

## Supporting Information

### A practical synthesis of chiral tricyclic cyclopenta[*b*]benzofuran, a key intermediate of Beraprost

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Prof. Ping Xie, email: xp@imm.ac.cn

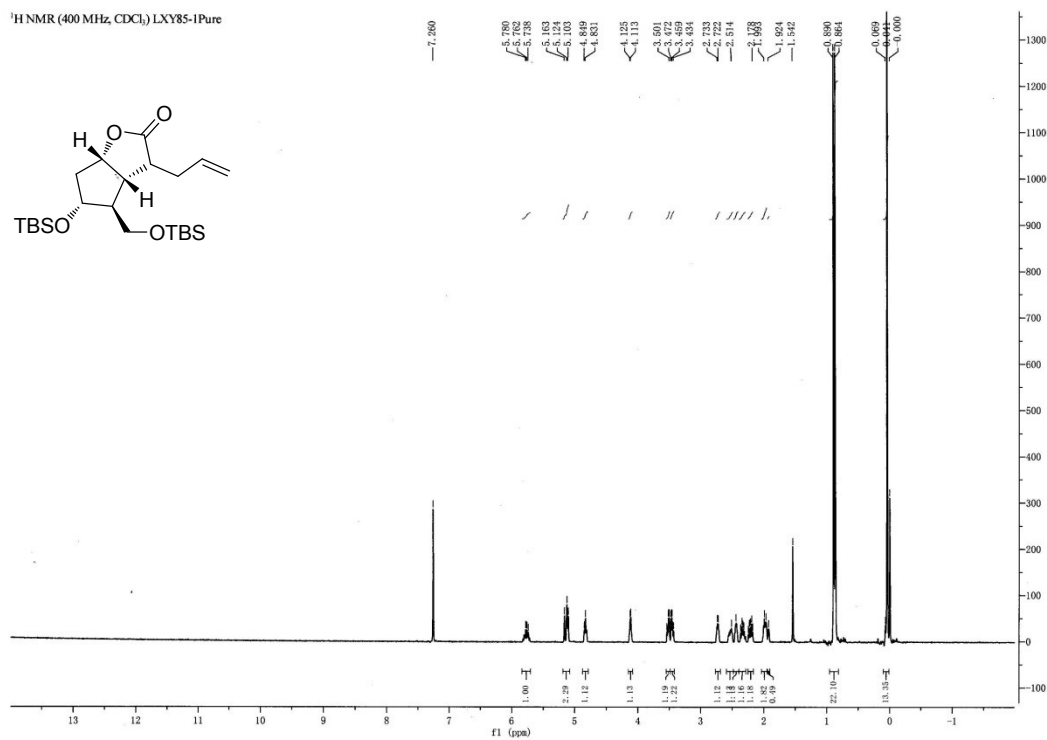
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2. <sup>1</sup> H, <sup>13</sup> C, DEPT, and 2D-NMR Spectral Copies.....	S3- S19

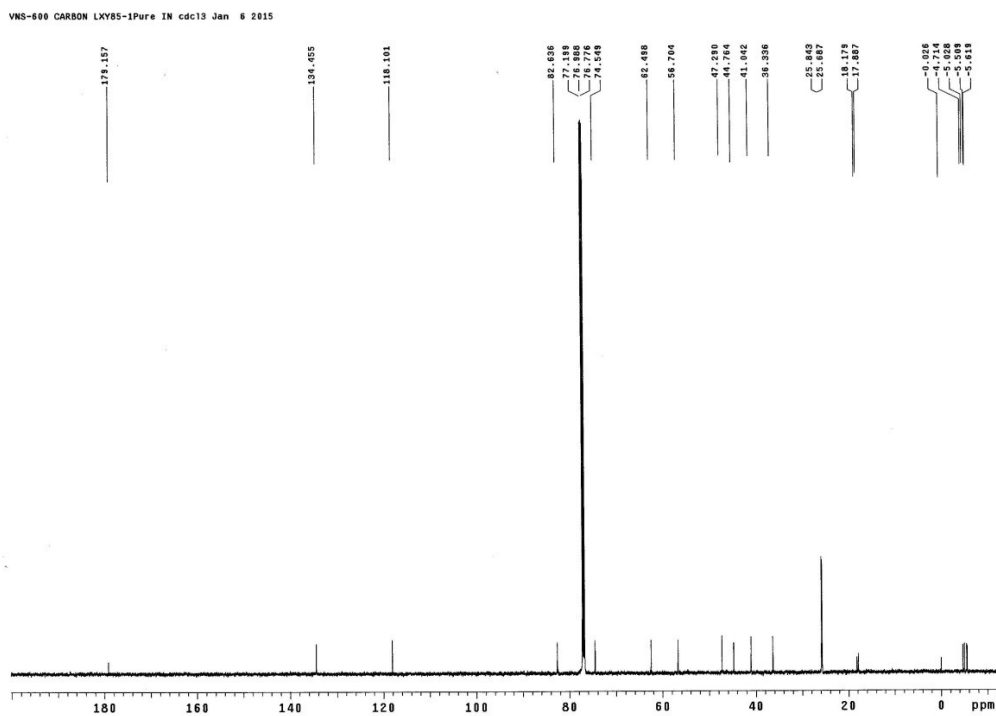
## 1. General Information

All the solvents and chemicals were purchased from commercial sources: Sigma-Aldrich Chemical Co., Arcos Chemical Co., and J&K Chemical Co. with the purity of more than 95%. Flash column chromatography was performed on Biotage Isolera one.  $^1\text{H}$  NMR and  $^{13}\text{C}$  NMR were recorded on Mercury 300, Mercury 400, Bruker AV500, AV600, JEOL ECZ400S spectrometer. Coupling constants are given in Hz and chemical shifts are expressed as  $\delta$  values in ppm. The following multiplicity abbreviations are used: (s) singlet, (d) doublet, (t) triplet, (q) quartet, (m) multiplet. ESI-HRMS data were measured on Thermo Exactive Orbitrap plus spectrometer.

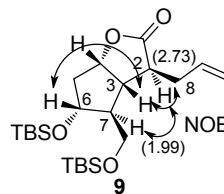
## 2. Experimental Procedures and Spectral Data of Products



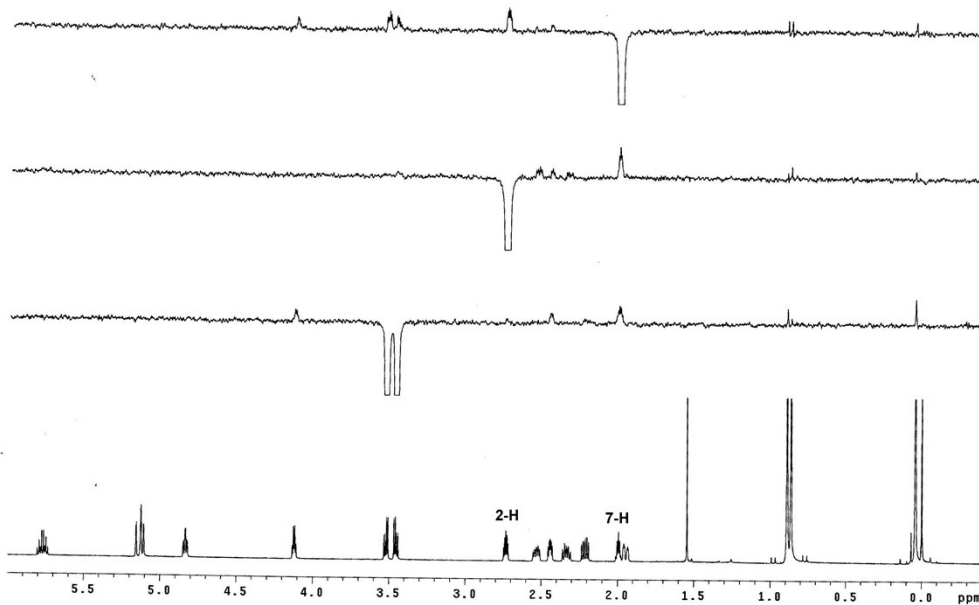
<sup>1</sup>H NMR spectra of compound 9



<sup>13</sup>C NMR spectra of compound 9



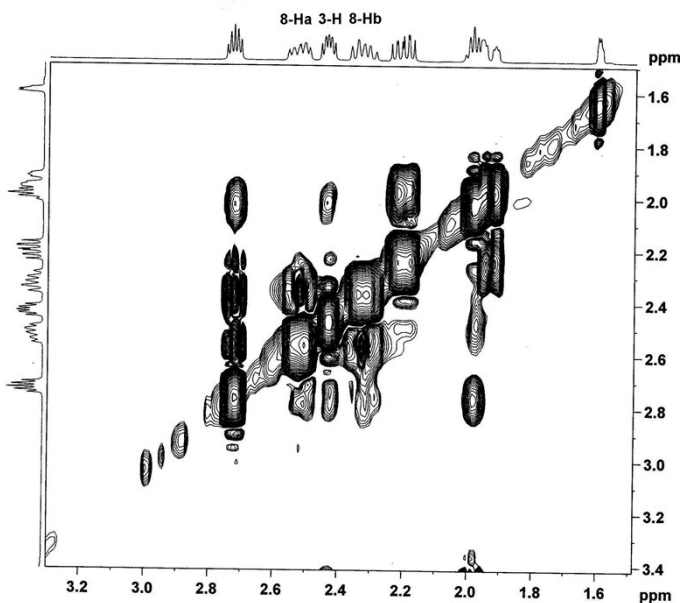
VNS-800 NOESY1D LXY85-1Pure IN cdc13 Dec 31 2014



NOE spectra of compound 9



Bruker AVANCEIII 400 20160713  
NOESY\_2D CDCl3 D:\DATA-2016 2



Current Data Parameters  
NAME 20160713 LXY85-1  
EXPNO 7  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20160714  
Time 18.39  
INSTRUM spect  
PROBHD 5 mm CYPBBO BB  
PULPROG noesy2dpp  
TD 2048  
SOLVENT CDCl3  
NS 32  
DS 16  
SWH 5597.015 Hz  
FIDRES 2.732918 Hz  
AQ 0.1829547 sec  
RG 110.56  
DW 89.333 usec  
DE 10.00 usec  
TE 295.0 K  
D0 0.00007521 sec  
D1 1.20000005 sec  
D8 0.60000002 sec  
D11 0.03000000 sec  
D12 0.00020000 sec  
D16 0.00020000 sec  
IN0 0.00017860 sec

