Synthesis

Reviews and Full Papers in Chemical Synthesis

Supporting Information

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Synthesis of Aza-polyquinanes via Fischer indolization and Ring-Rearrangement Metathesis as Key Steps

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Supporting Information

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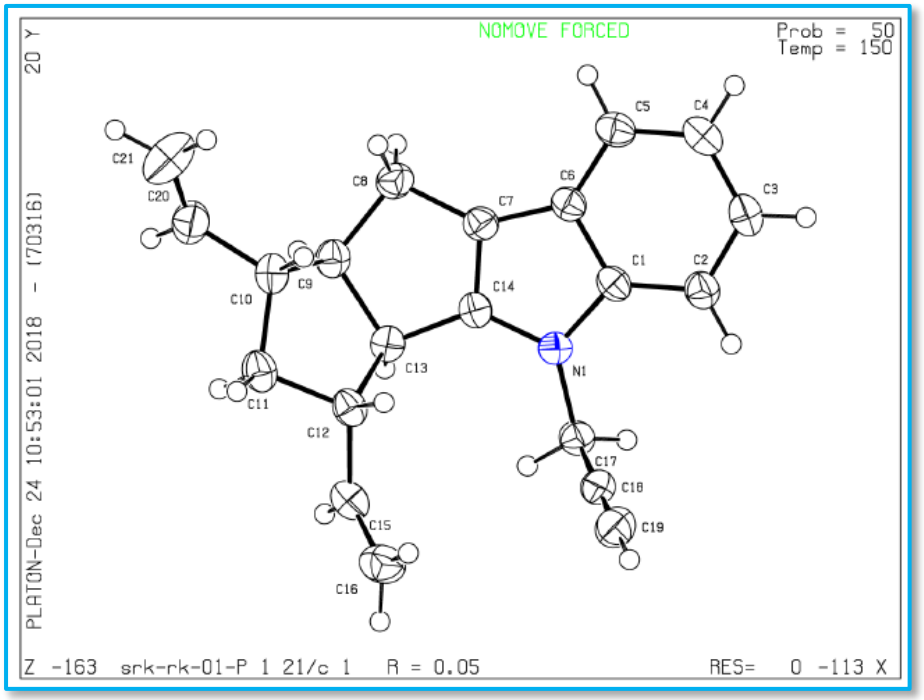
1. X-ray data and refinement parameters for target compounds

(1R,3S,3aS,9aR)-4-(prop-2-yn-1-yl)-1,3-divinyl-2,3,3a,4,9,9a-hexahydro-1H-pentaleno[1,2-b]indole (19b)

CCDC Number = *1886978*



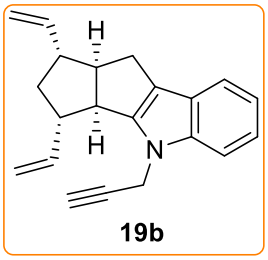


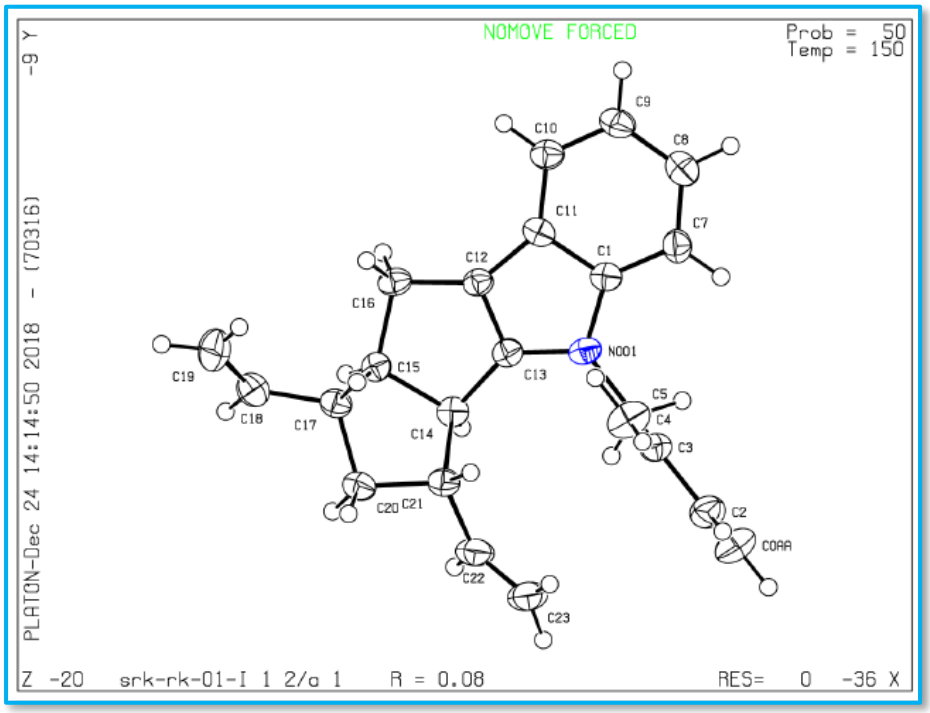
Table S1. X-ray crystallographic data and refinement parameters for **19b (CCDC 1886978)**

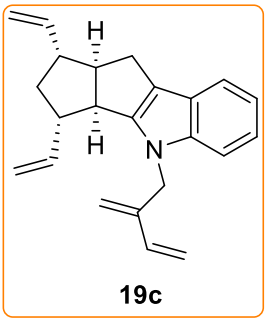
Identification code	SRK-RK-01-50-2_mo
Empirical formula	$C_{21}H_{21}N$
Formula weight	287.39
Temperature	150 K
Crystal system	Monoclinic
Space group	$P2_1/c$
Unit cell dimensions	a = 16.9888 (12) Å α = 90°
	b = 4.9020 (4) Å β = 90.857° (7)
	c = 19.1064 (15) Å γ = 90°
Volume	1591.0 (2) $Å^3$
Z	4
Density (calculated)	1.200 g/cm^3
Absorption coefficient (μ)	0.069 mm^{-1}
Absorption correction	Multi-scan
Max. and Min. transmission	0.997-0.992

F (000)	616.0
Crystal size	0.172 x 0.099 x 0.038 mm^3
Index ranges	$-20 \leq h \leq 19$, $-5 \leq k \leq 4$, $-19 \leq l \leq 22$
Theta range for data collection	2.398 to 24.994°
Reflections collected	0.0544 (1930)
Diffraction radiation wavelength	0.71073
Independent reflections	2792 [$R_{(int)} = 0.0734$]
Completeness to $\theta = 24.994°$	99.8%
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	2792/0/199
Goodness-of-fit on F^2	1.059
Final *R* indices [I>=2σ (I)]	*R*1 = 0.0544, *wR*2 = 0.1180
R indices (all data)	*R*1 = 0.0851, *wR*2 = 0.1429
Largest diff. peak and hole	0.24/-0.24 e $Å^{-3}$

(1R,3S,3aS,9aR)-4-(2-methylenebut-3-en-1-yl)-1,3-divinyl-2,3,3a,4,9,9a-hexahydro-1H-pentaleno[1,2-b]indole (19c)

CCDC Number = *1887151*





19c

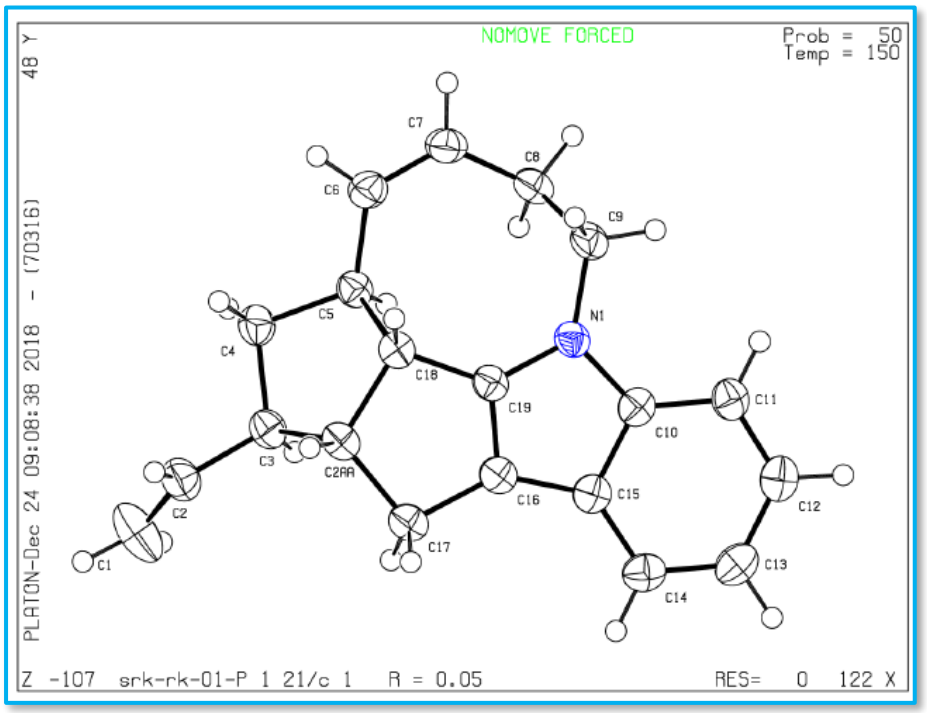
Table S2. X-ray crystallographic data and refinement parameters for **19c (CCDC 1887151)**

Identification code	SRK-RK-01-87_cu
Empirical formula	$C_{23}H_{25}N$
Formula weight	315.44
Temperature	150 K
Crystal system	Monoclinic
Space group	I2/a
Unit cell dimensions	a = 22.8136 (15) Å α = 90°
	b = 8641 (4) Å β = 109.643° (7)
	c = 24.7742 (13) Å γ = 90°
Volume	3653.8 (4) $Å^3$
Z	8
Density (calculated)	1.147 g/cm^3
Absorption coefficient (μ)	0.494 mm^{-1}
Absorption correction	Multi-scan
Max. and Min. transmission	0.980-0.900
F (000)	1360.0

Crystal size	0.213 x 0.198 x 0.041 mm^3
Index ranges	$-26 \leq h \leq 24$, $-8 \leq k \leq 4$, $-28 \leq l \leq 29$
Theta range for data collection	3.789 to 64.998°
Reflections collected	0.0833 (1837)
Diffraction radiation wavelength	1.54184
Independent reflections	3029 [$R_{(int)} = 0.1256$]
Completeness to $\theta = 64.998°$	97.6%
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	3029/0/217
Goodness-of-fit on F^2	1.074
Final *R* indices [I>=2σ (I)]	*R*1 = 0.0833, *wR*2 = 0.2020
R indices (all data)	*R*1 = 0.1370, *wR*2 = 0.2750
Largest diff. peak and hole	0.55/-0.58 e $Å^{-3}$

