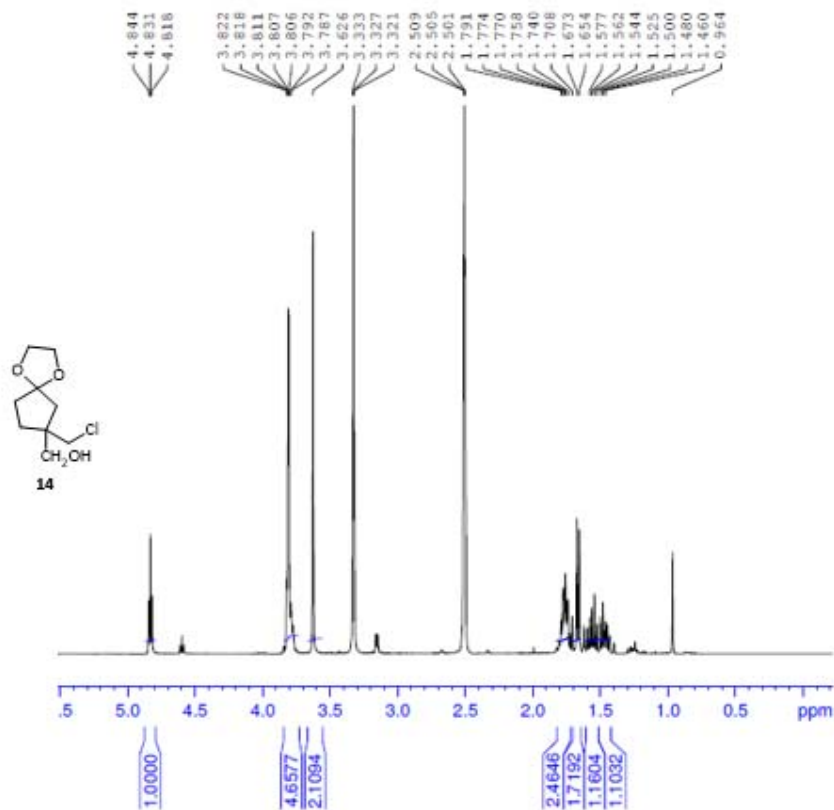
¹³C NMR Spectrum of Compound 13



¹HNMR Spectrum of Compound 14



¹H NMR Spectrum (expansion) of Compound 14



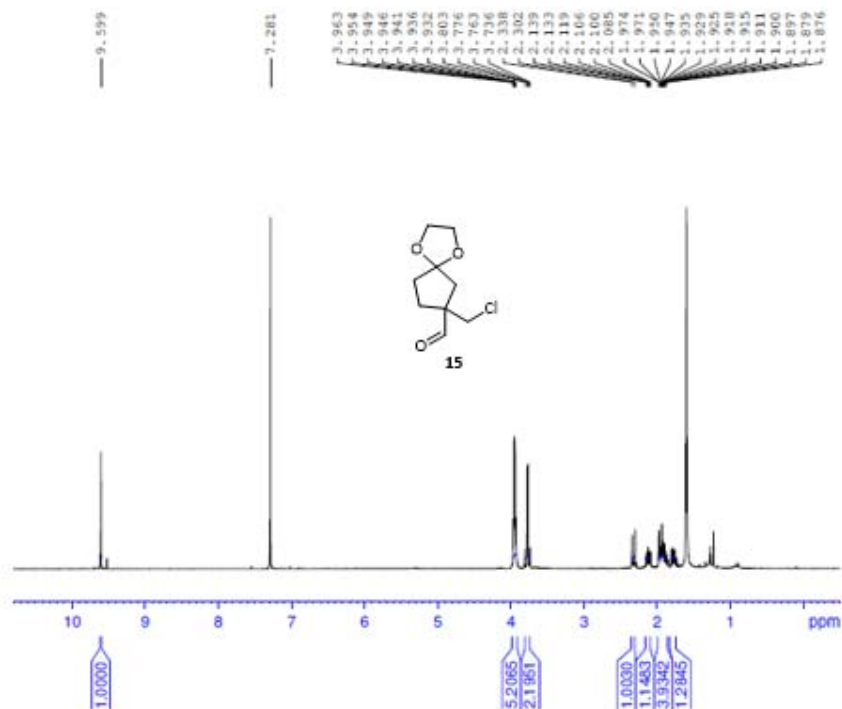
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PROCNO        1
Date_         20160929
Time          9.41
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            32768
SOLVENT       DMSO
NS            16
DS            2
SWH           8417.509 Hz
FIDRES        0.256882 Hz
AQ            1.9464692 sec
RG            322
IW            59.400 usec
DE            6.00 usec
TE            296.5 K
D1            2.00000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          1H
P1            14.50 usec
PL1           -0.50 dB
PL1W          10.37164879 W
SFO1          400.1528010 MHz
SI            32768
SF            400.1500000 MHz
WDW            EM
SSB            0
LB            0.30 Hz
GB            0
PC            1.40
    
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¹HNMR Spectrum of Compound 15