CHANNEL f1  
SF01 400.1324008 MHz  
NUC1 1H  
P1 11.07 usec  
P2 22.14 usec  
P17 2500.00 usec  
PLW1 8.19999981 W  
PLW10 1.48650002 W

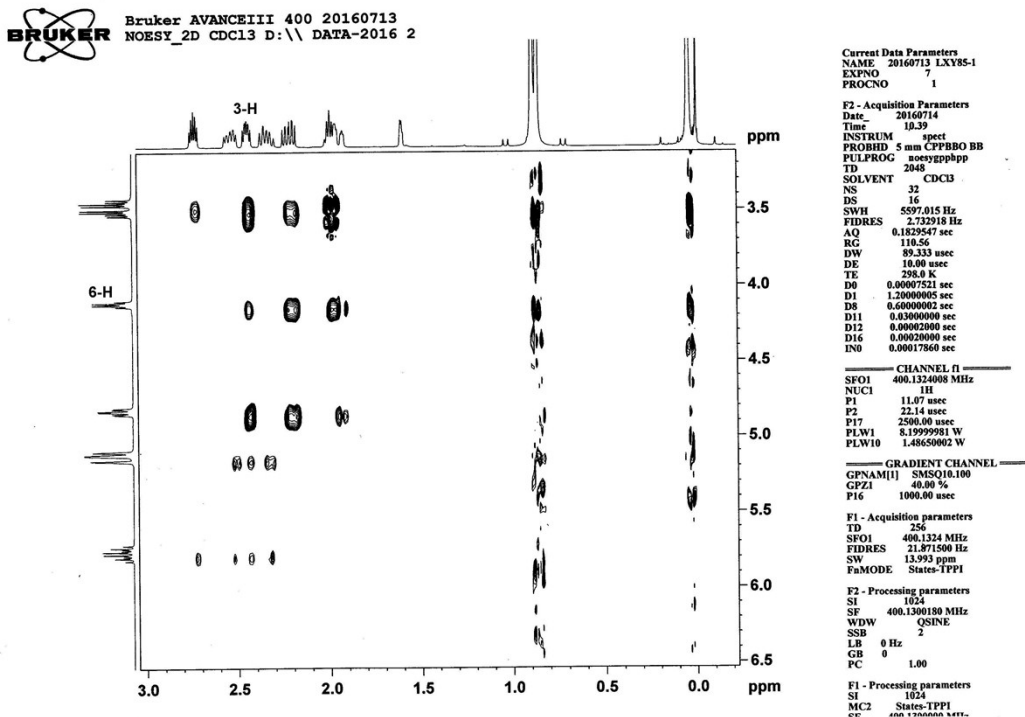
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GPNAM[f1] SMSQ10.100  
GFZ1 40.00 %  
P16 1000.00 usec

F1 - Acquisition parameters  
TD 256  
SF01 400.1324 MHz  
FIDRES 21.871500 Hz  
SW 13.993 ppm  
F0MODE States-TFPI

F2 - Processing parameters  
SI H24  
SF 400.1300180 MHz  
WDW Q  
SSB Q  
LB 0 Hz  
GB 0  
PC 1.00

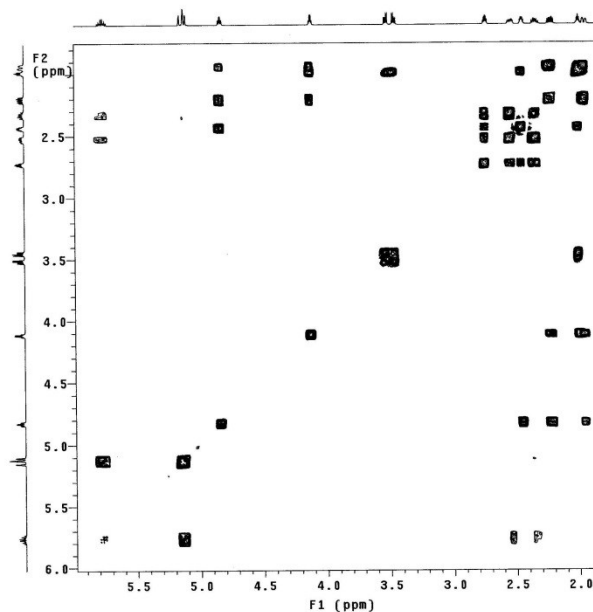
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MC2 States-TFPI  
AQ 1.700000000 sec.

### D-NOESY spectra of compound 9



### 2D-NOESY spectra of compound 9

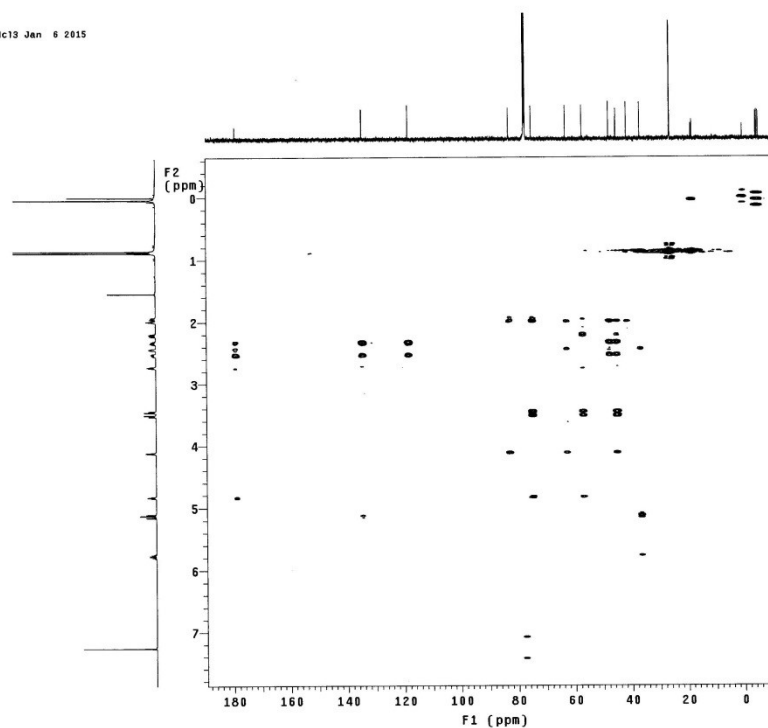
VNS-600 gCOSY LXY85-1Pure IN cdc13 Jan 6 2015  
 Temp. 25.0 C / 298.1 K  
 Sample #8, Operator: vjwalk  
 Relax. delay 1.000 sec  
 Acq. time 0.150 sec  
 Width 8443.3 Hz  
 2D width 6043.3 Hz  
 2 repetitions  
 200 increments  
 OBSERVE H1 599.6876837 MHz  
**DATA PROCESSING**  
 Ss. sine bell 0.075 sec  
 F1 DATA PROCESSING  
 Ss. sine bell 0.021 sec  
 FT size 2048 x 2048  
 Total time 8 min 35 sec



### H-H cosy spectra of compound 9

VNS-600 gHMBCAD LXY85-1Pure IN cdc13 Jan 6 2015

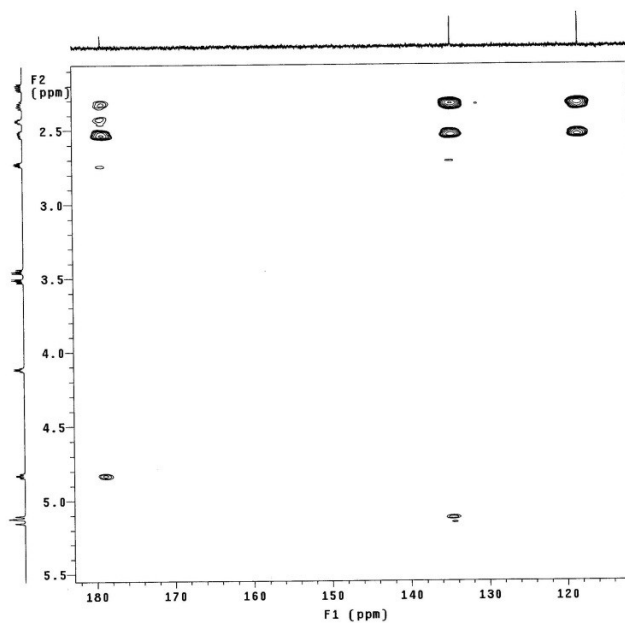
Temp. 25.0 C / 298.1 K  
Sample #8, Operator: vjwalk  
Relax. delay 1.000 sec  
Acq. time 0.150 sec  
Width 6443.3 Hz  
2D Width 38182.7 Hz  
32 repetitions  
2 x 90 increments  
OBSERVE H1, 599.6876837 MHz  
DATA PROCESSING  
Sg, sine bell 0.075 sec  
F1 DATA PROCESSING  
Gauss apodization 0.002 sec  
F1 size 4096 x 2048  
Total time 1 hr, 59 min



HMBC spectra of compound 9

VNS-600 gHMBCAD LXY85-1Pure IN cdc13 Jan 6 2015

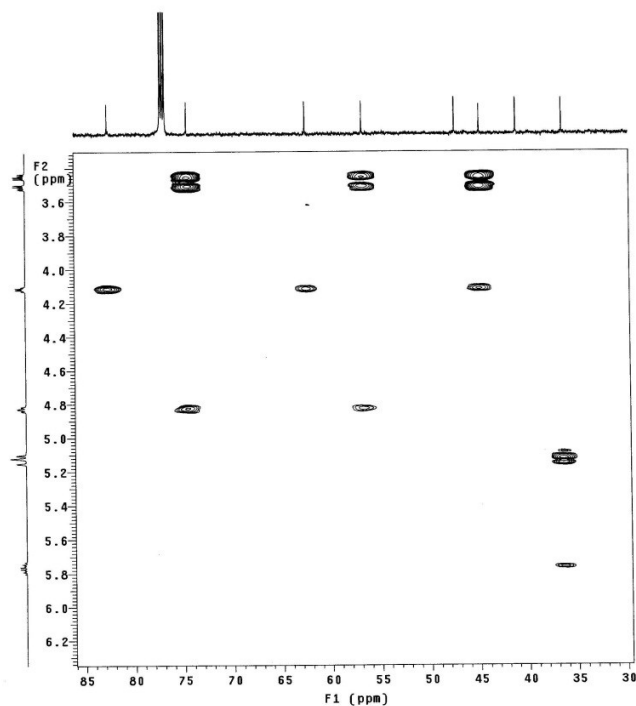
Temp. 25.0 C / 298.1 K  
Sample #8, Operator: vjwalk  
Relax. delay 1.000 sec  
Acq. time 0.150 sec  
Width 6443.3 Hz  
2D Width 38182.7 Hz  
32 repetitions  
2 x 90 increments  
OBSERVE H1, 599.6876837 MHz  
DATA PROCESSING  
Sg, sine bell 0.075 sec  
F1 DATA PROCESSING  
Gauss apodization 0.002 sec  
F1 size 4096 x 2048  
Total time 1 hr, 59 min