Compound 10a

CCDC Number = *1886977*



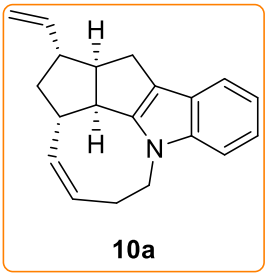
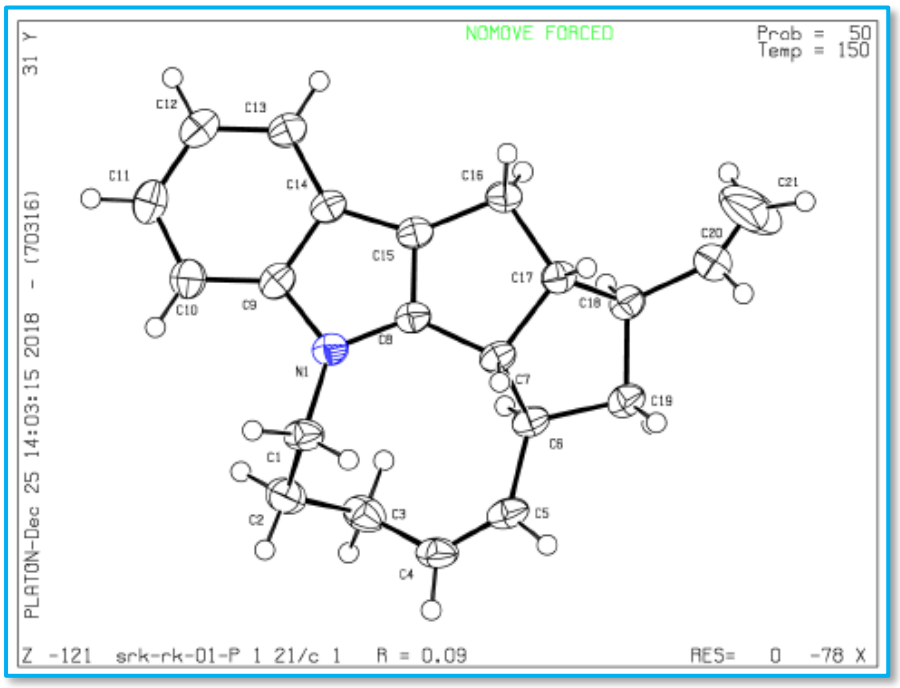


Table S3. X-ray crystallographic data and refinement parameters for **10a (CCDC 1886977)**		
Identification code	SRK-RK-01-81-2_mo	
Empirical formula	$C_{20}H_{21}N$	
Formula weight	275.38	
Temperature	150 K	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 5.3134(3) Å	α = 90°
	b = 12.2320(6) Å	β = 96.223° (5)
	c = 23.2718(11) Å	γ = 90°
Volume	1503.60(13) $Å^3$	
Z	4	
Density (calculated)	1.217 g/cm^3	
Absorption coefficient (μ)	0.070 mm^{-1}	
Absorption correction	Multi-scan	
Max. and Min. transmission	0.994-0.992	
F (000)	592.0	

Crystal size	0.235 x 0.097 x 0.08 mm^3
Index ranges	$-6 \leq h \leq 6$, $-14 \leq k \leq 14$, $-27 \leq l \leq 27$
Theta range for data collection	2.423 to 25.000°
Reflections collected	0.0499 (1990)
Diffraction radiation wavelength	0.71073
Independent reflections	2549 [$R_{(int)} = 0.0753$]
Completeness to $\theta = 24.99°$	96.3%
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	2549/0/190
Goodness-of-fit on F^2	1.057
Final *R* indices [I>=2σ (I)]	*R*1 = 0.0499, *wR*2 = 0.1211
R indices (all data)	*R*1 = 0.0685, *wR*2 = 0.1397
Largest diff. peak and hole	0.25/-0.33 e $Å^{-3}$

Compound 11a

CCDC Number = *1887288*



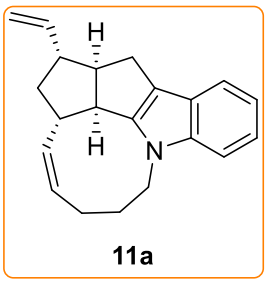


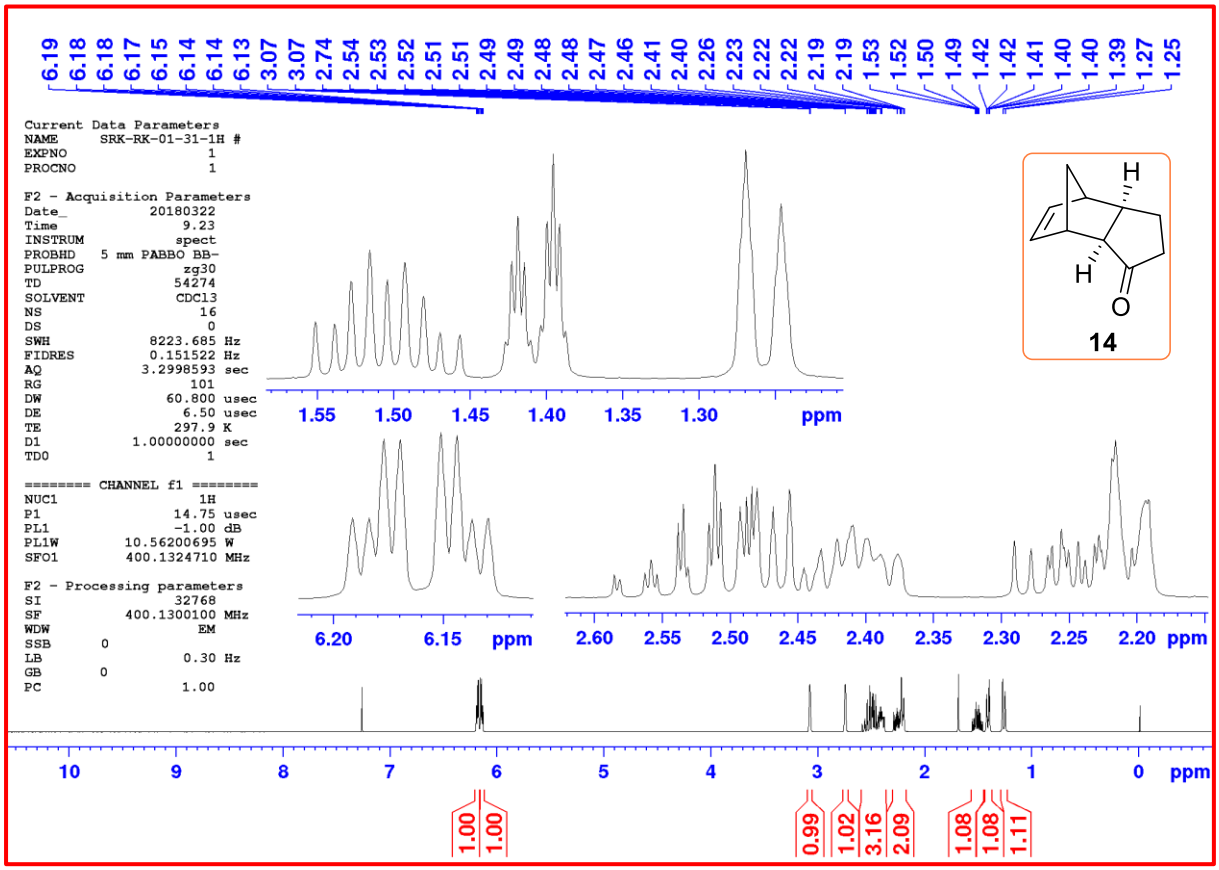
Table S4. X-ray crystallographic data and refinement parameters for **11a (CCDC 1887288)**

Identification code	SRK-RK-01-82_mo
Empirical formula	$C_{21}H_{23}N$
Formula weight	289.40
Temperature	150 K
Crystal system	Monoclinic
Space group	$P2_1/c$
Unit cell dimensions	a = 14.3351 (8) Å α = 90°
	b = 5.0684 (3) Å β = 98.988° (5)
	c = 22.3710 (14) Å γ = 90°
Volume	1605.43 (17) $Å^3$
Z	4
Density (calculated)	1.197 g/cm^3
Absorption coefficient (μ)	0.069 mm^{-1}
Absorption correction	Multi-scan
Max. and Min. transmission	0.998-0.993
F (000)	624.0
Crystal size	0.302 x 0.087 x 0.035 mm^3

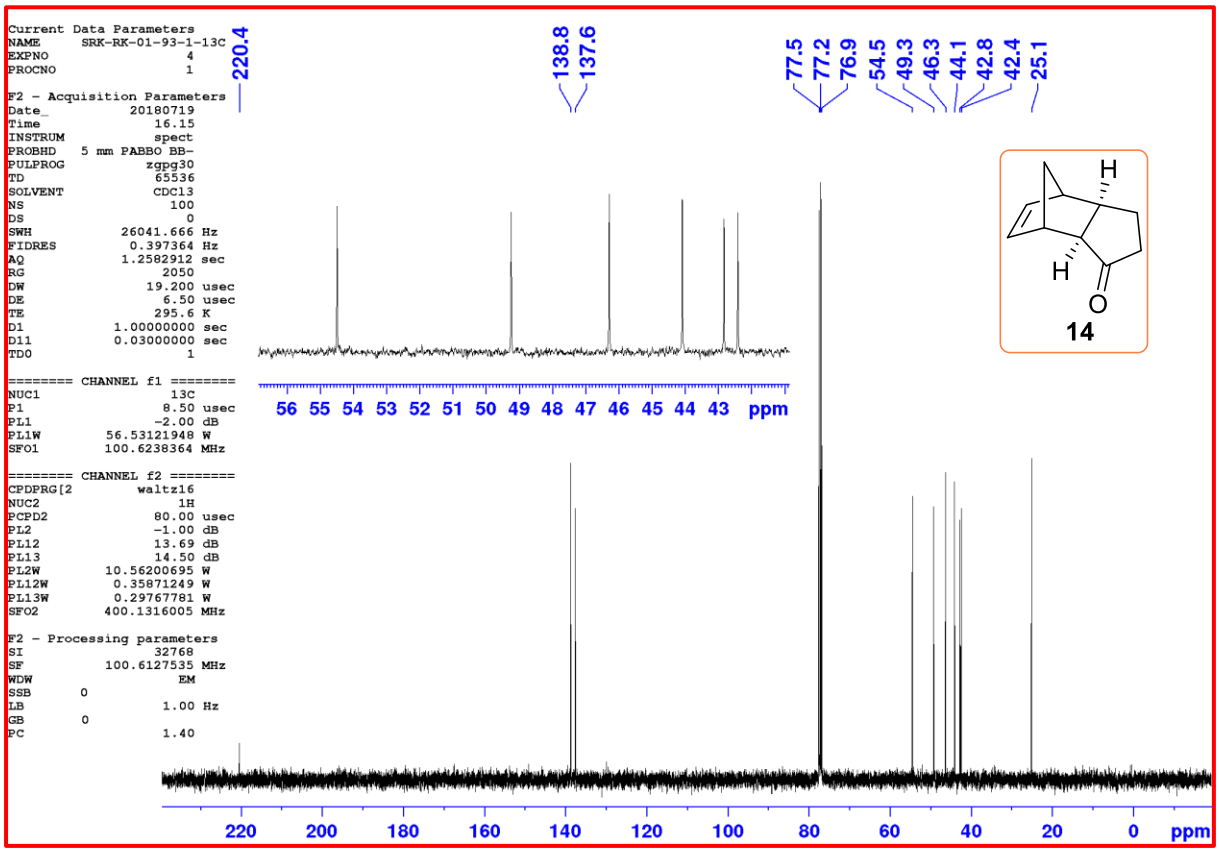
Index ranges	$-17 \leq h \leq 17$, $-6 \leq k \leq 5$, $-26 \leq l \leq 26$
Theta range for data collection	1.843 to 24.998°
Reflections collected	0.0857 (2063)
Diffraction radiation wavelength	0.71073
Independent reflections	2804 [$R_{(int)} = 0.0778$]
Completeness to $\theta = 24.998°$	100%
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	2804/0/199
Goodness-of-fit on F^2	1.075
Final *R* indices [I>=2σ (I)]	*R*1 = 0.0857, *wR*2 = 0.2324
R indices (all data)	*R*1 = 0.1153, *wR*2 = 0.2734
Largest diff. peak and hole	1.48/-1.11 e $Å^{-3}$