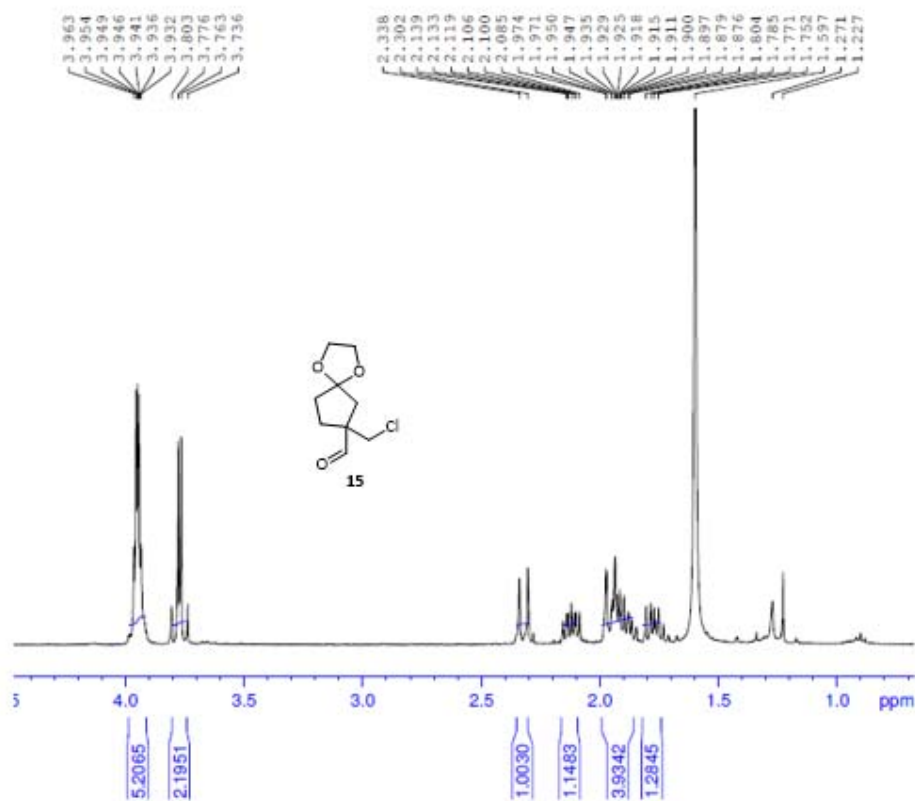


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PROCNO        1
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TIME          10.11
INSTRUM       spect
PROBHD        5 mm PABBO MS-
PULPROG       zg30
TD            32768
SOLVENT       CDCl3
MS            64
DS            2
SWH           6223.685 Hz
FIDRES        0.250967 Hz
AQ            1.9923444 sec
RG            575
WM            60.900 usec
DE            6.50 usec
TE            295.7 K
D1            1.00000000 sec
TD0           1
===== CHANNEL f1 =====
NUC1          1H
P1            14.50 usec
PL1           -0.50 dB
PL1W          10.37164879 W
SFO1          400.1524711 MHz
SI            32768
SF            400.1500000 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
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¹HNMR Spectrum (expansion) of Compound 15

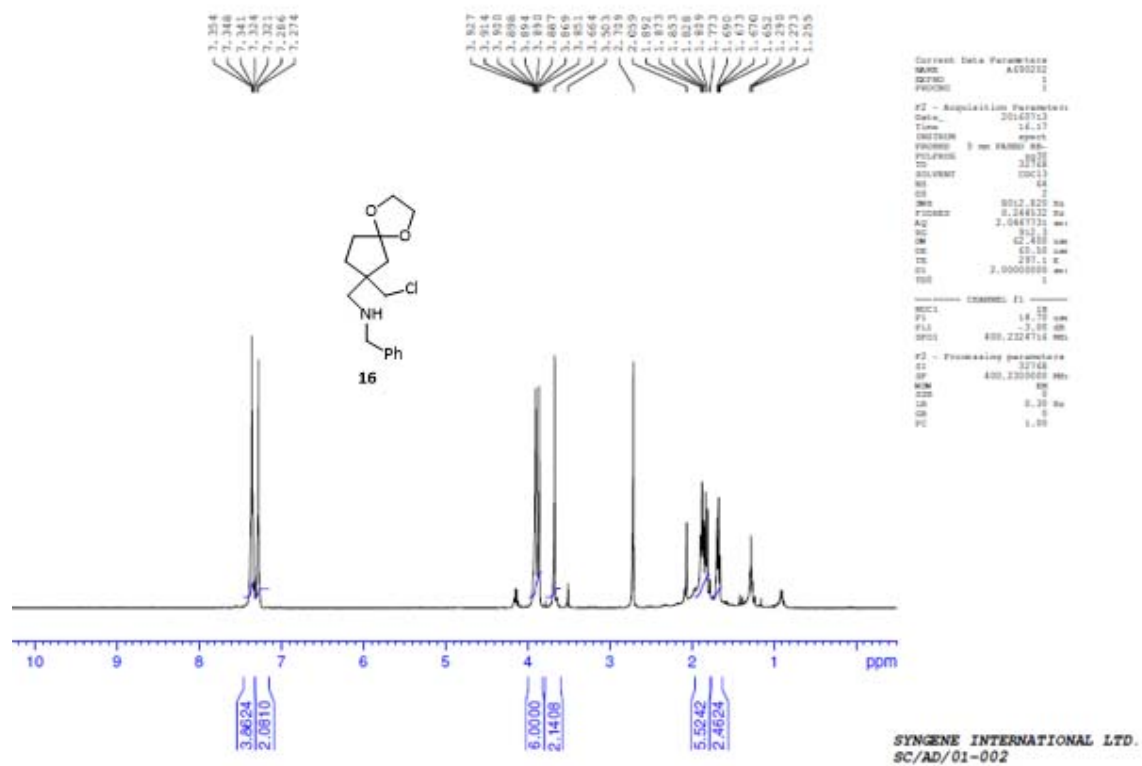


NAME A680733
 EXPNO 1
 PROCNO 1
 Date_ 20160704
 Time 10.11
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 64
 DS 2
 SWH 8223.685 Hz
 FIDRES 0.250967 Hz
 AQ 1.9923444 sec
 RG 575
 DW 60.800 usec
 DE 6.50 usec
 TE 295.7 K
 D1 1.00000000 sec
 TD0 1