HBC spectra of compound 9

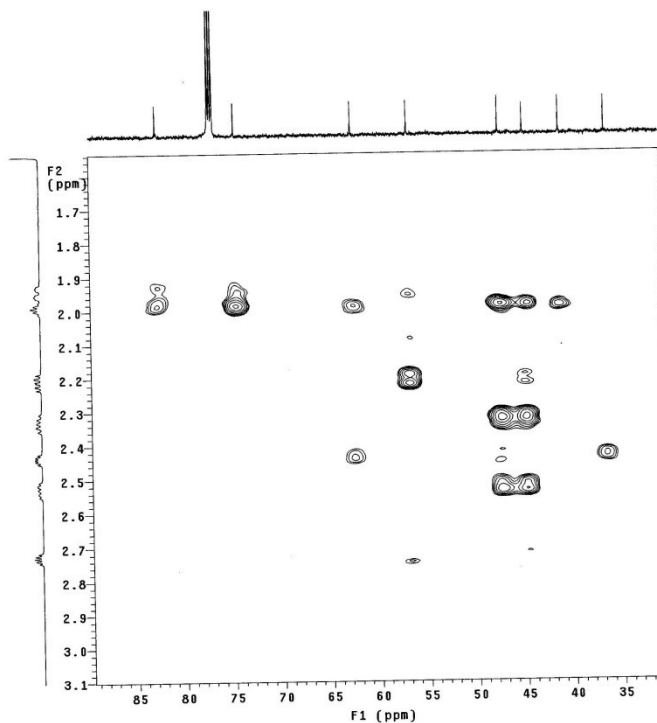
H

VNS-600 gHMBCAD LXY85-1Pure IN cdc13 Jan 6 2015  
 Temp. 25.0 C / 298.1 K  
 Sample #8, Operator: vjwalk  
 Relax. delay 1.000 sec  
 Acq. time 0.150 sec  
 Width 6443.3 Hz  
 2D Width 36182.7 Hz  
 32 repetitions  
 2 x 99 increments  
 OBSERVE HI 599.6876837 MHz  
 DATA PROCESSING  
 S1. sine bell 0.075 sec  
 F1 DATA PROCESSING  
 Gauss apodization 0.002 sec  
 FT size 4096 x 2048  
 Total time 1 hr, 59 min



HMBC spectra of compound 9

VNS-600 gHMBCAD LXY85-1Pure IN cdc13 Jan 6 2015  
 Temp. 25.0 C / 298.1 K  
 Sample #8, Operator: vjwalk  
 Relax. delay 1.000 sec  
 Acq. time 0.150 sec  
 Width 6443.3 Hz  
 2D Width 36182.7 Hz  
 32 repetitions  
 2 x 99 increments  
 OBSERVE HI 599.6876837 MHz  
 DATA PROCESSING  
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 F1 DATA PROCESSING  
 Gauss apodization 0.002 sec  
 FT size 4096 x 2048  
 Total time 1 hr, 59 min

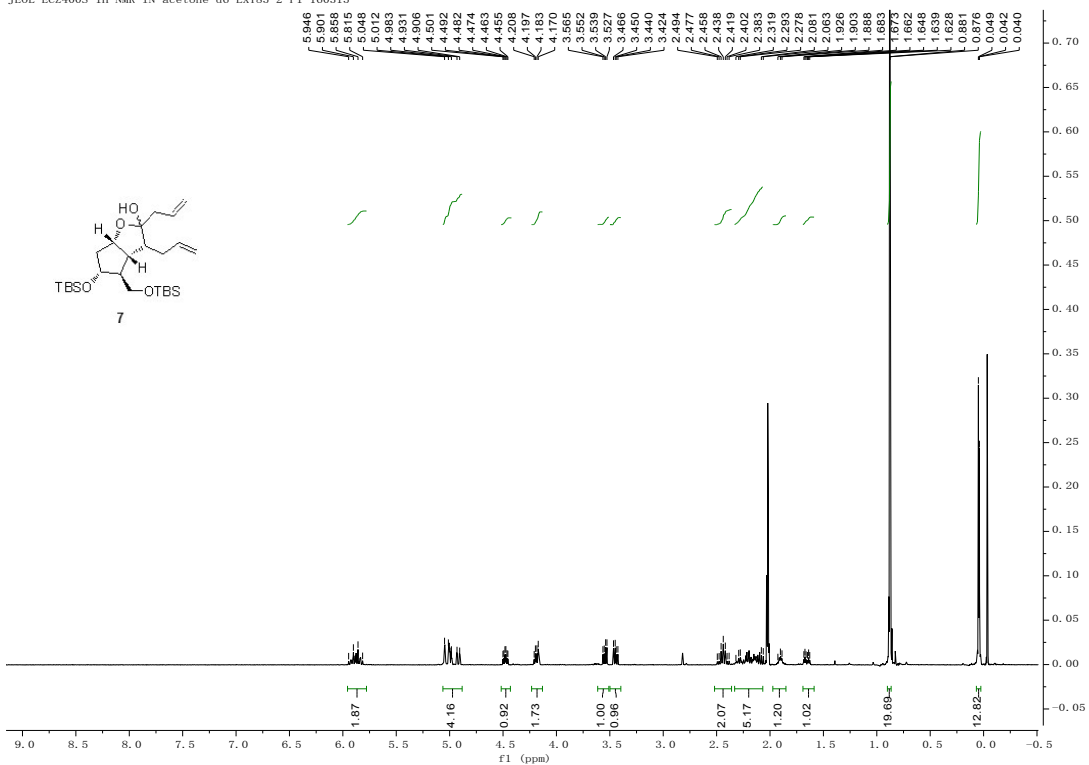


HMBC spectra of compound 9



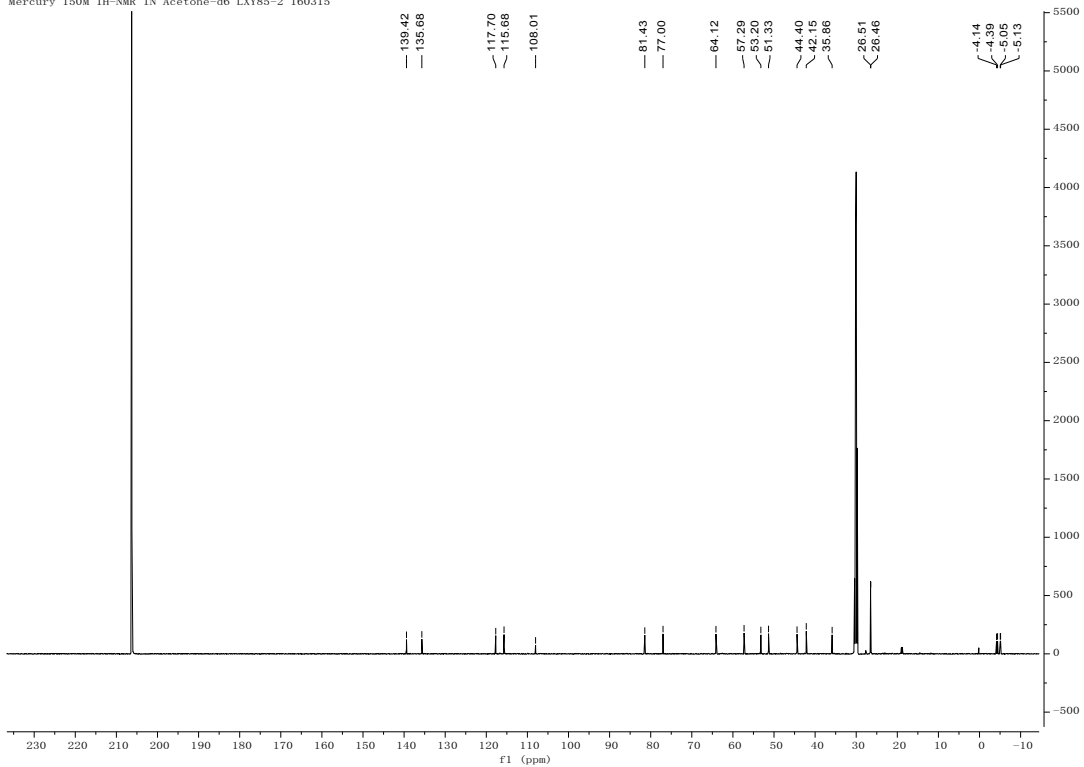


JEOL ECZ400S 1H-NMR IN acetone-d6 LXY85-2-P1 160315



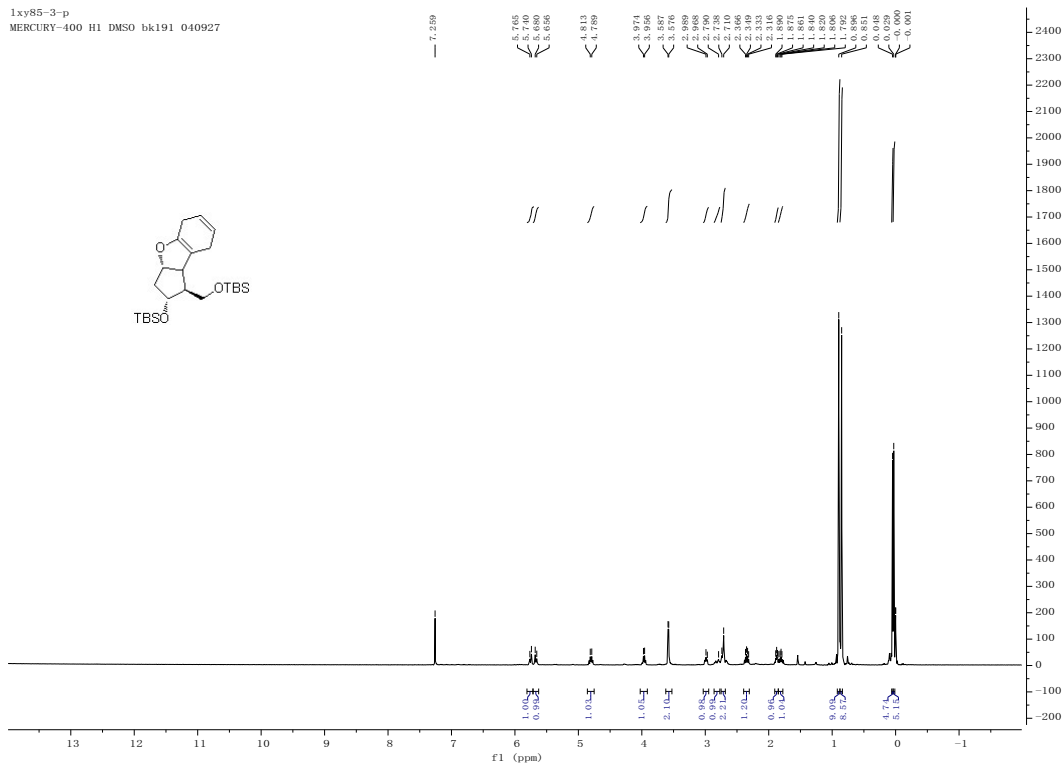
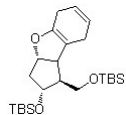
<sup>1</sup>H NMR spectra of compound 7