2. Plots of ^{1}H NMR, ^{13}C NMR and DEPT-135 spectral data all new compounds

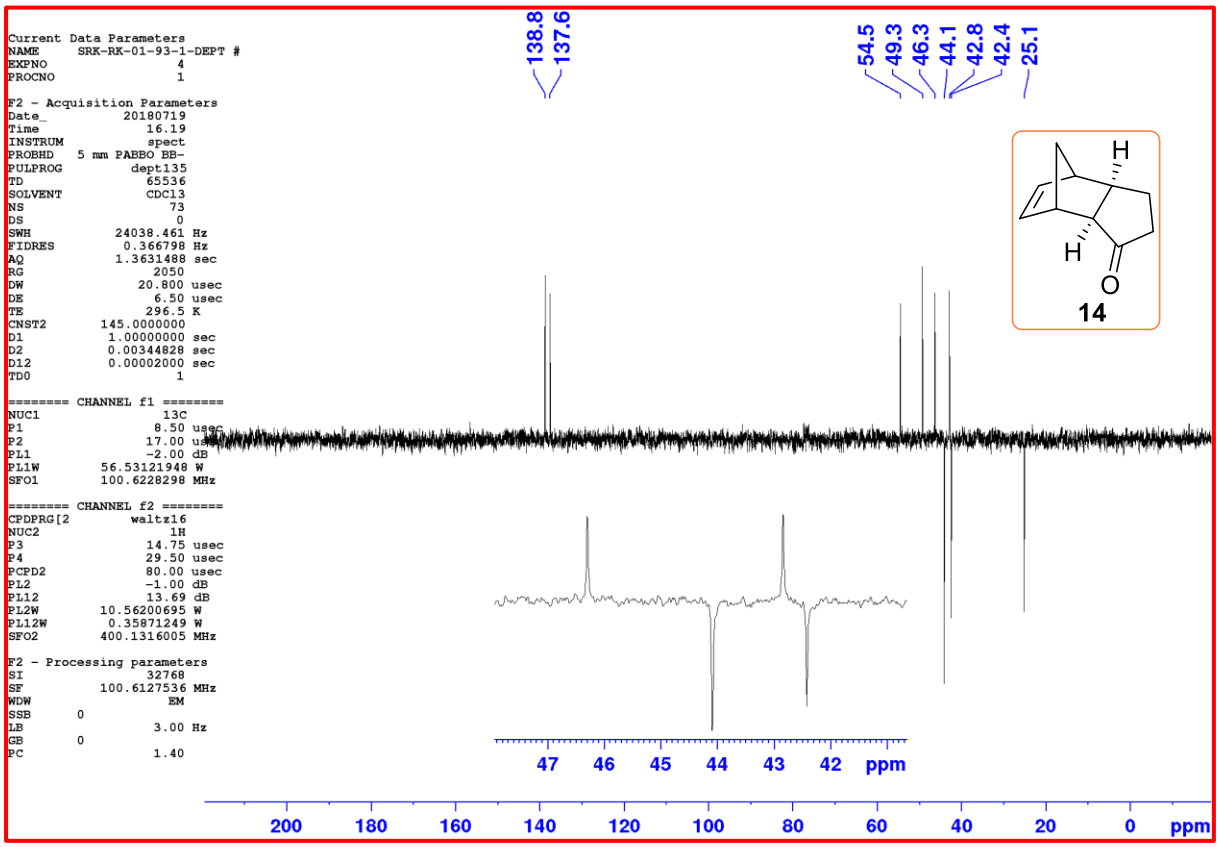
^{1}H NMR (400 MHz, $CDCl_3$) of **14**



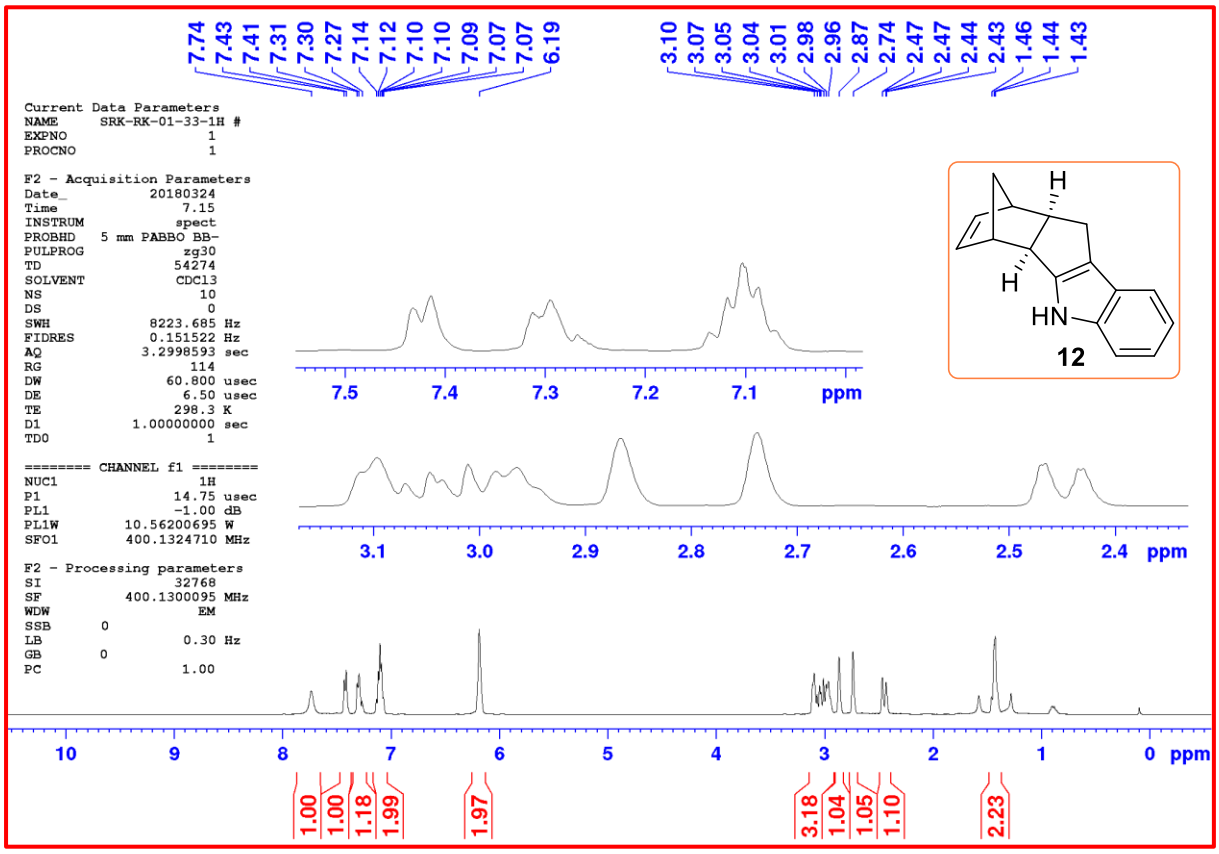
^{13}C NMR (100 MHz, $CDCl_3$) of **14**