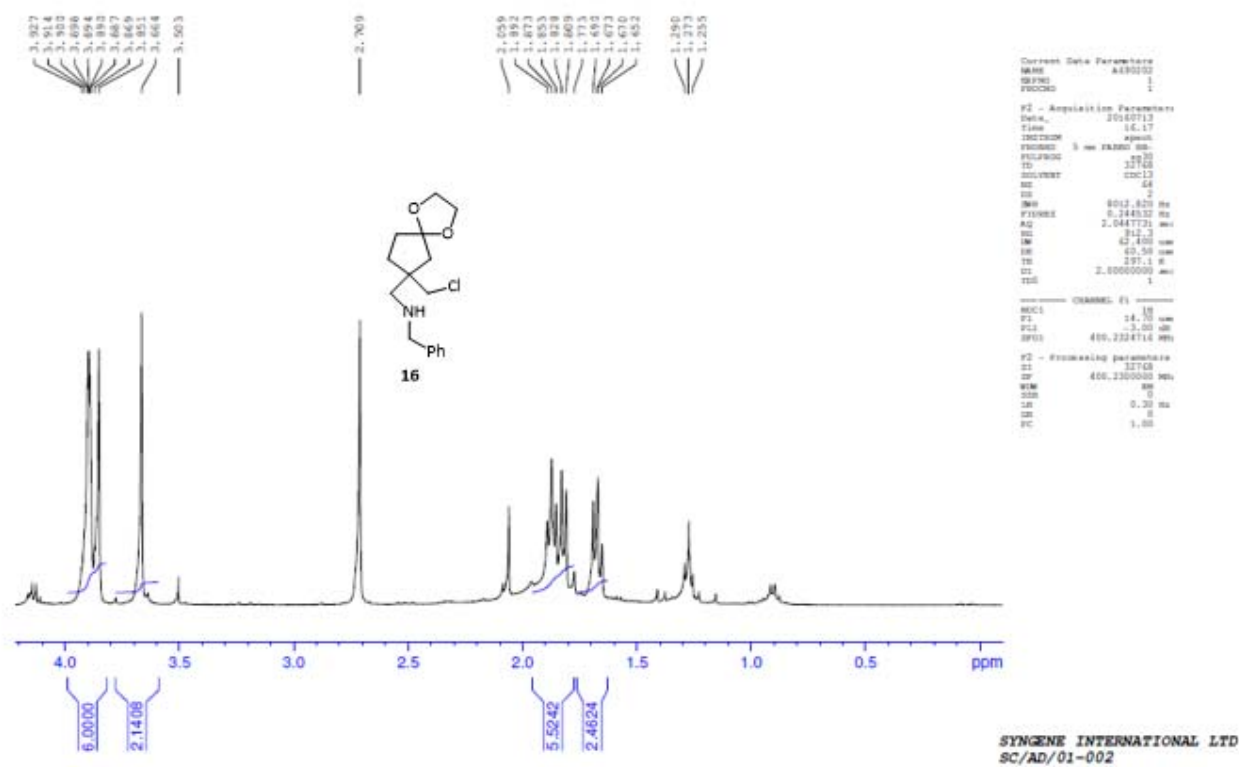
===== CHANNEL f1 =====
 NUC1 1H
 P1 14.50 usec
 PL1 -0.50 dB
 PL1W 10.37164879 W
 SFO1 400.1524711 MHz
 SI 32768
 SF 400.1500000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

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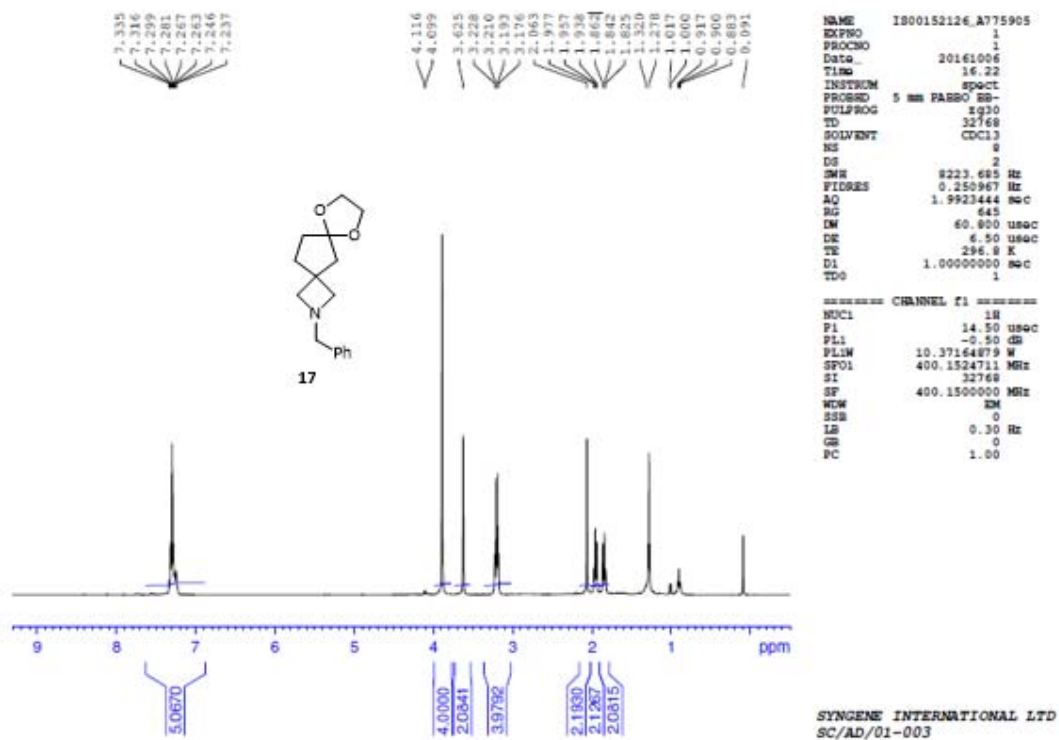
¹HNMR Spectrum of Compound 16