Mercury 150M 1H-NMR IN Acetone-d6 LXY85-2 160315



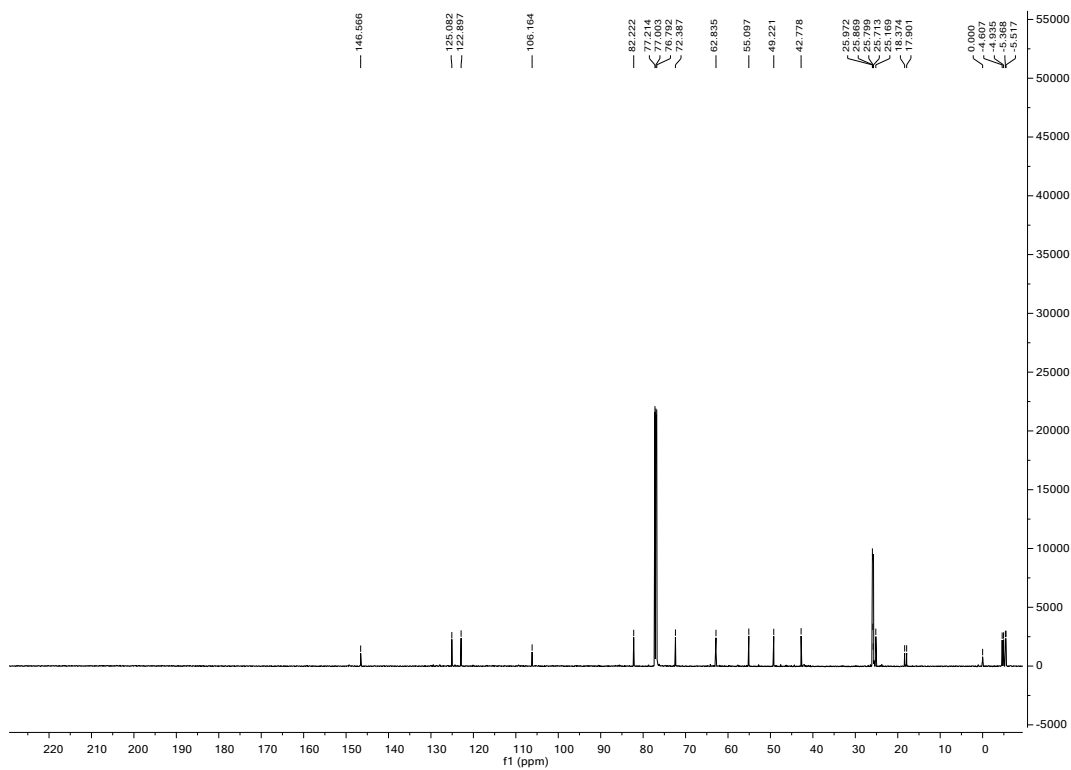
<sup>13</sup>C NMR spectra of compound 7