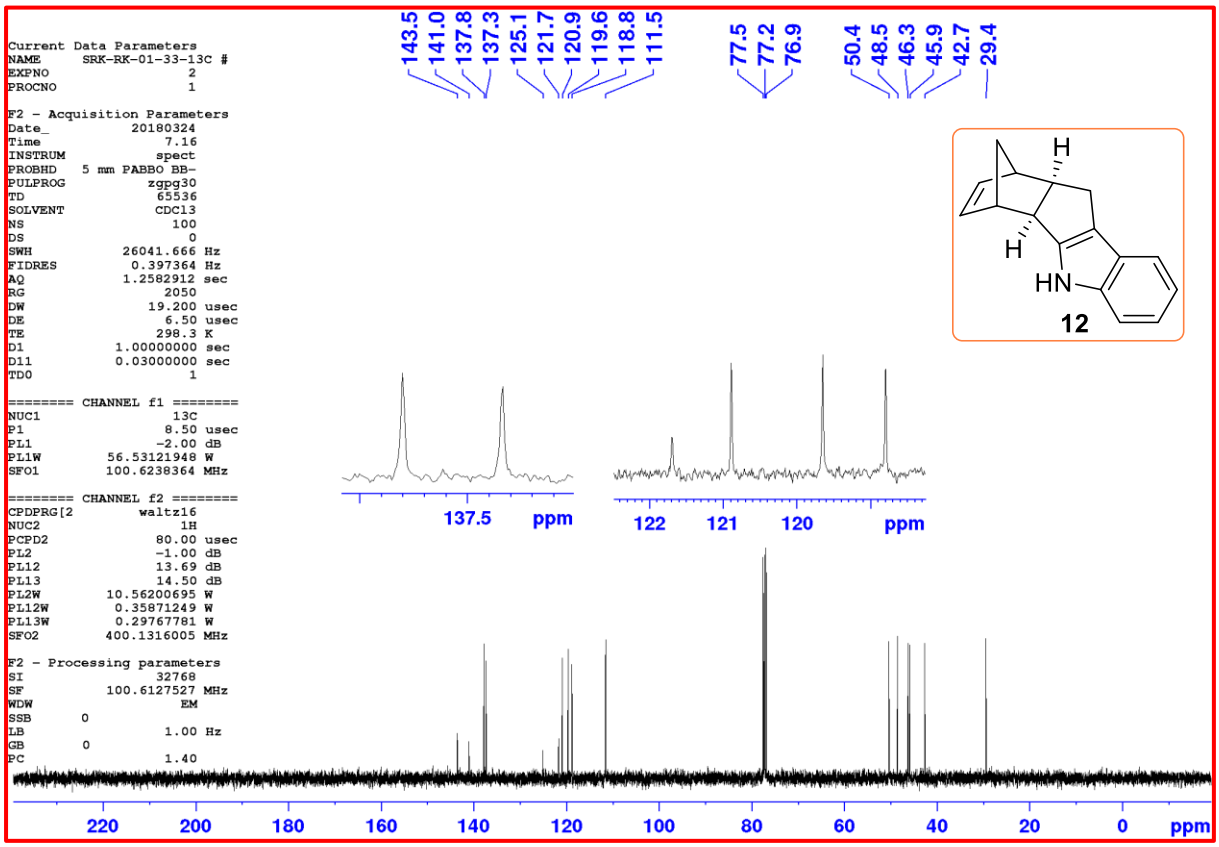
DEPT-135 (100 MHz, $CDCl_3$) of **14**



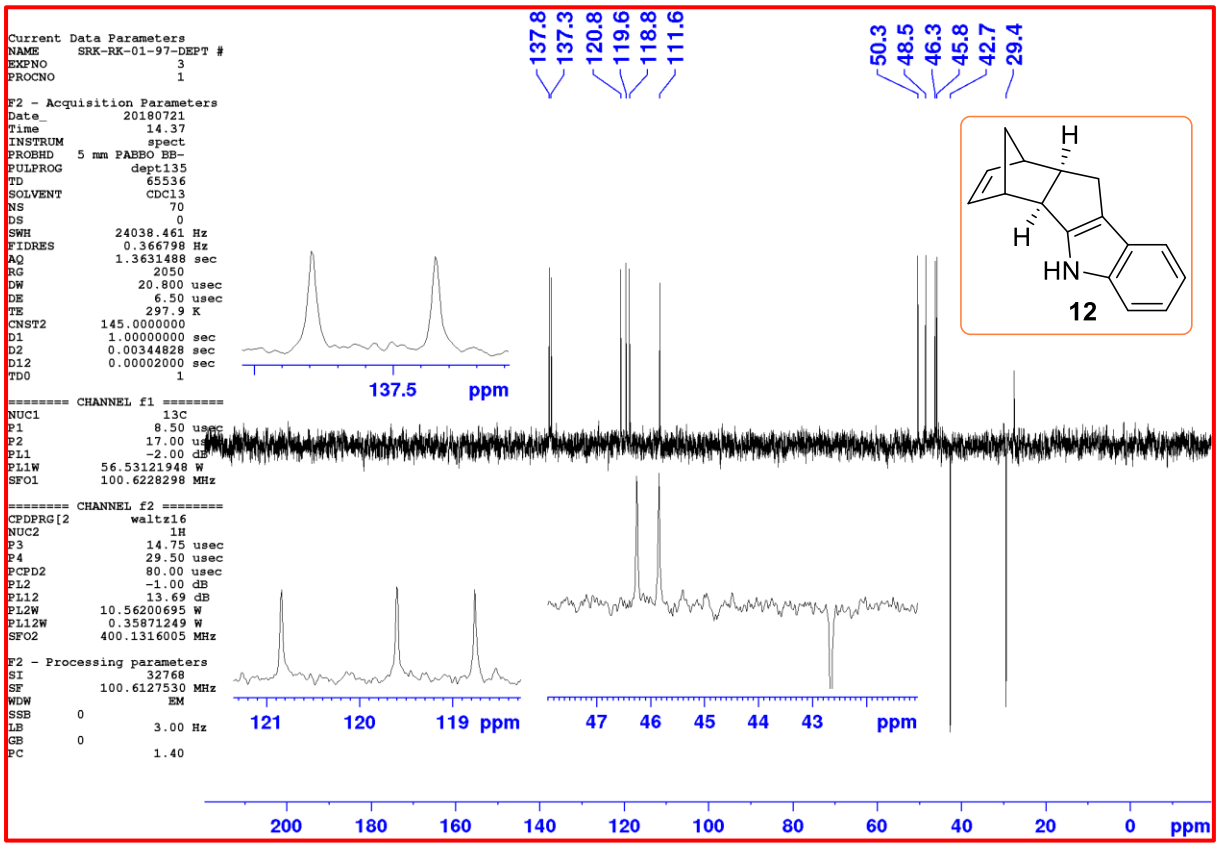
^{1}H NMR (400 MHz, $CDCl_3$) of **12**



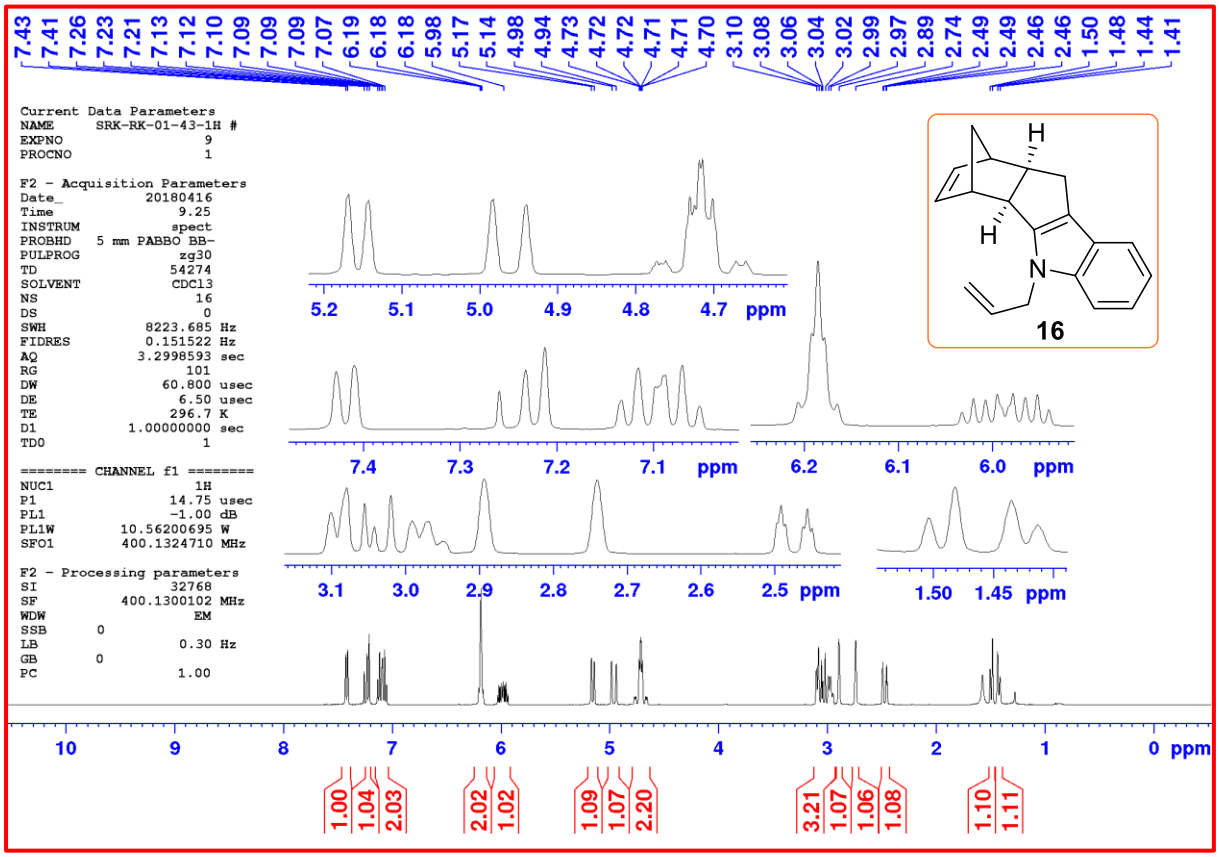
^{13}C NMR (100 MHz, $CDCl_3$) of **12**