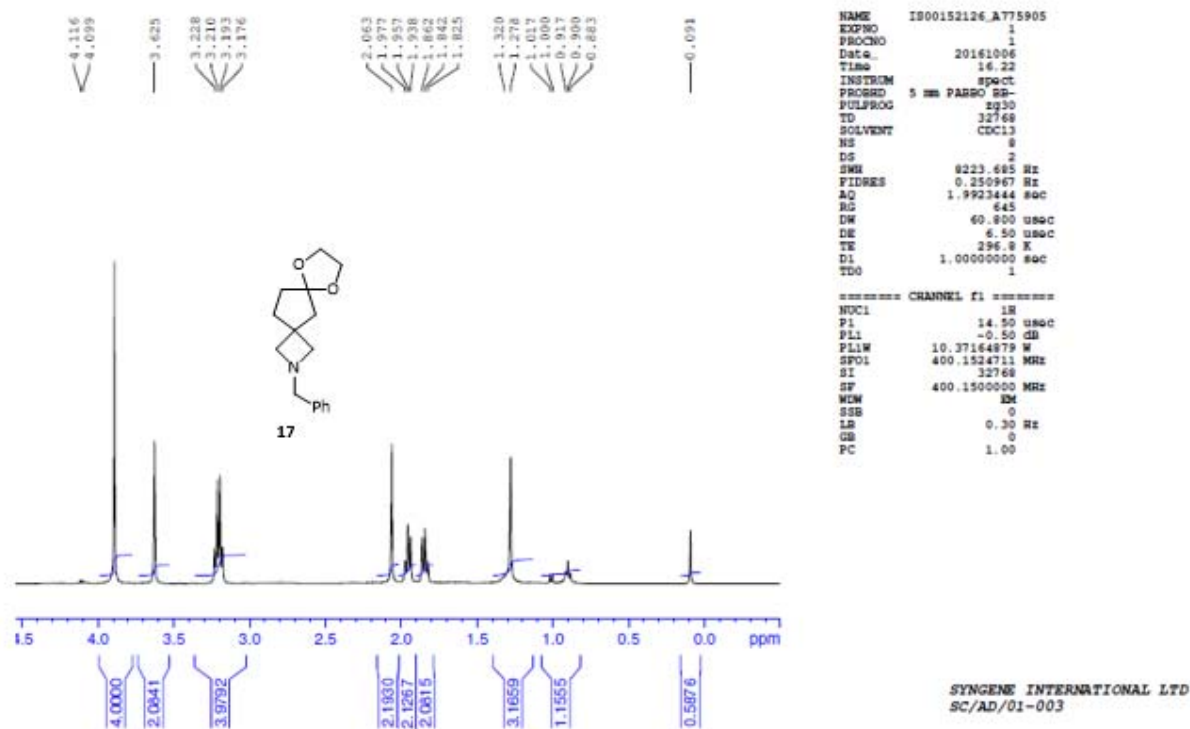
¹HNMR Spectrum (expansion) of Compound 16