1xy85-3-p  
MERCURY-400 H1 DMSO bk191 040927

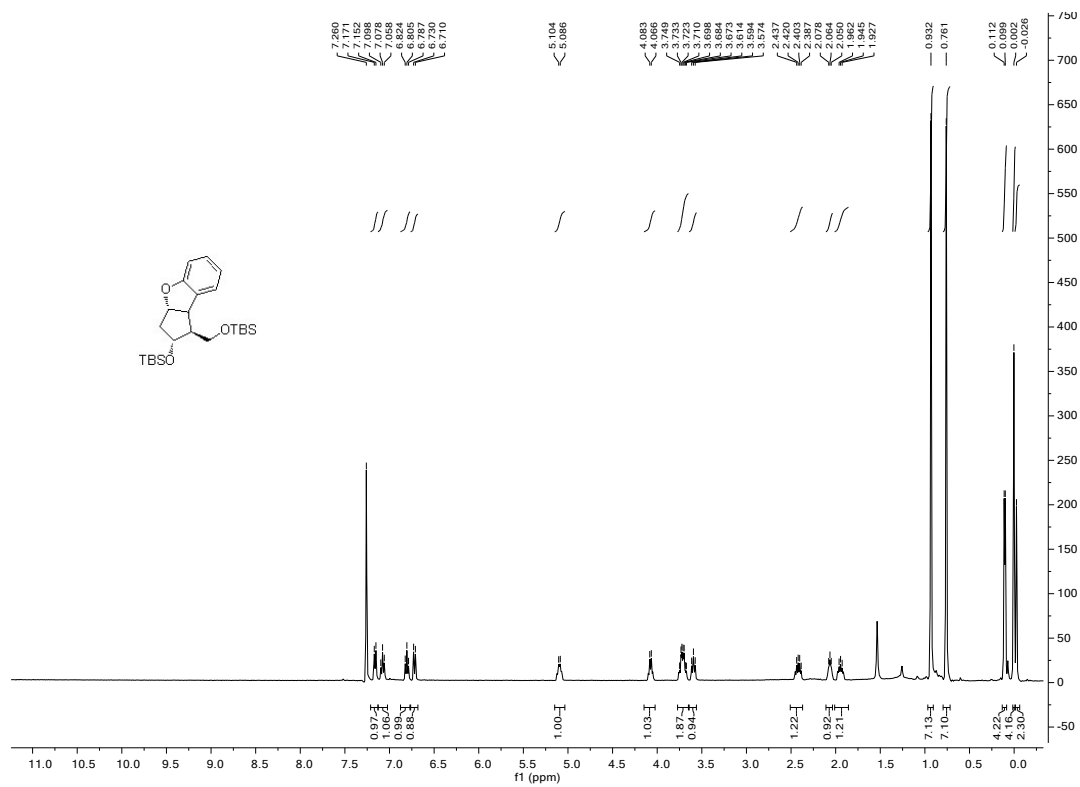


1

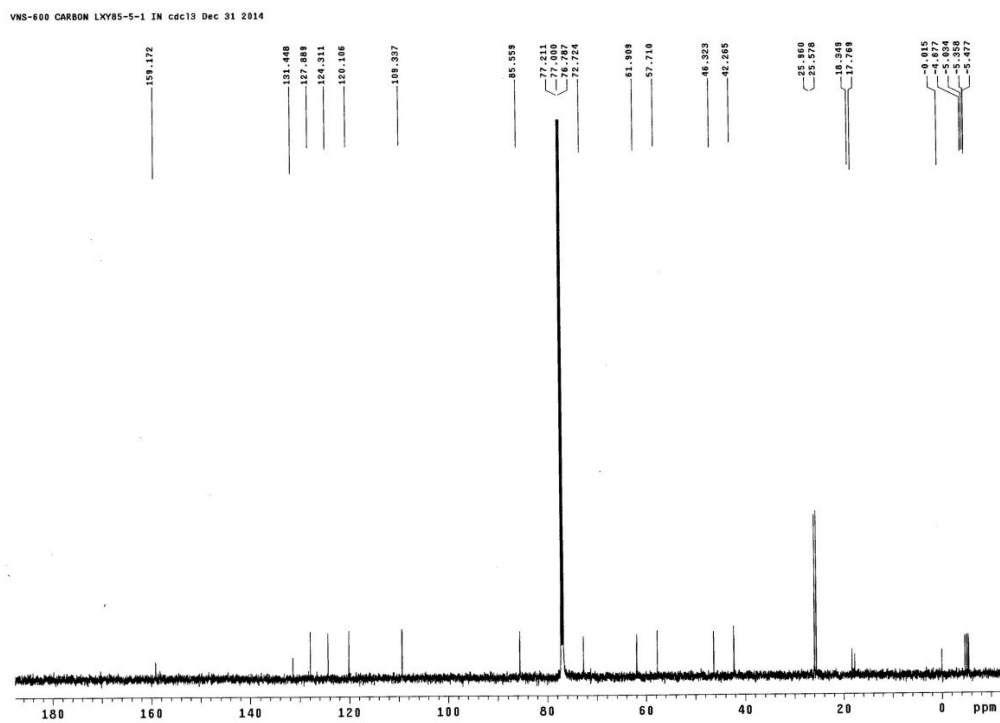
H NMR spectra of compound 10



13C NMR spectra of compound 10

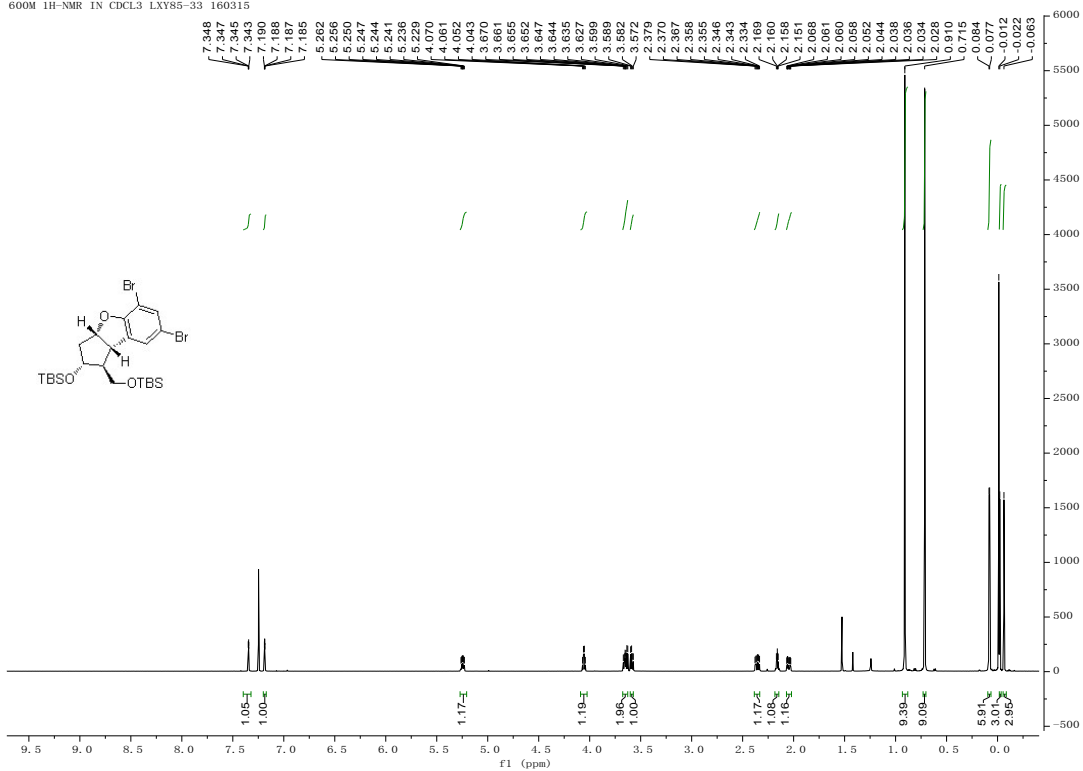


<sup>1</sup>H NMR spectra of compound 6



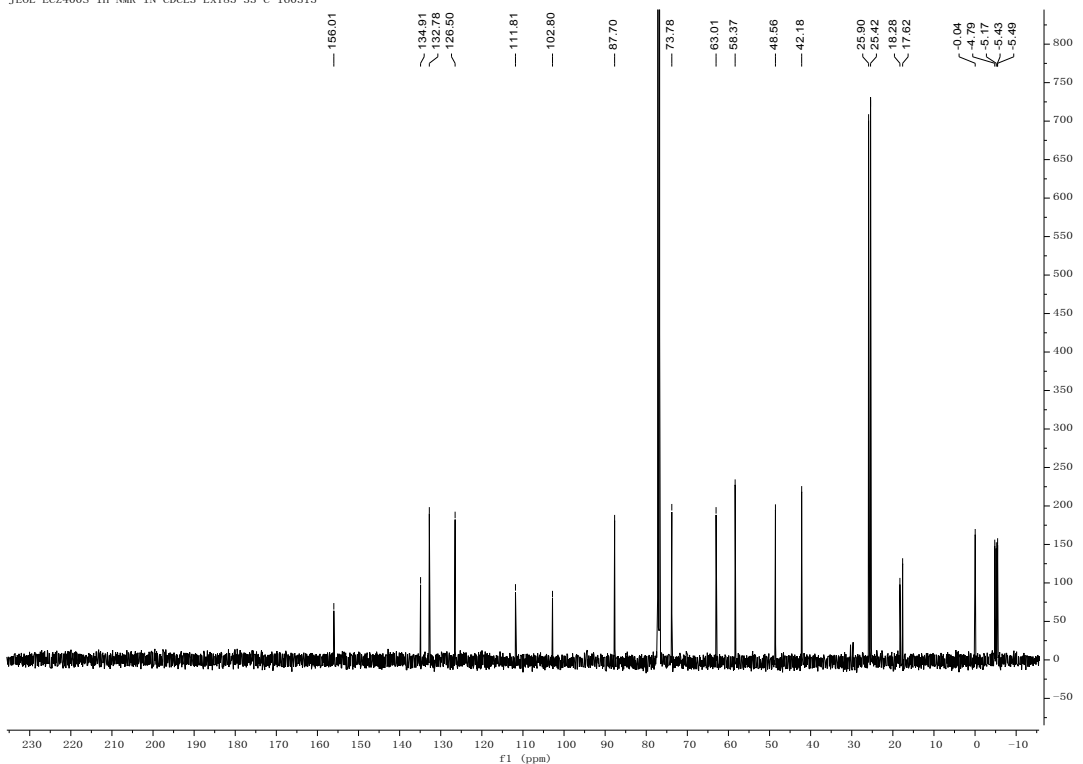
<sup>13</sup>C NMR spectra of compound 6

600M 1H-NMR IN CDCL3 LXY85-33 160315

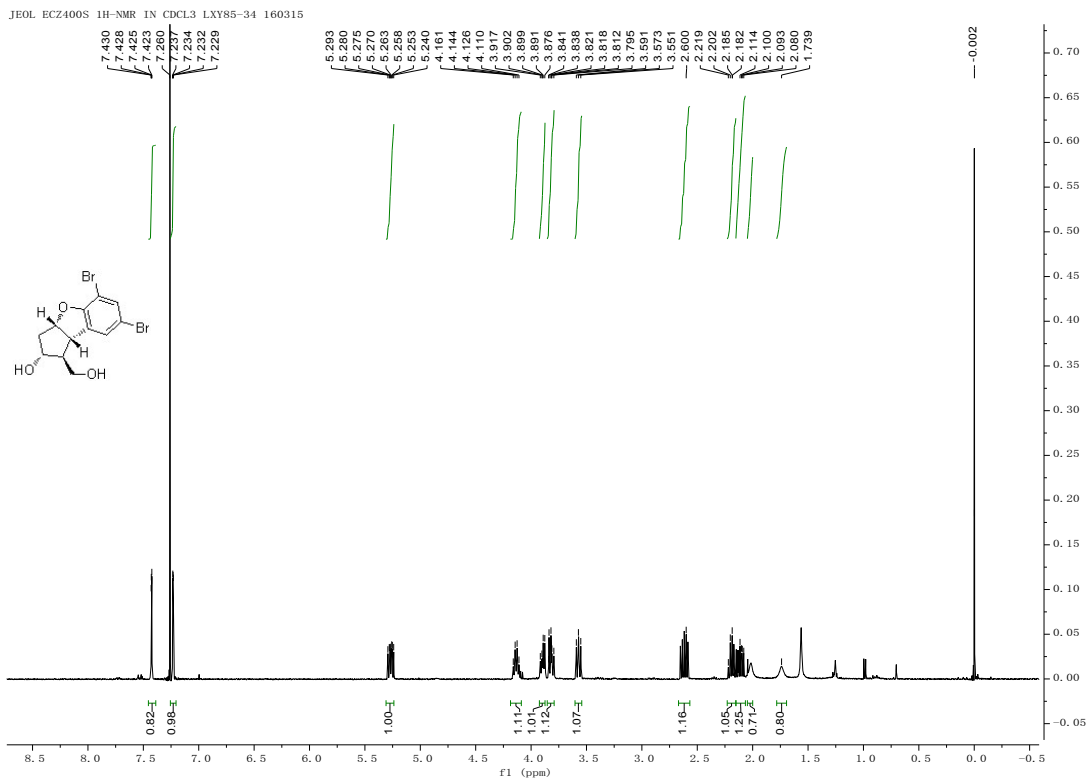


### <sup>1</sup>H NMR spectra of compound 13

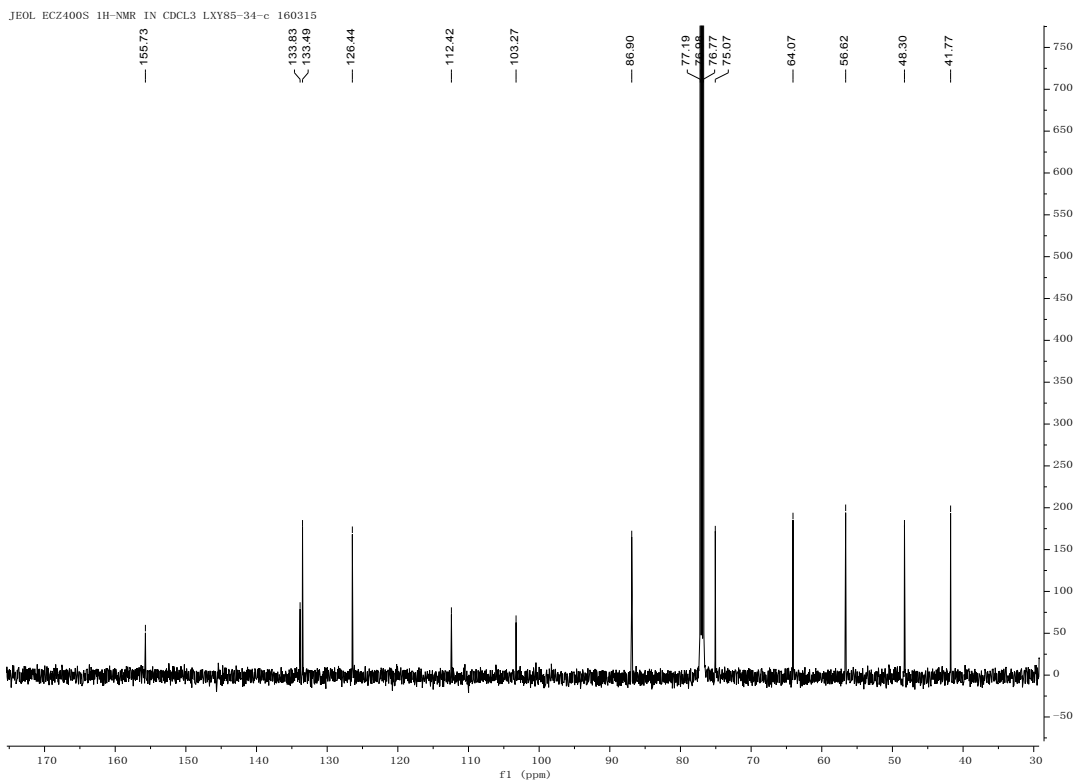
JEOL ECZ400S 1H-NMR IN CDCL3 LXY85-33-C 160315