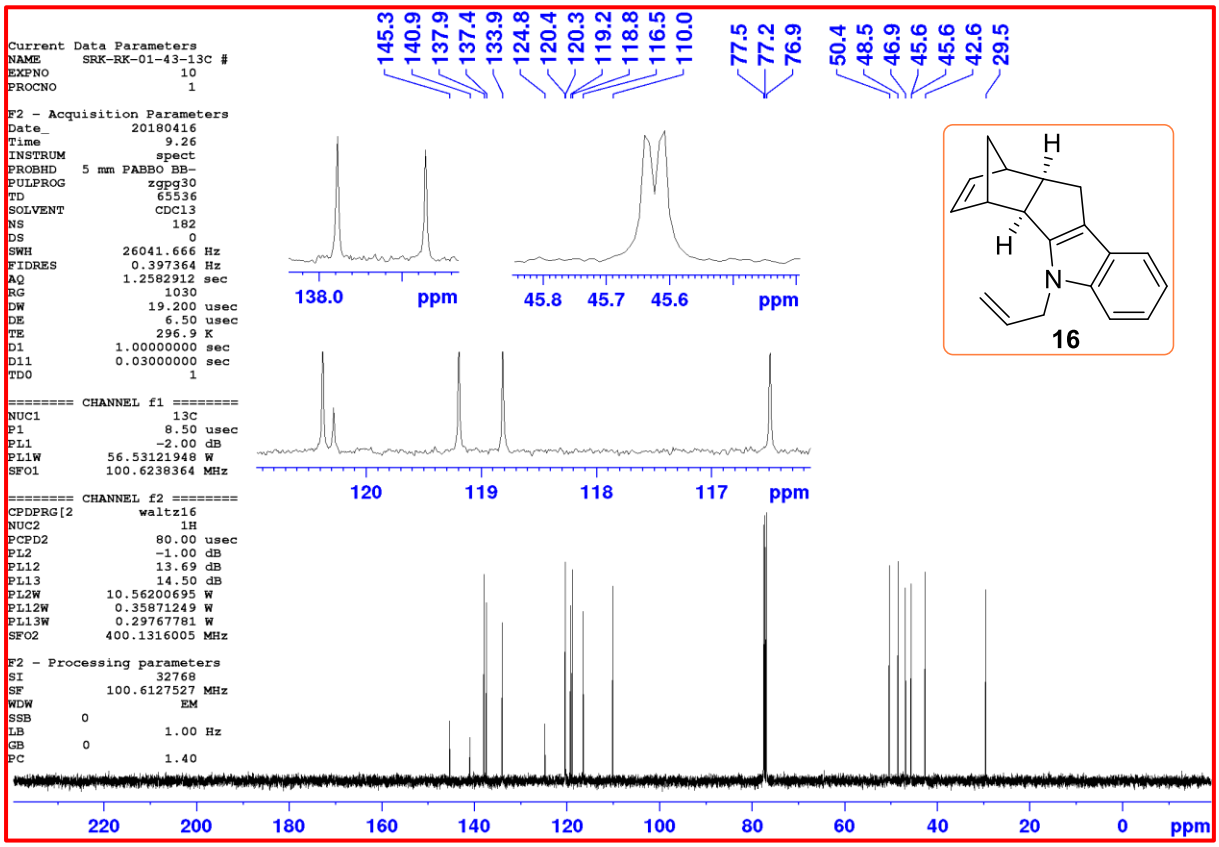
DEPT-135 (100 MHz, $CDCl_3$) of **12**



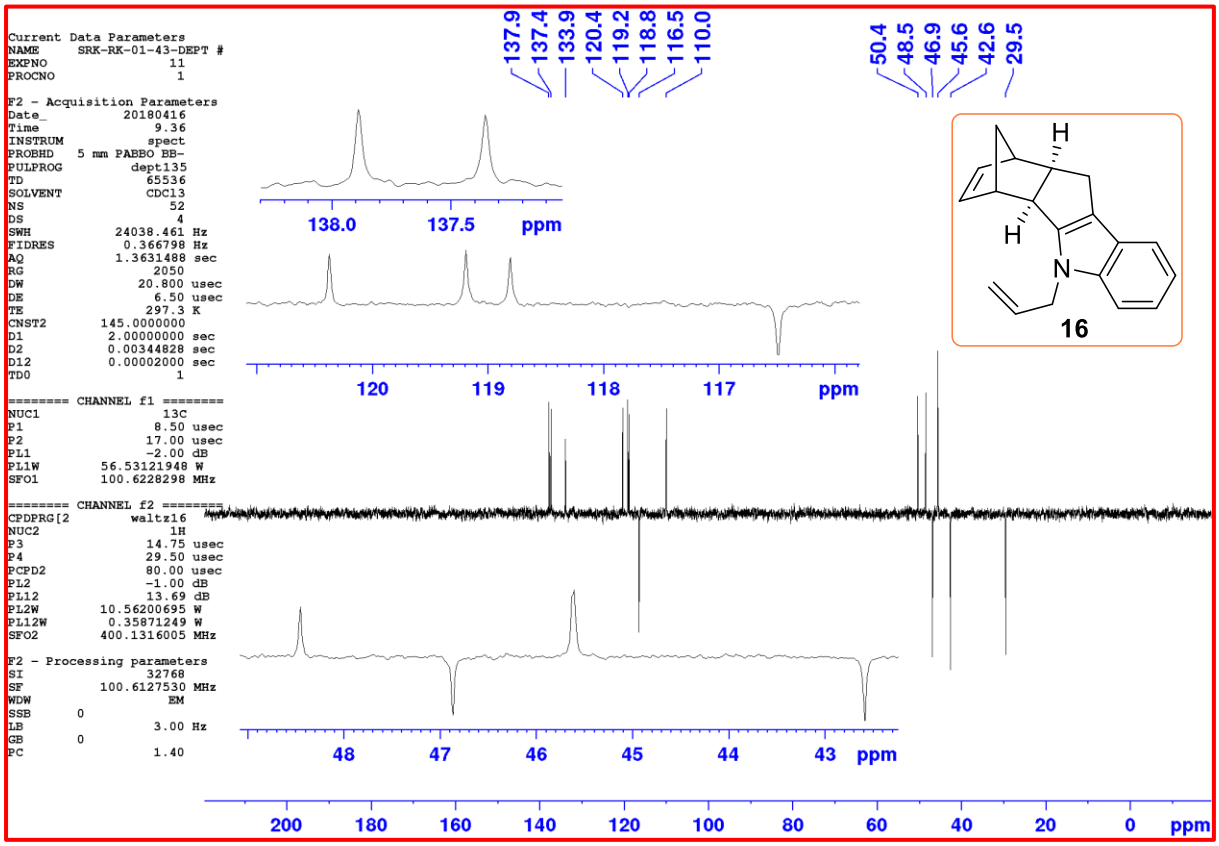
^{1}H NMR (400 MHz, $CDCl_3$) of **16**



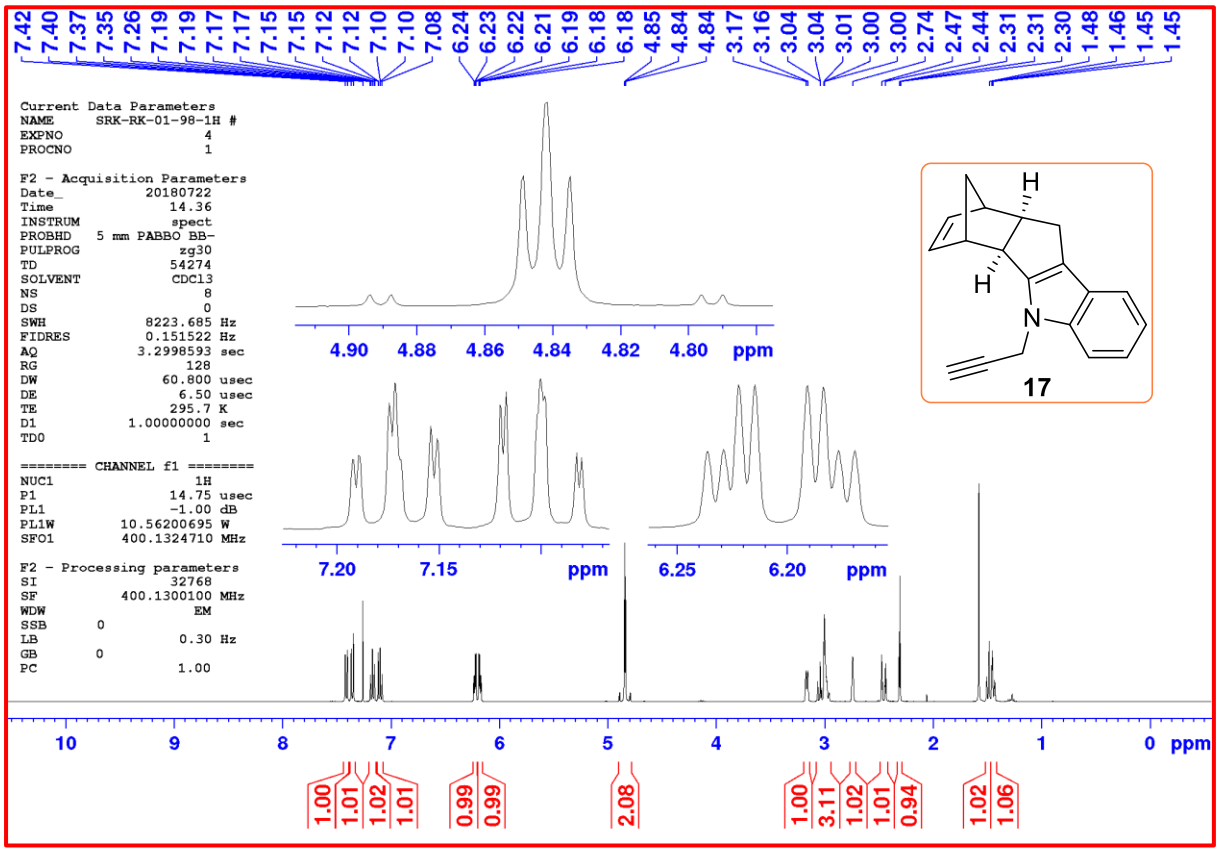
^{13}C NMR (100 MHz, $CDCl_3$) of **16**