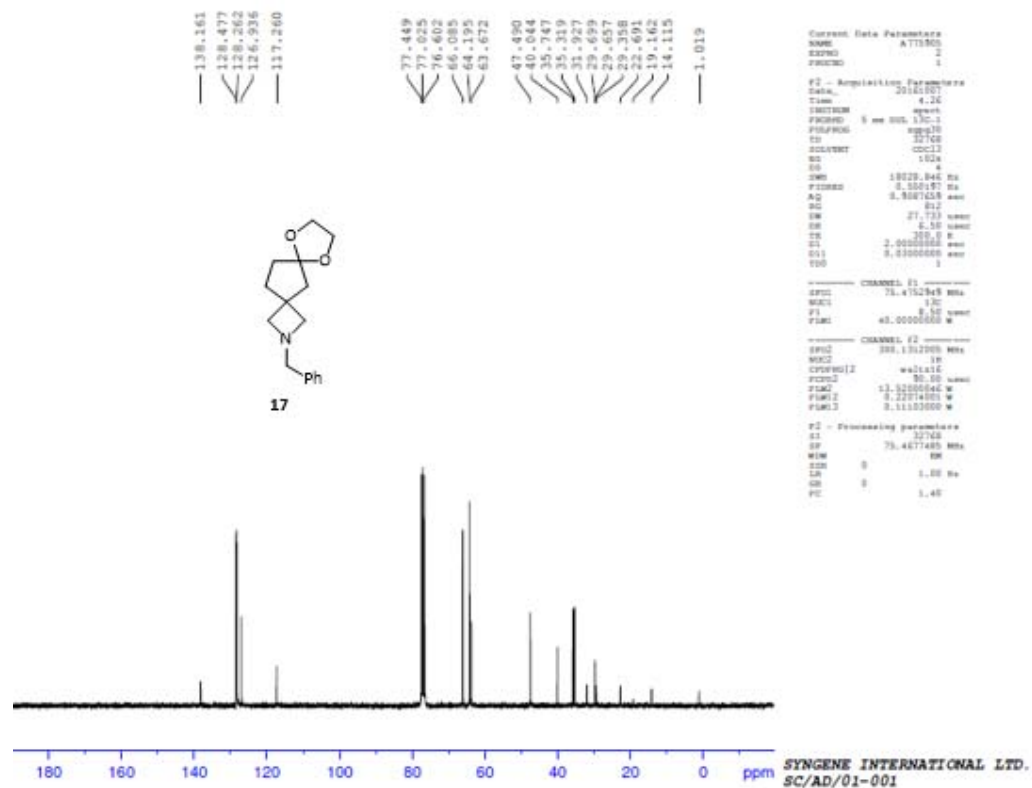
¹HNMR Spectrum of Compound 17



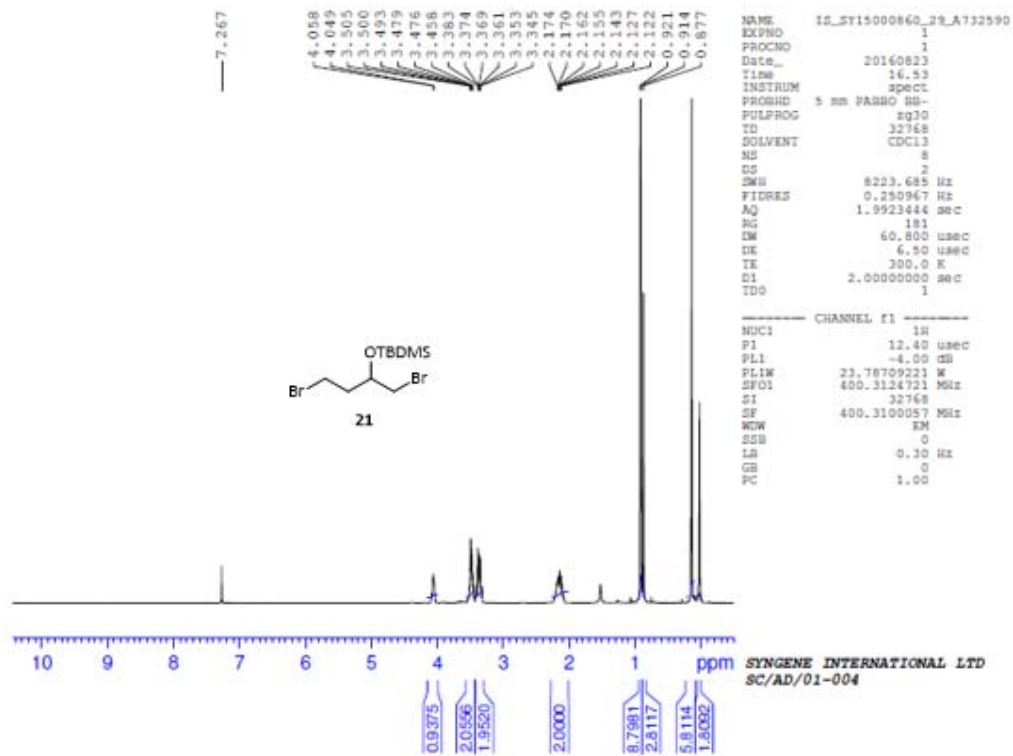
¹HNMR Spectrum (expansion) of Compound 17