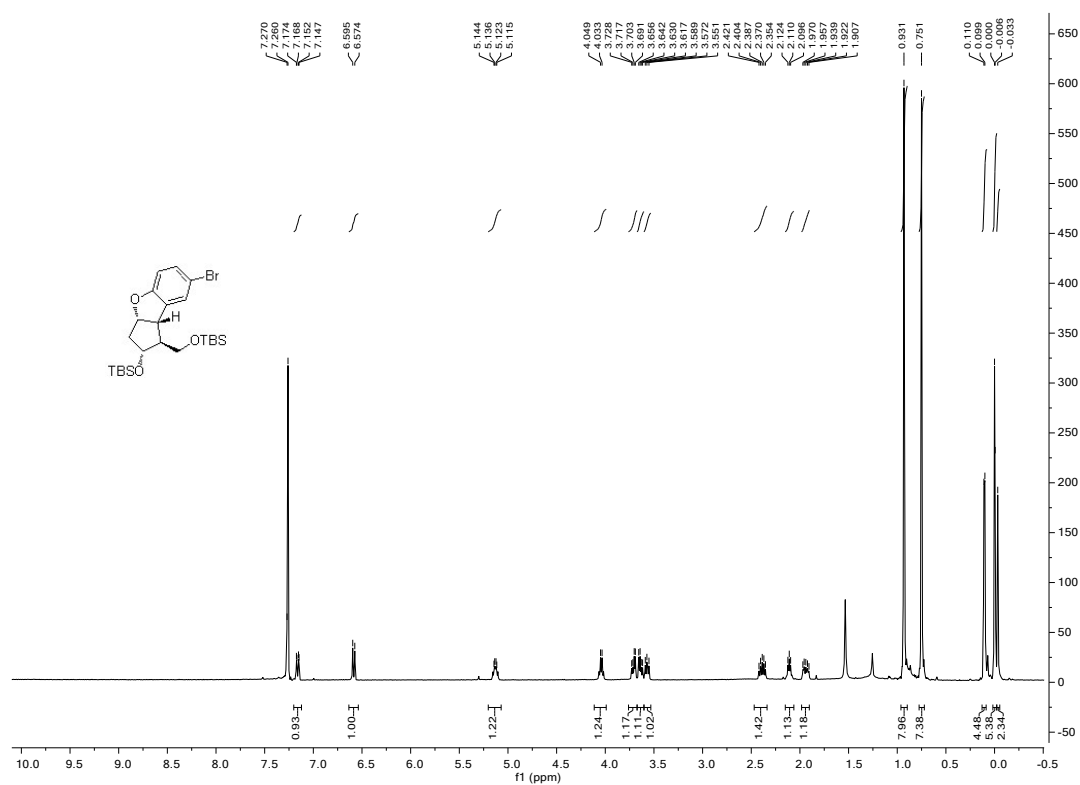
### <sup>13</sup>C NMR spectra of compound 13



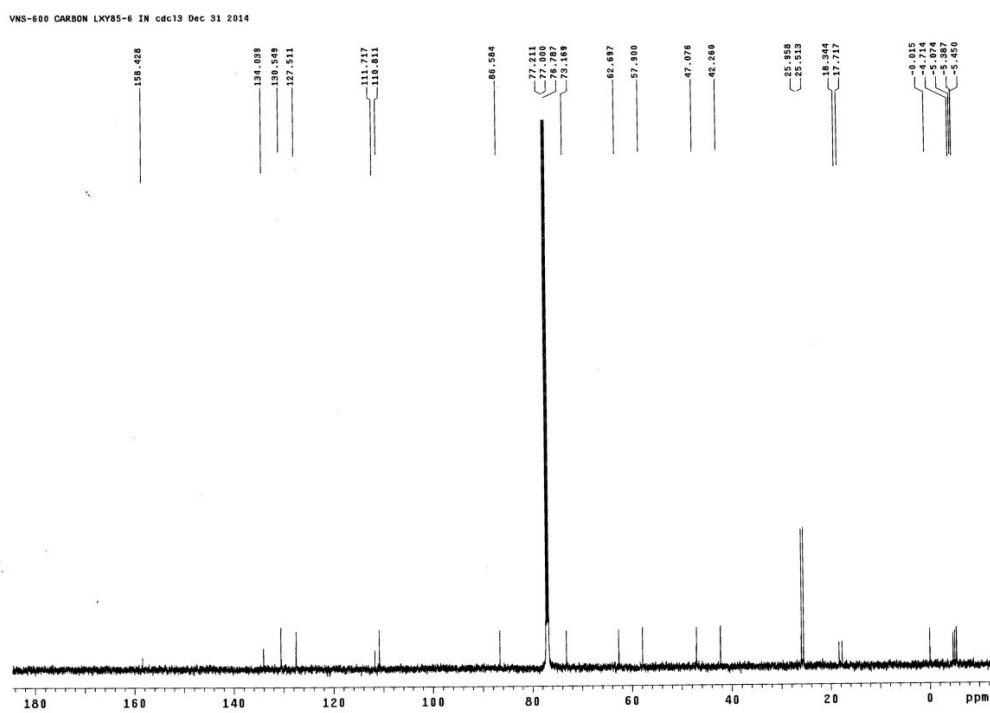
<sup>1</sup>H NMR spectra of compound 12



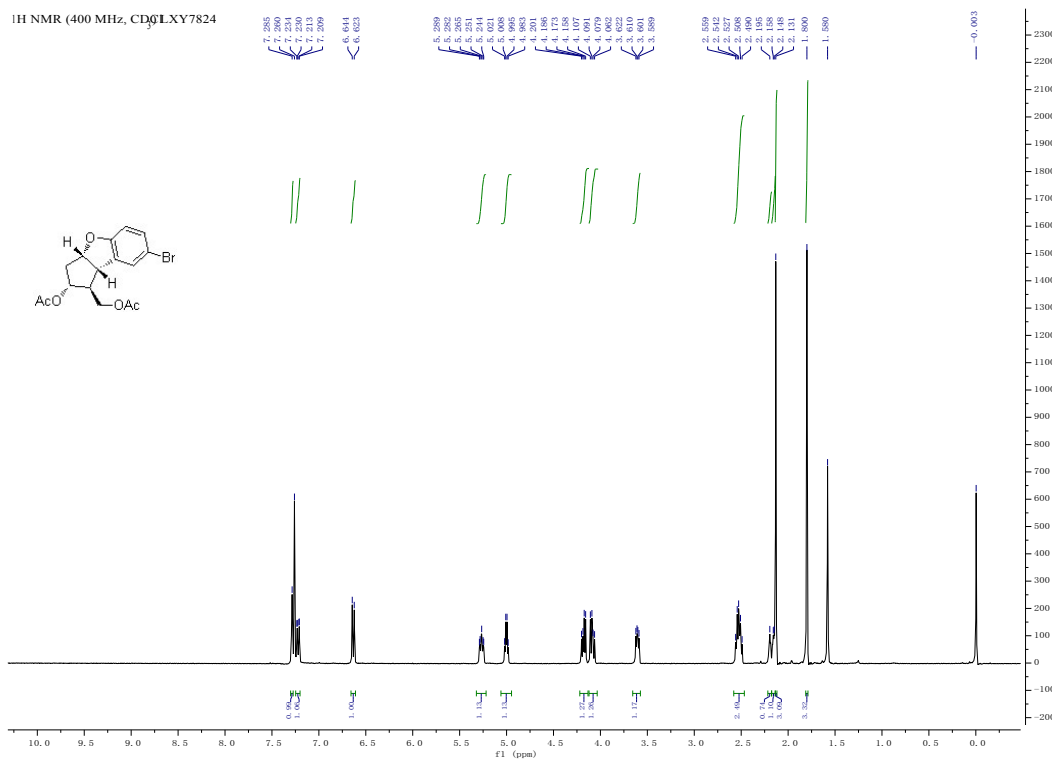
<sup>13</sup>C NMR spectra of compound 13



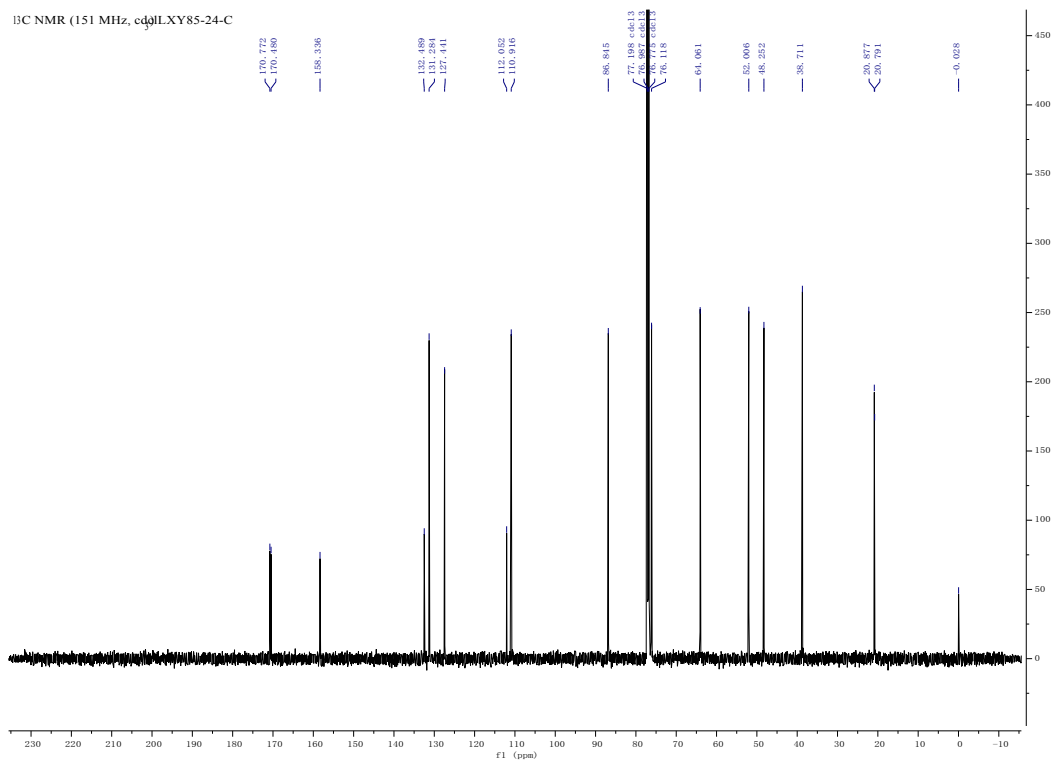
<sup>1</sup>H NMR spectra of compound 11