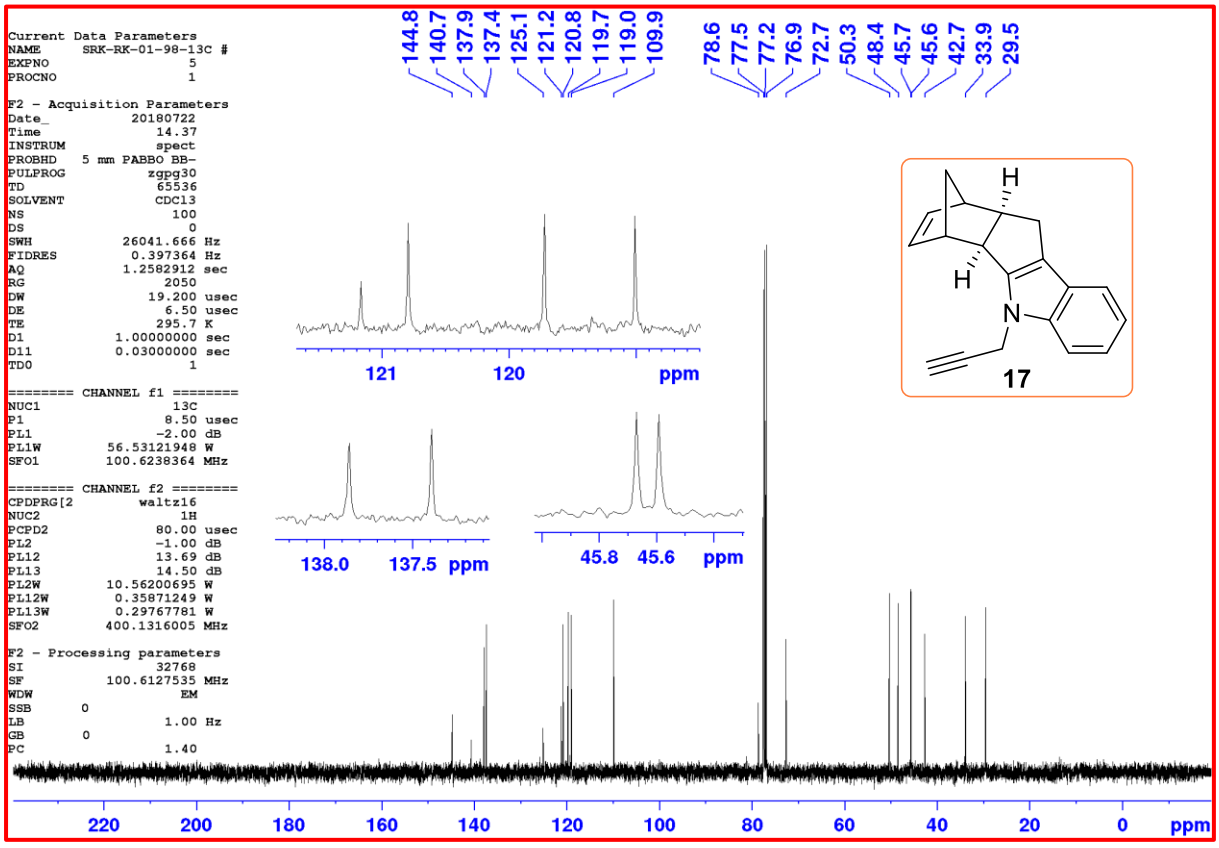
DEPT-135 (100 MHz, $CDCl_3$) of **16**



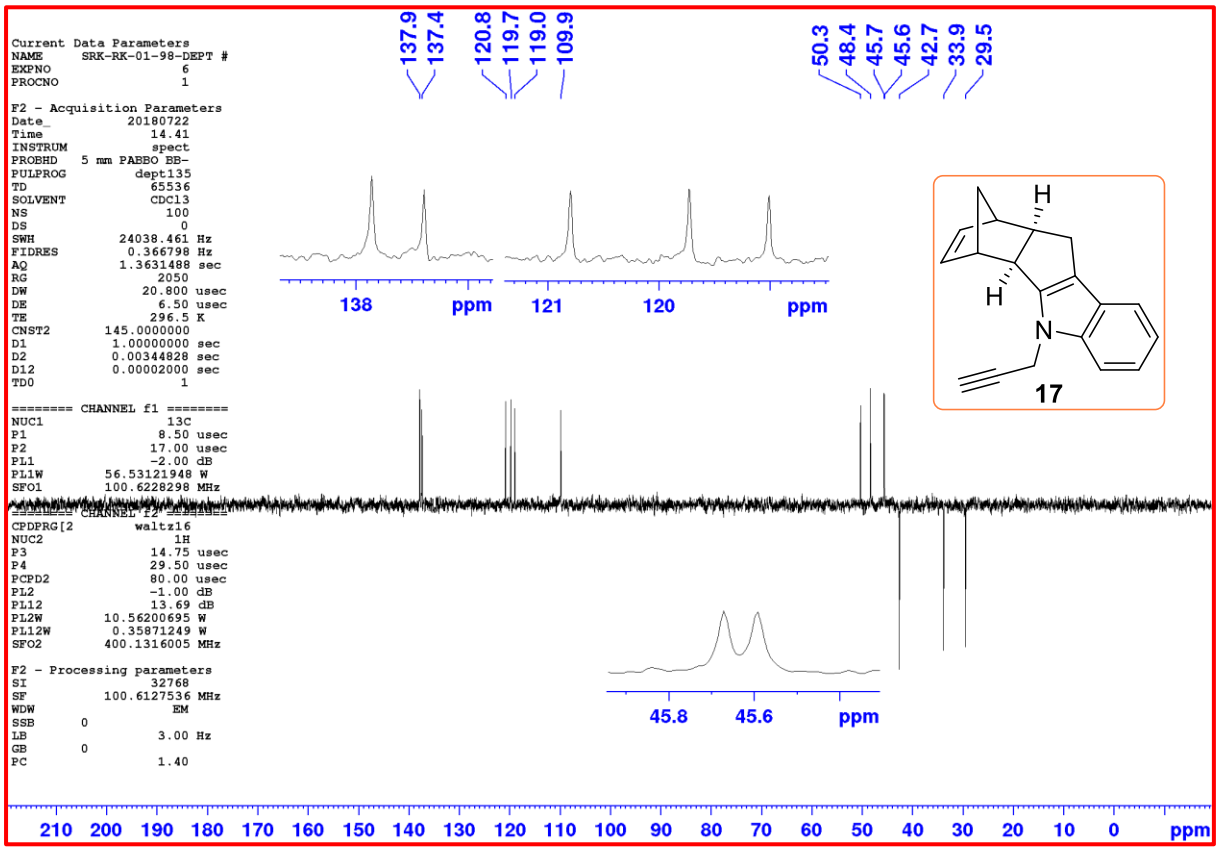
^{1}H NMR (400 MHz, $CDCl_3$) of **17**



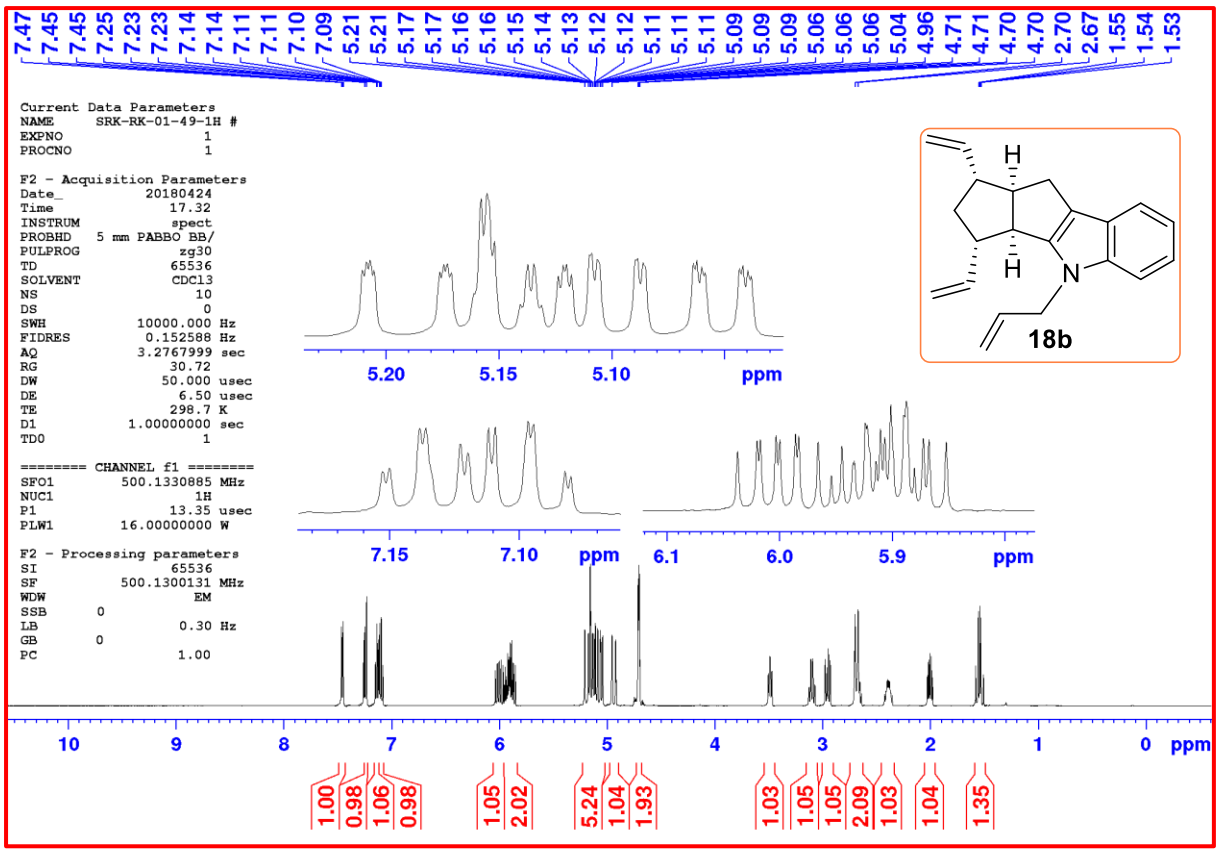
^{13}C NMR (100 MHz, $CDCl_3$) of **17**



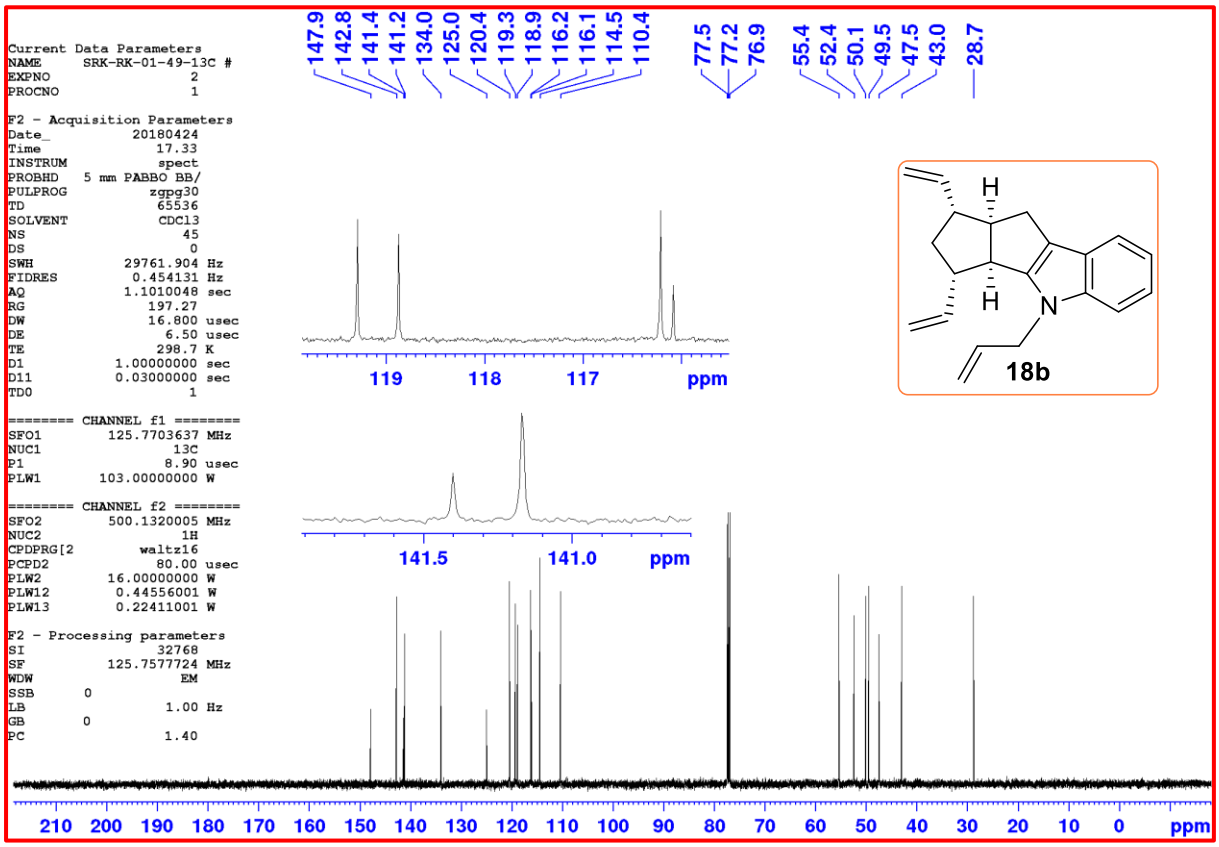
DEPT-135 (100 MHz, $CDCl_3$) of **17**



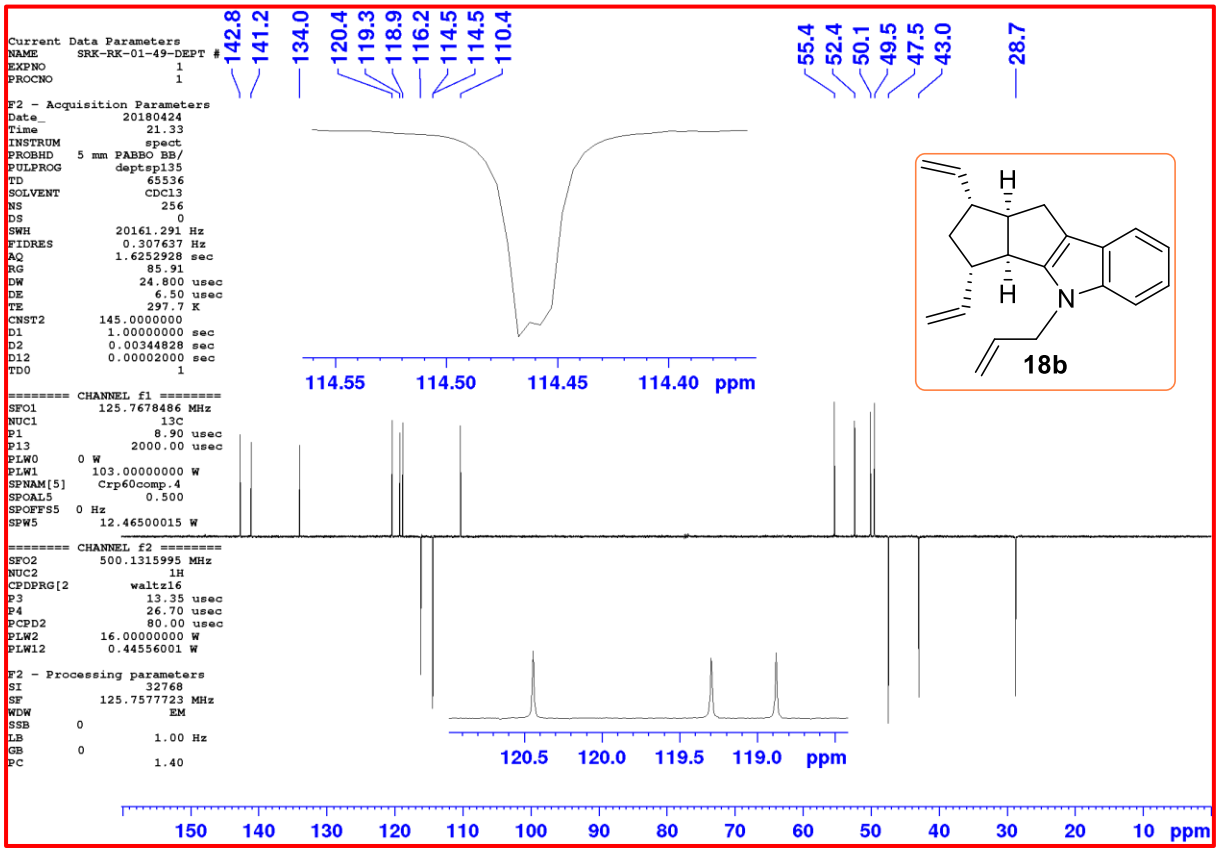
^{1}H NMR (500 MHz, $CDCl_3$) of **18b**