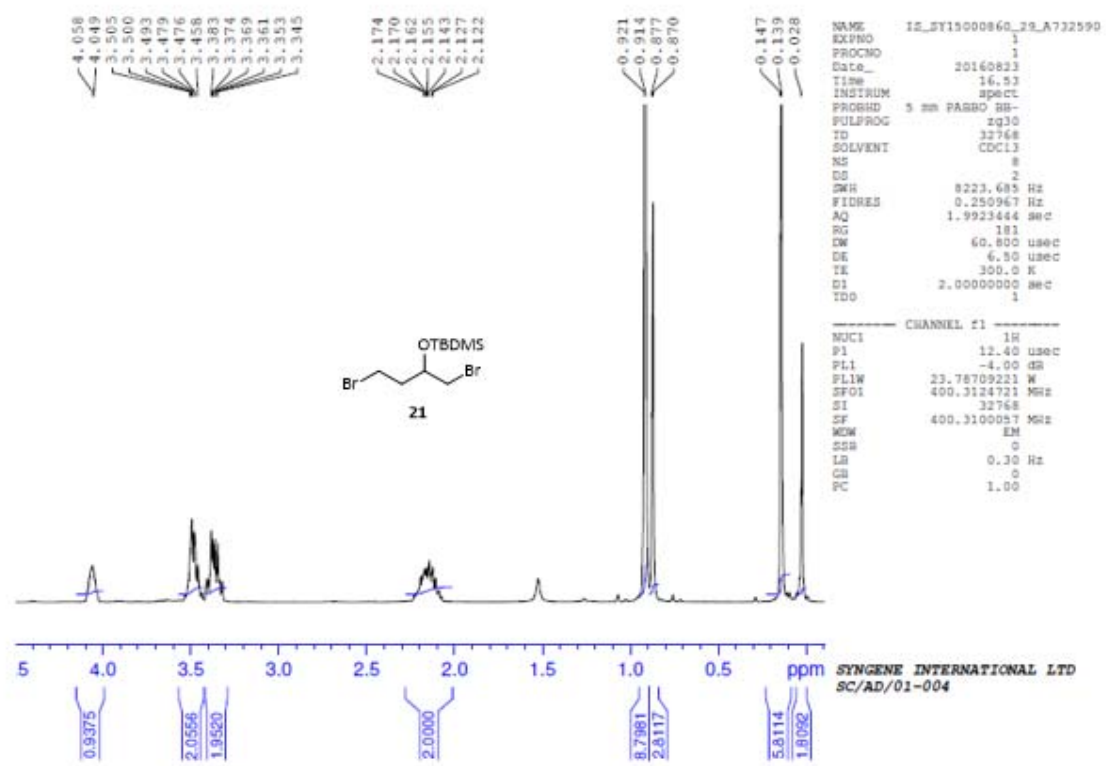
¹³C NMR Spectrum of Compound 17



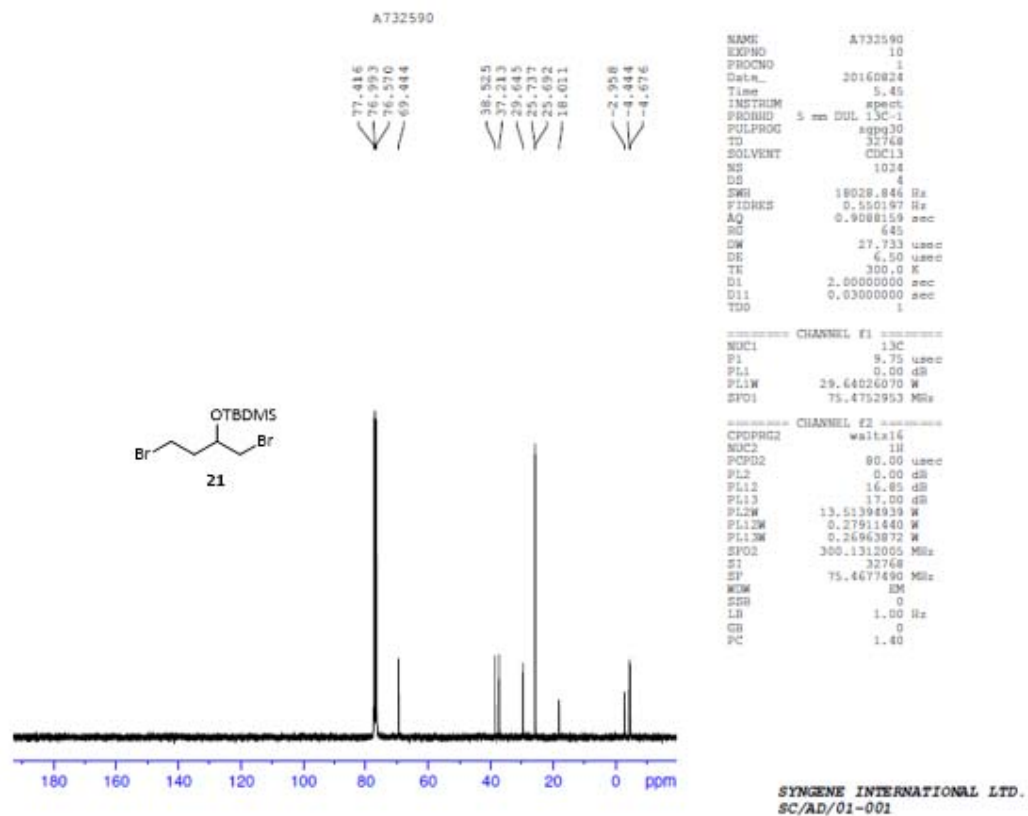
¹HNMR Spectrum of Compound 21



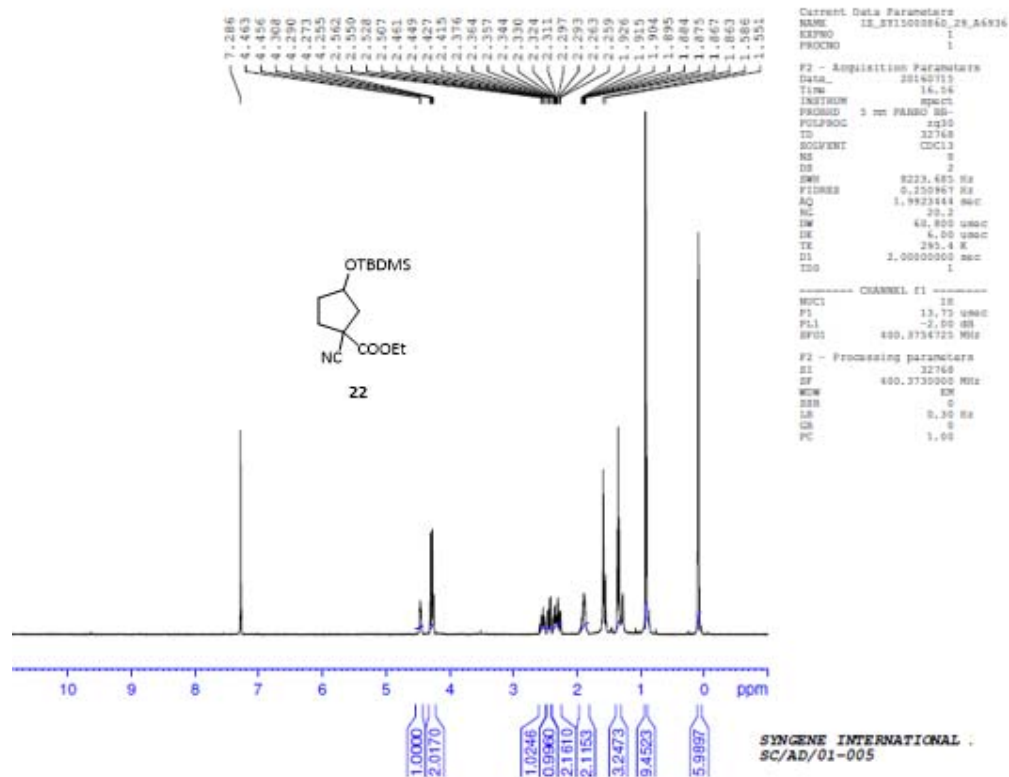
¹HNMR Spectrum (expansion) of Compound 21