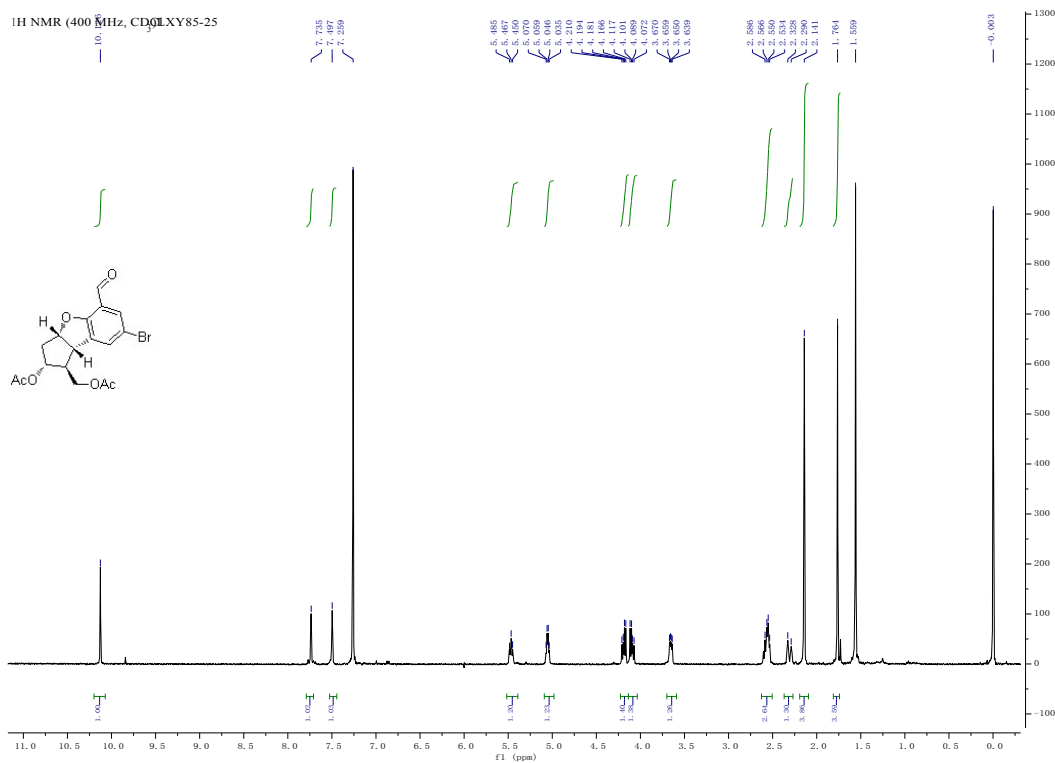
<sup>13</sup>C NMR spectra of compound 11



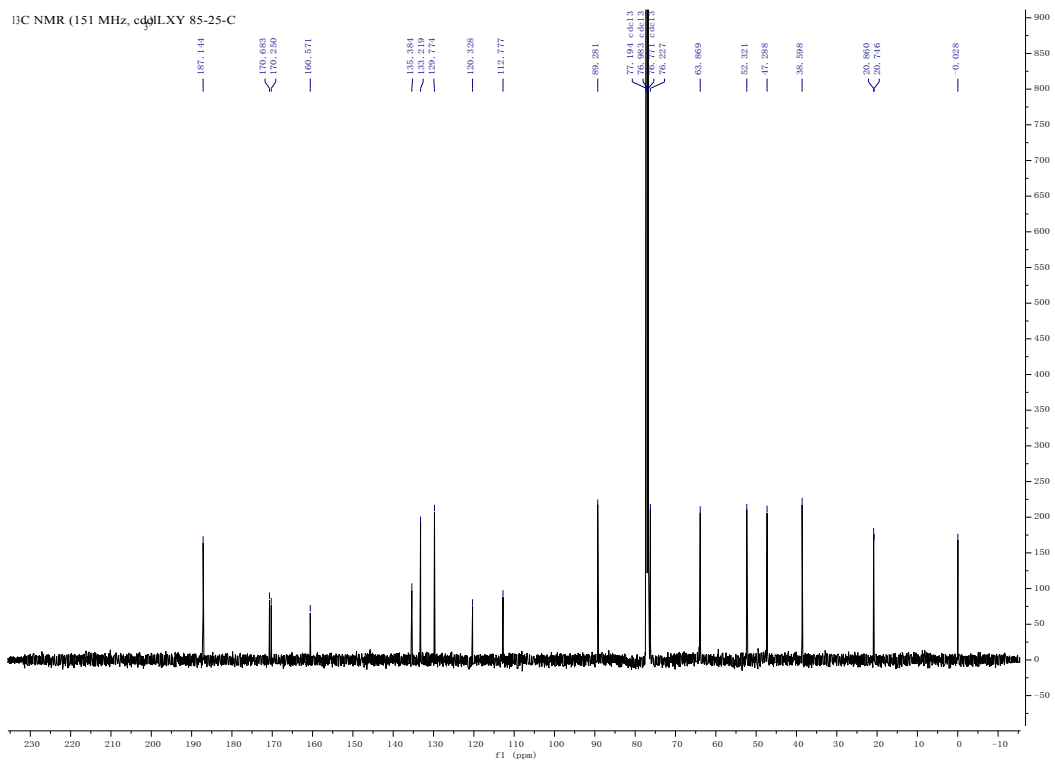
**<sup>1</sup>H NMR spectra of compound 15**



**<sup>13</sup>C NMR spectra of compound 15**



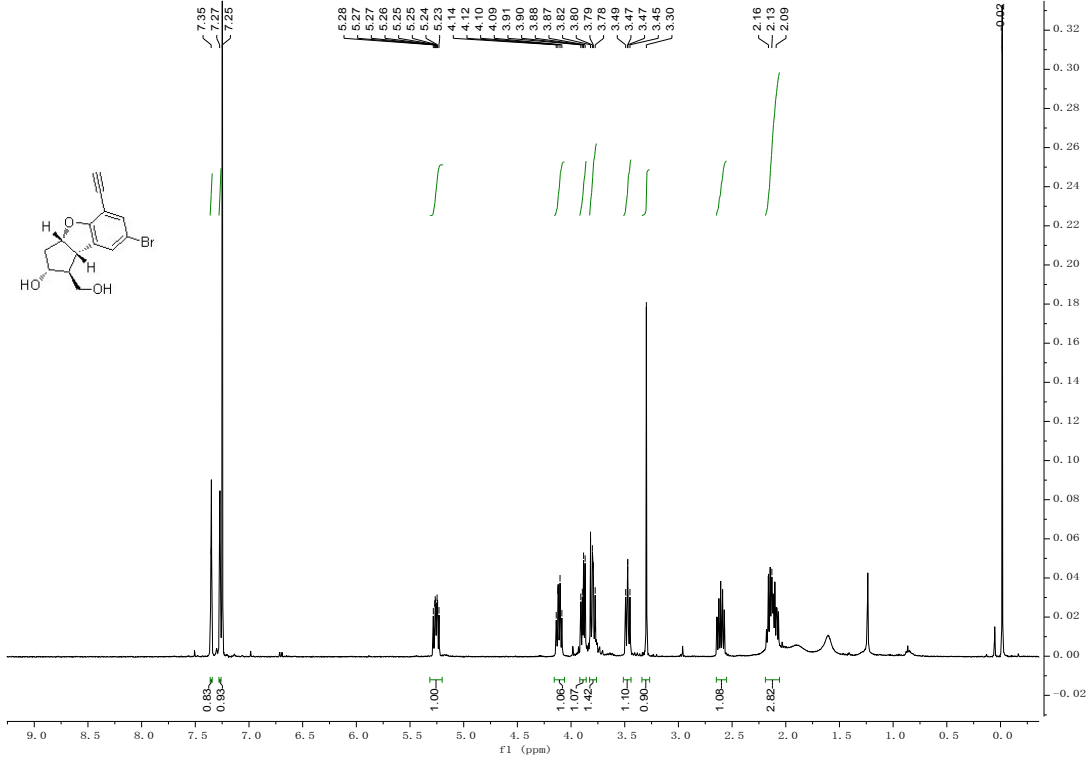
<sup>1</sup>H NMR spectra of compound 16