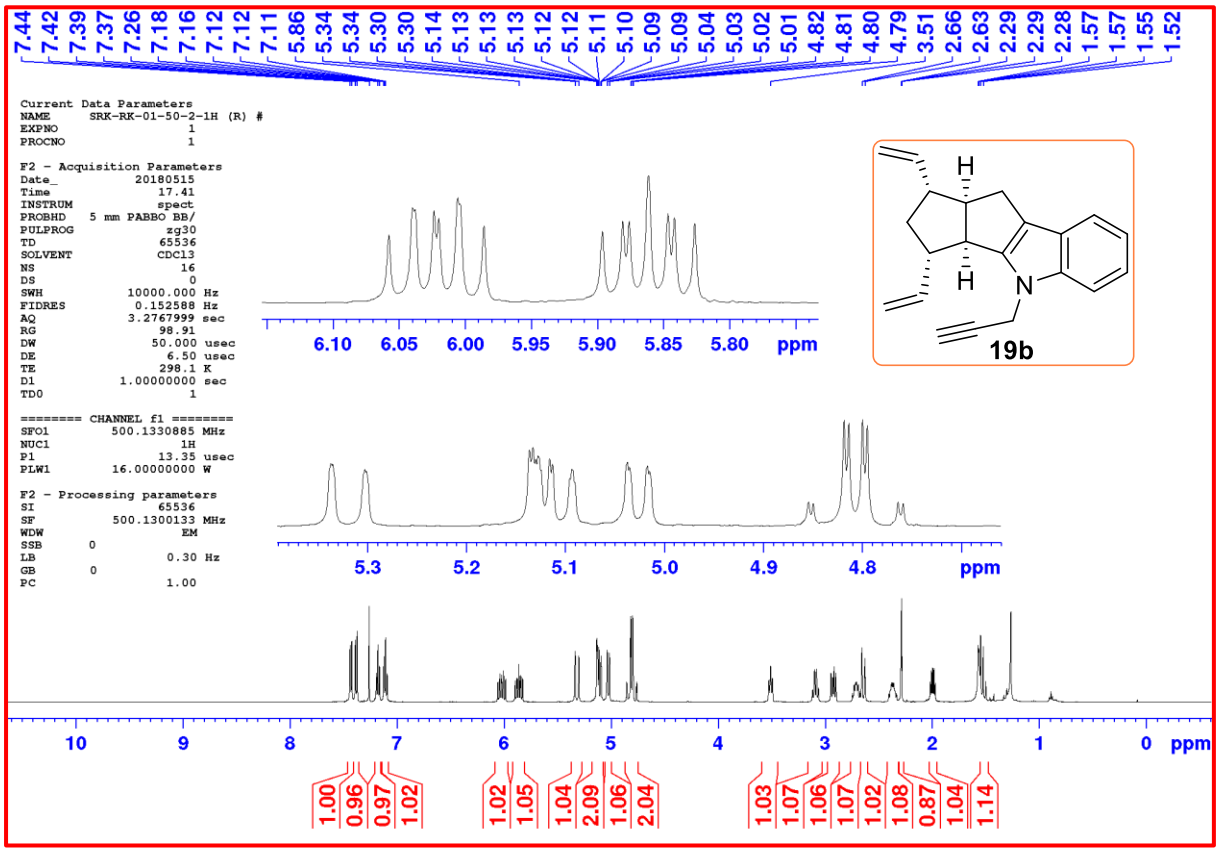
^{13}C NMR (125 MHz, $CDCl_3$) of **18b**



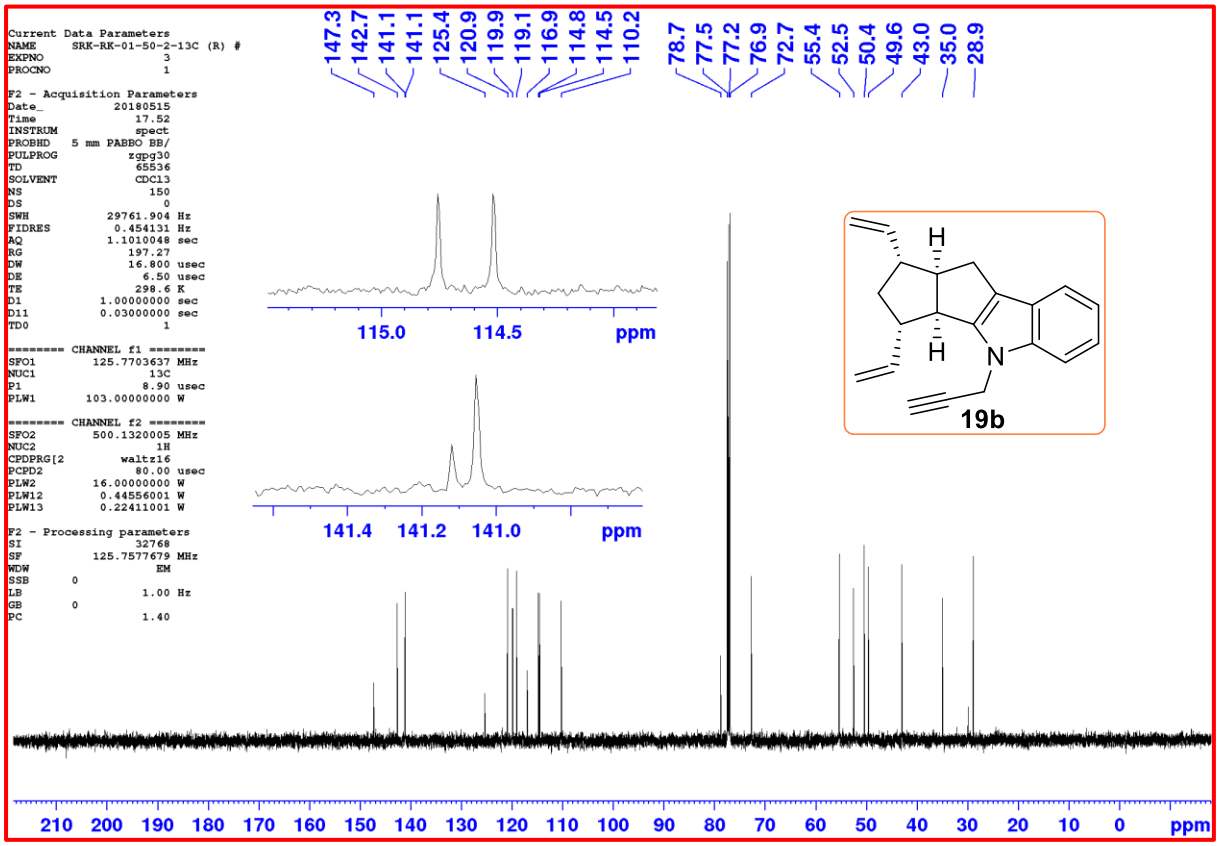
DEPT-135 (125 MHz, $CDCl_3$) of **18b**



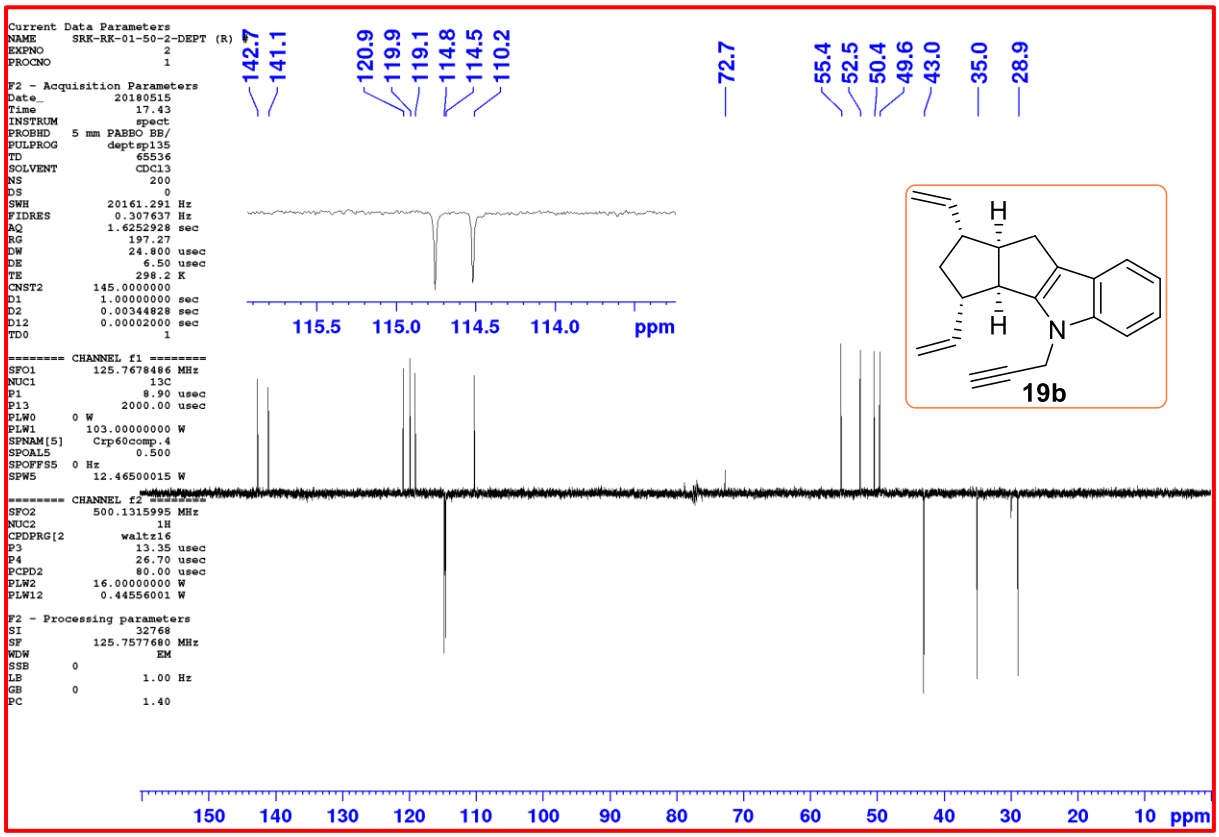
^{1}H NMR (500 MHz, $CDCl_3$) of **19b**