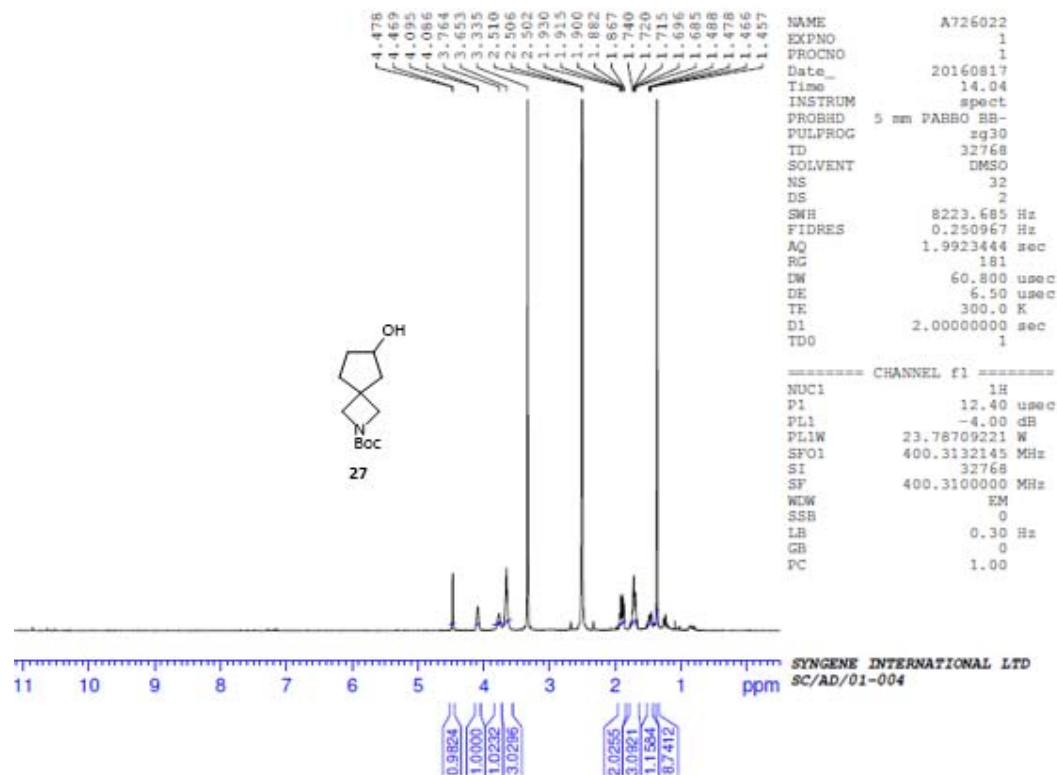
¹³C NMR Spectrum of Compound 21



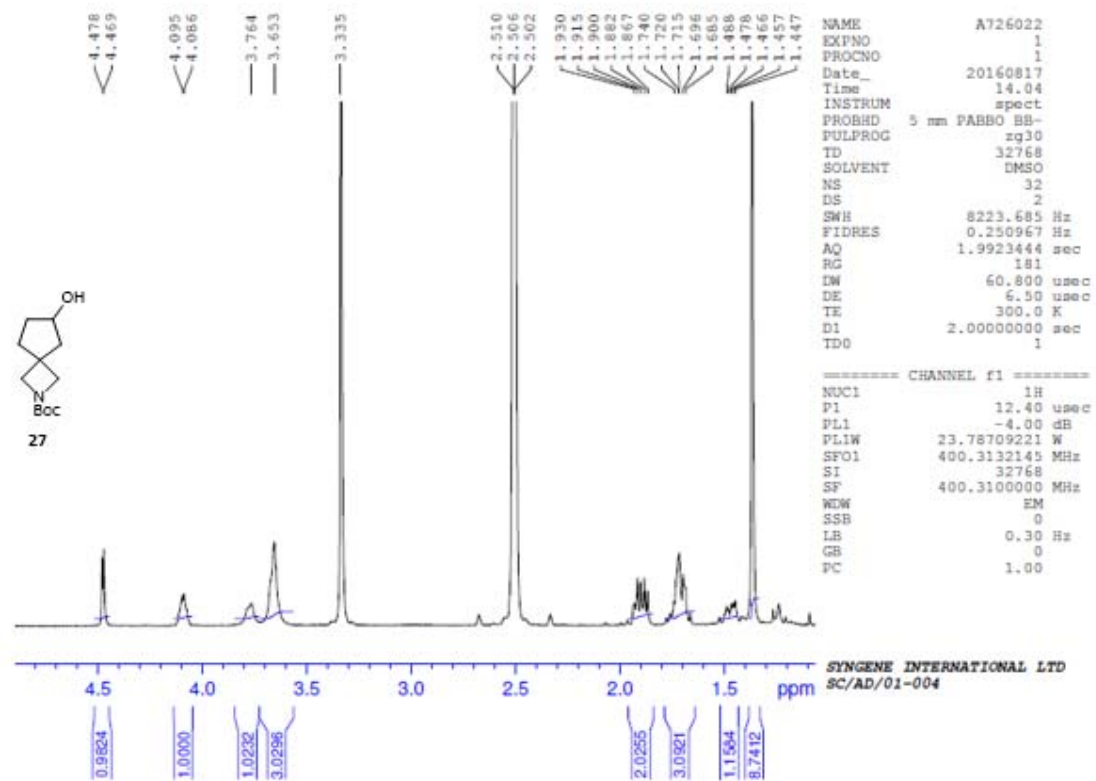
¹HNMR Spectrum of Compound 22



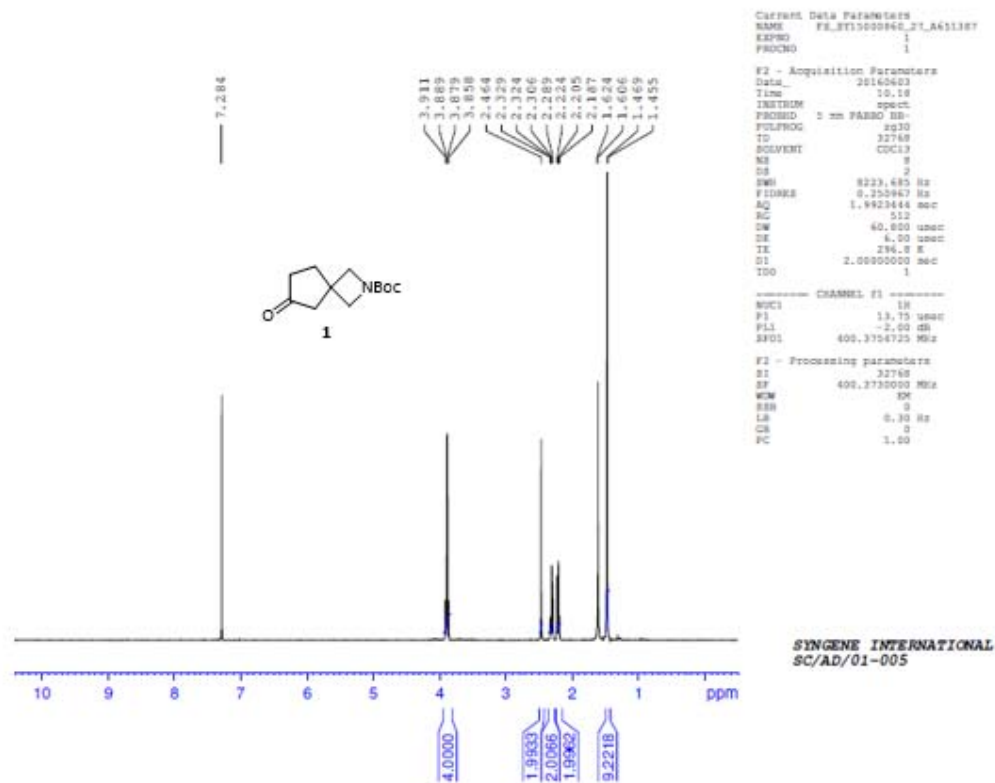
¹HNMR Spectrum of Compound 27