<sup>13</sup>C NMR spectra of compound 16

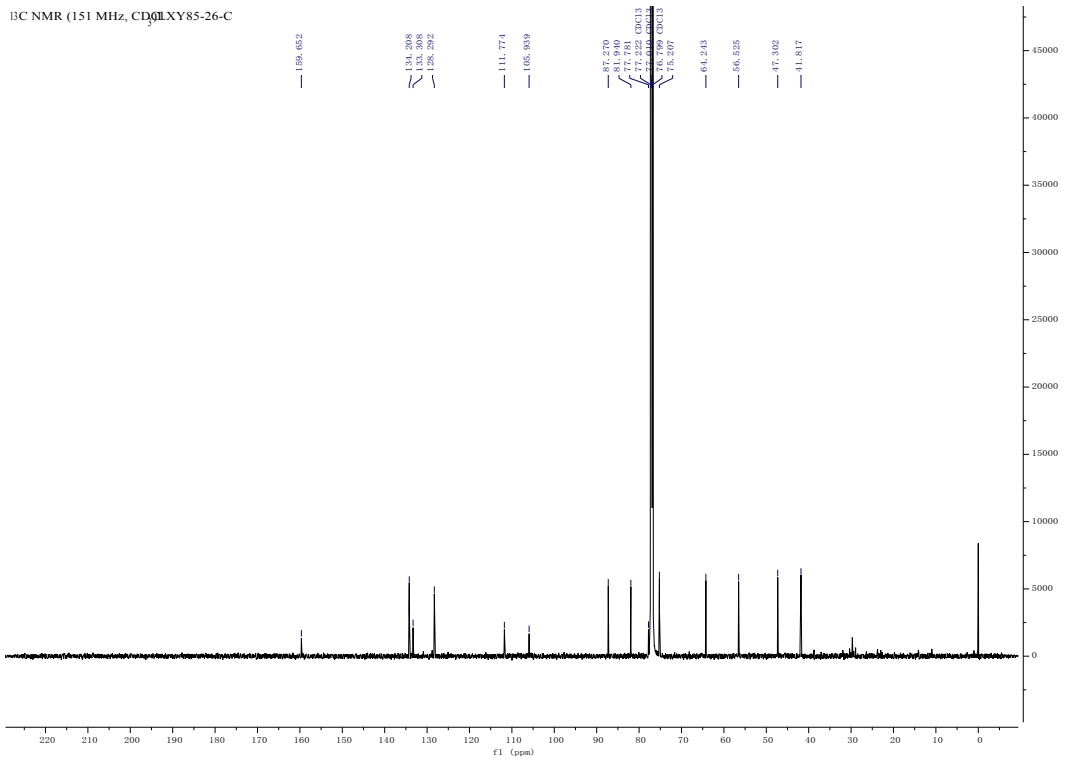


MERCURY-500 1H-NMR IN CDCL3 LXYS-26-P 160315

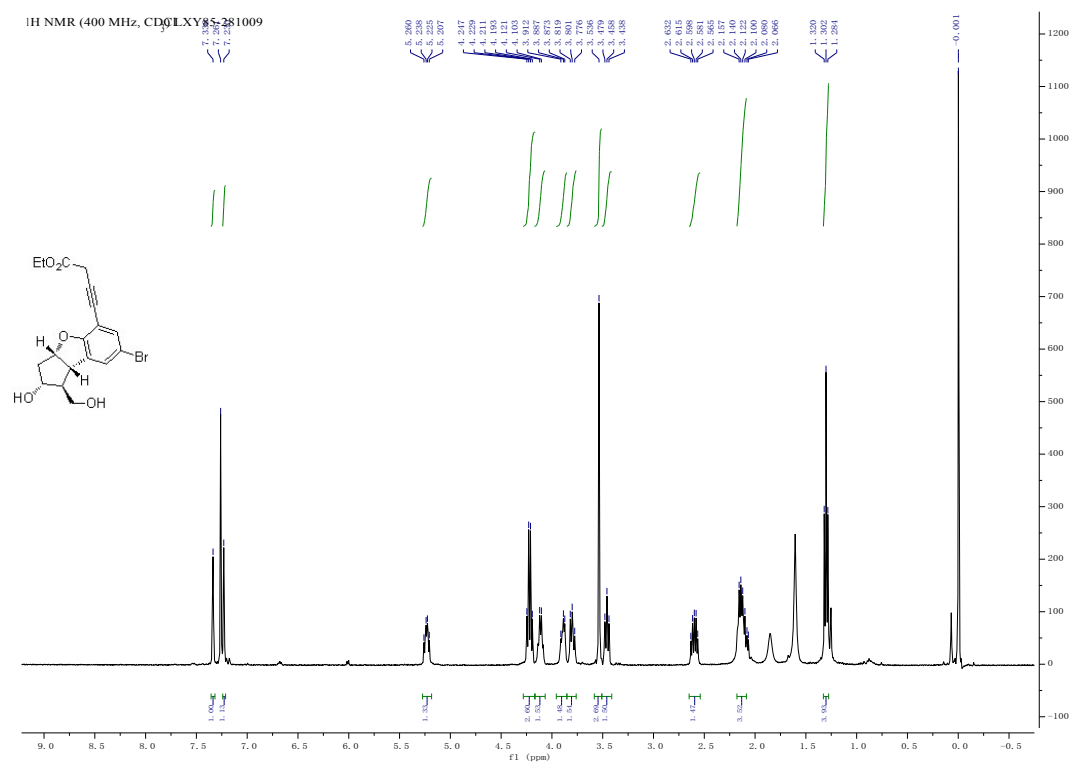


<sup>1</sup>H NMR spectra of compound 17

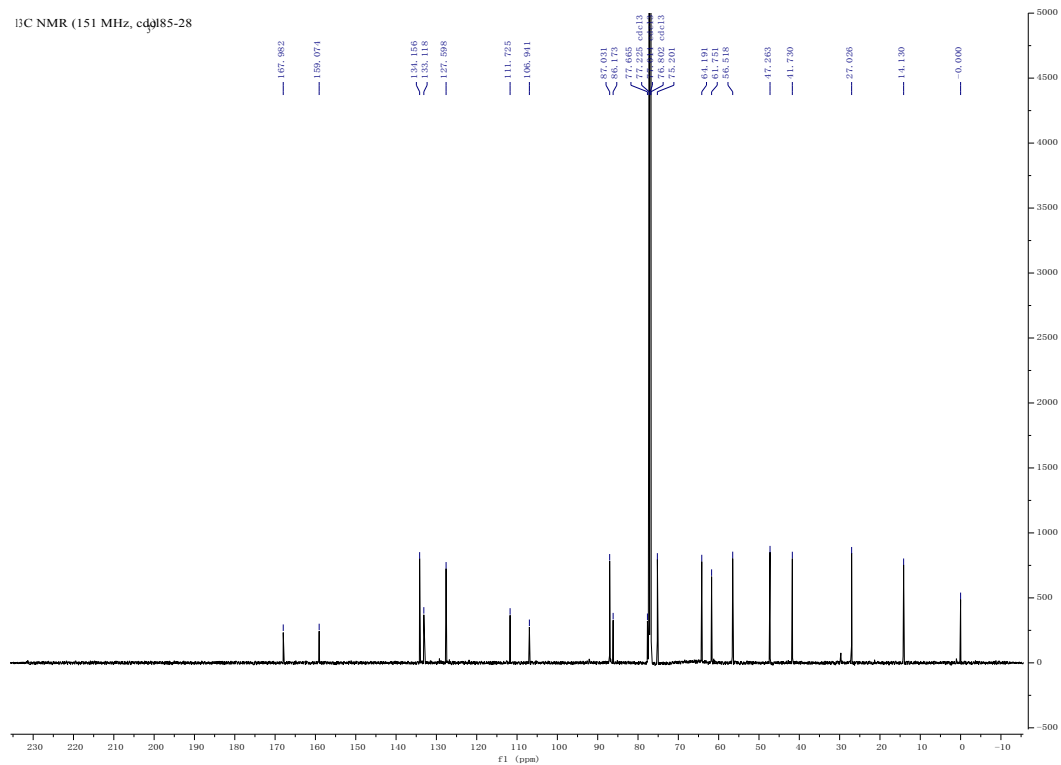
13C NMR (151 MHz, CDCl3) LXYS-26-C



<sup>13</sup>C NMR spectra of compound 17

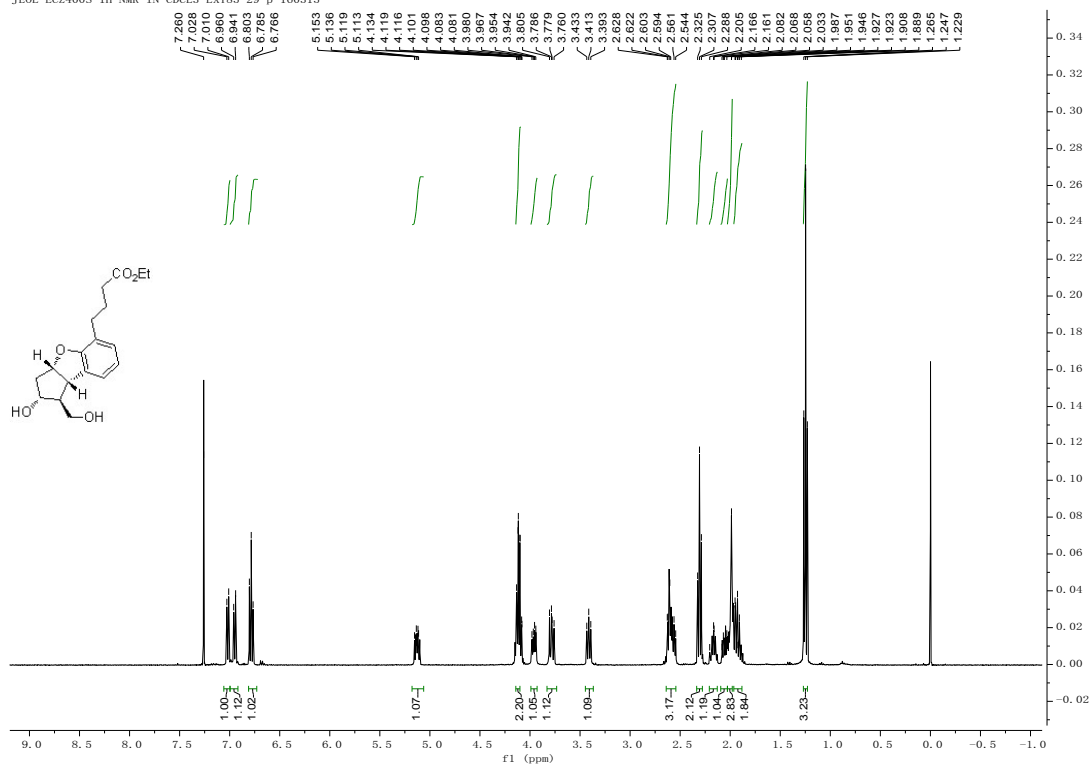


<sup>1</sup>H NMR spectra of compound 18



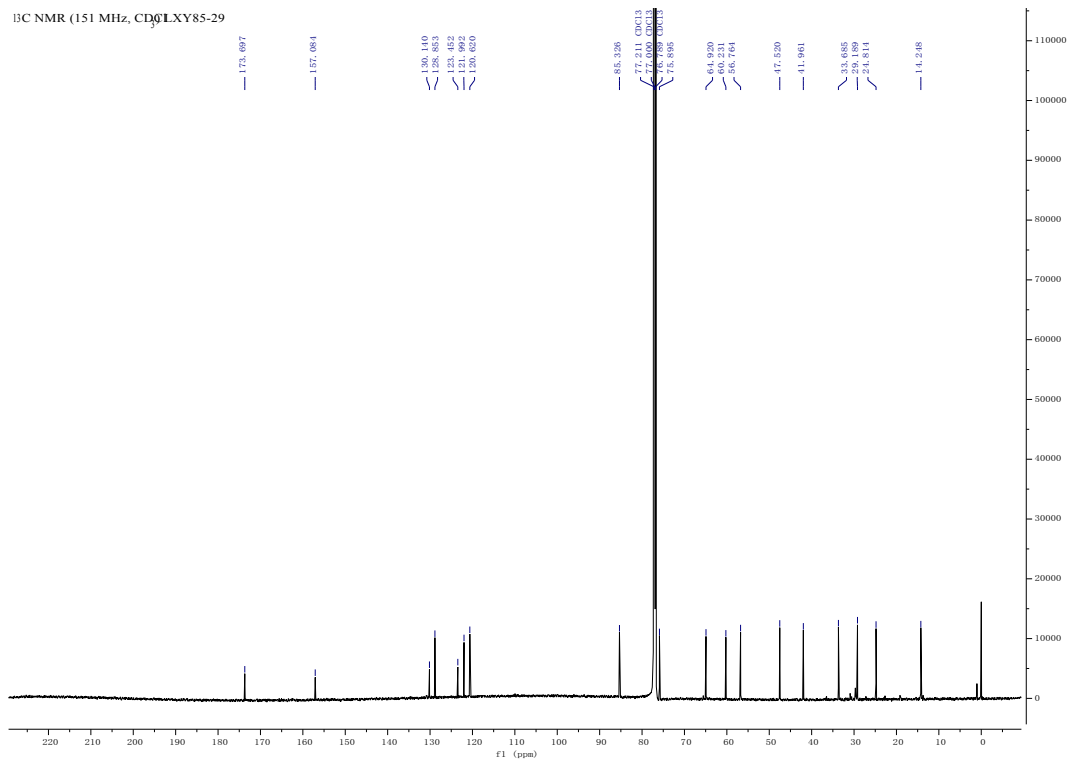
<sup>13</sup>C NMR spectra of compound 18

JEOL ECZ400S 1H-NMR IN CDCL3 LXY85-29-p 160315



<sup>1</sup>H NMR spectra of compound 4b

13C NMR (151 MHz, CDCl3) LXY85-29



<sup>13</sup>C NMR spectra of compound 4b