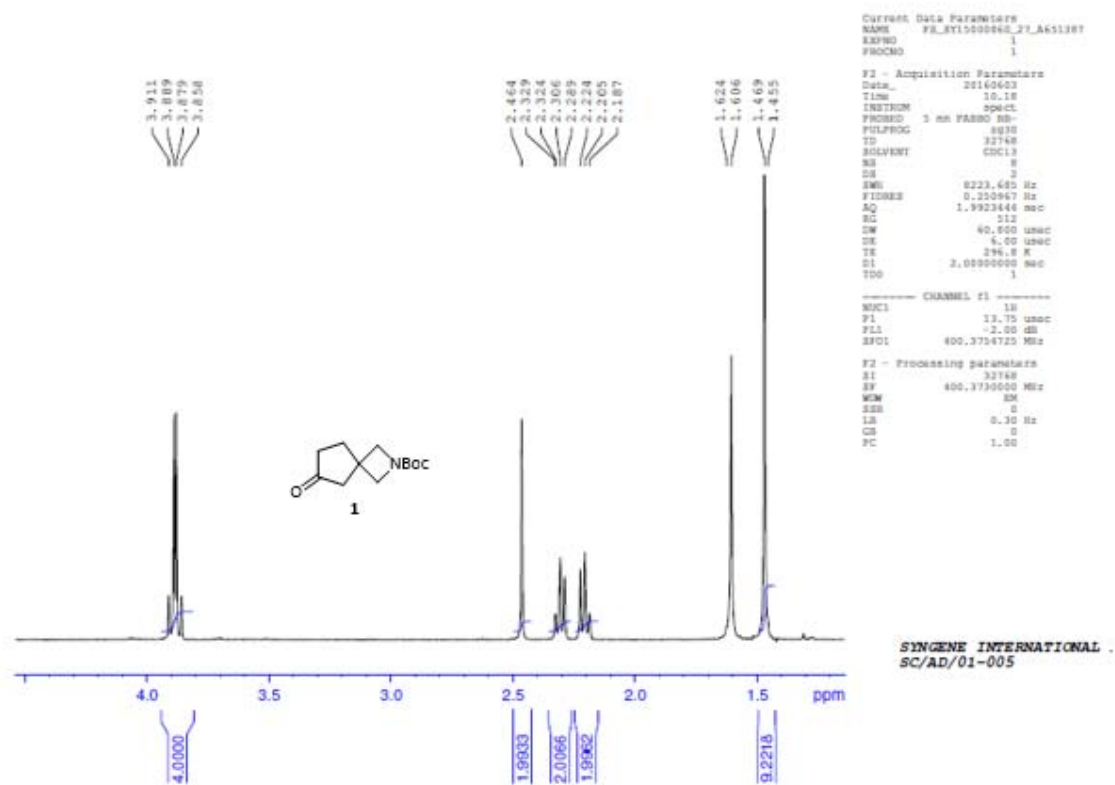
¹HNMR Spectrum (expansion) of Compound 27



¹HNMR Spectrum of Compound 1



¹HNMR Spectrum (expansion) of Compound 1



¹³C NMR Spectrum of Compound 1