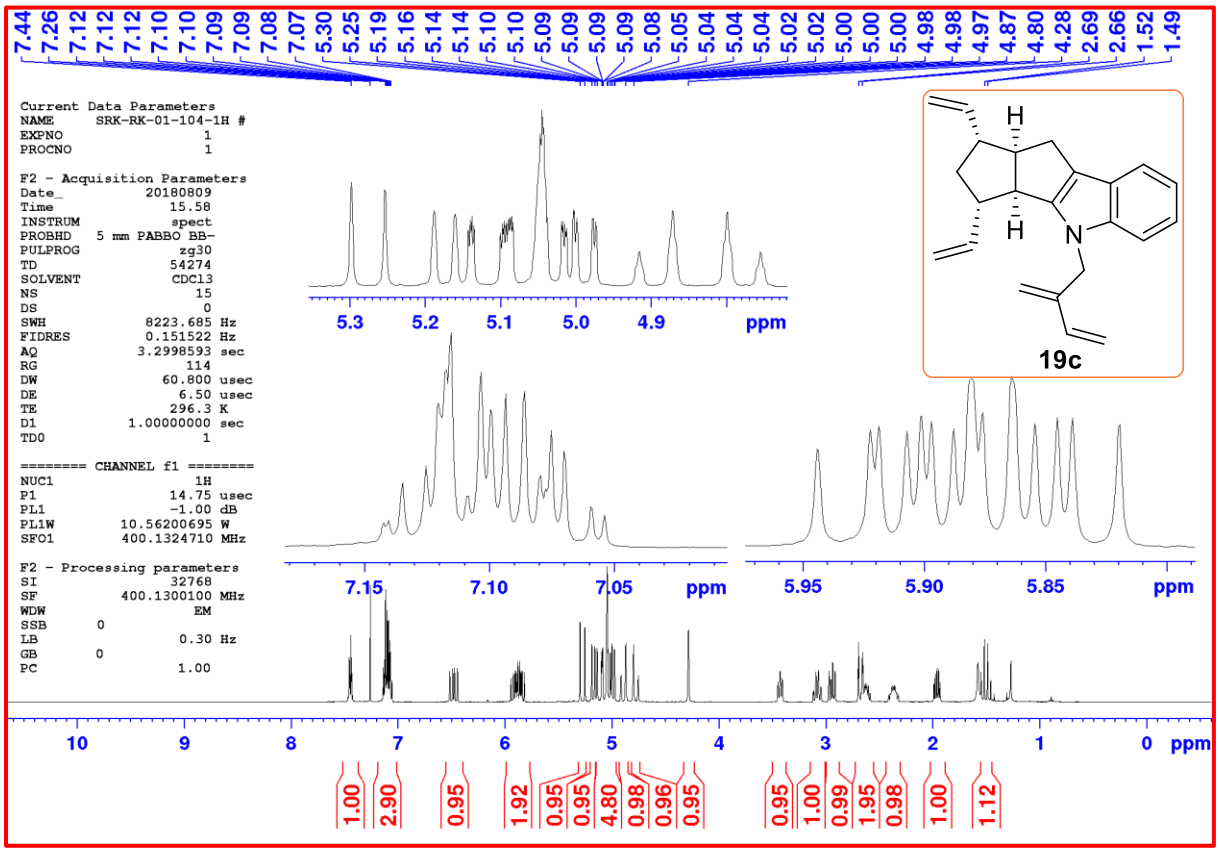
^{13}C NMR (125 MHz, $CDCl_3$) of **19b**



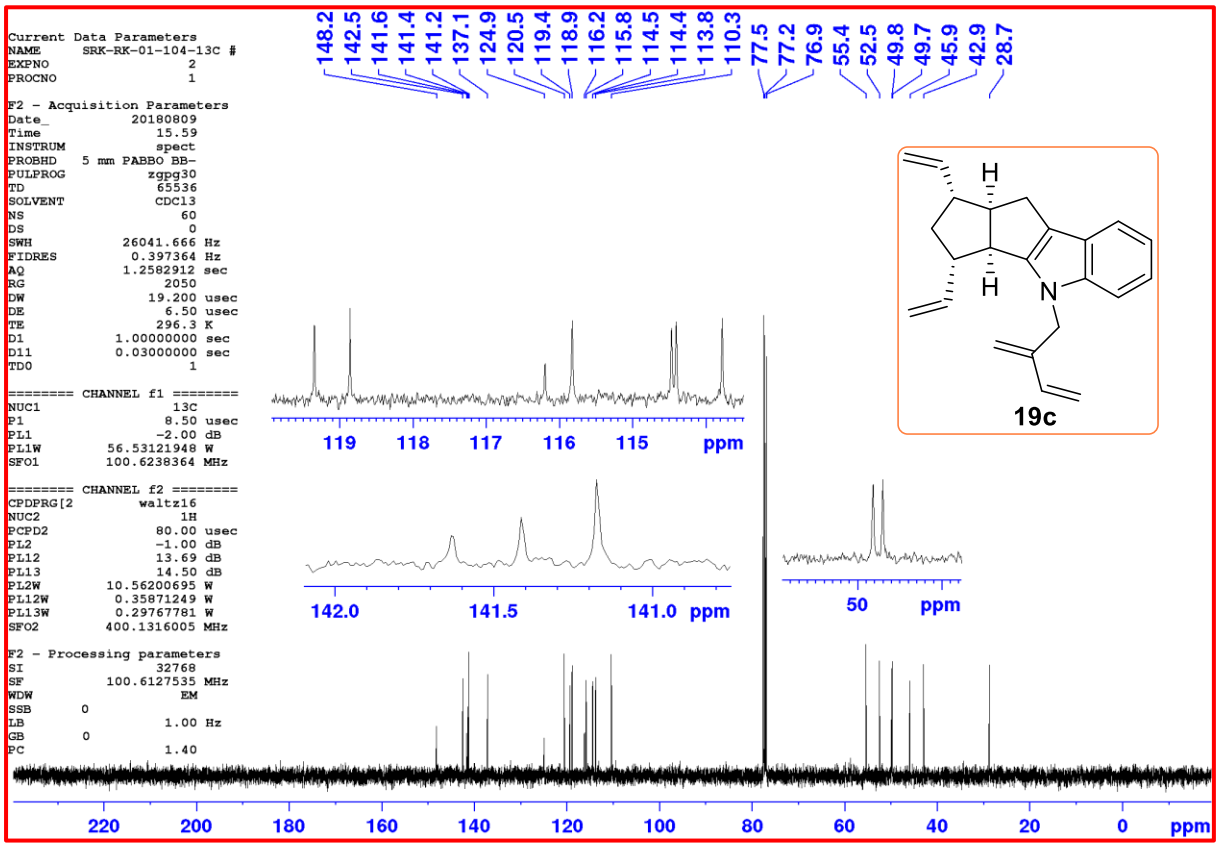
DEPT-135 (125 MHz, $CDCl_3$) of **19b**



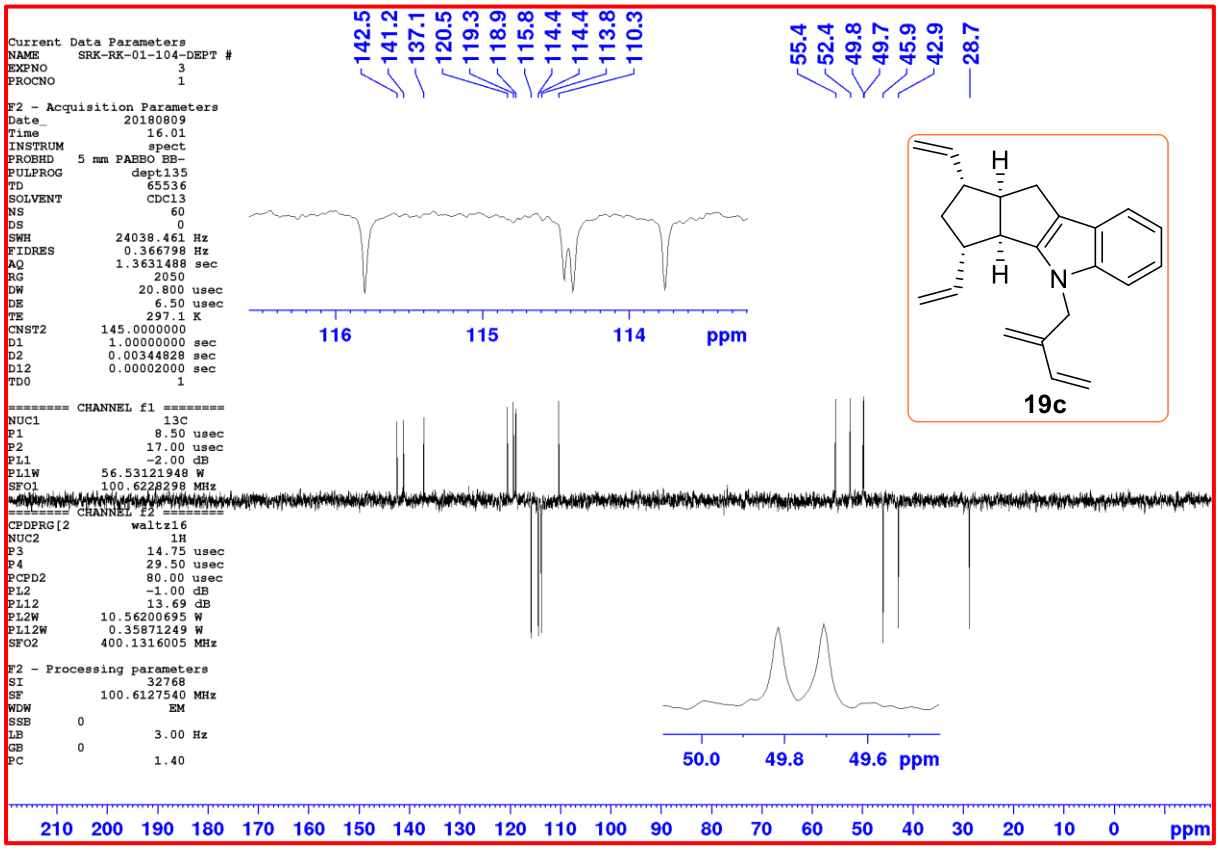
^{1}H NMR (400 MHz, $CDCl_3$) of **19c**



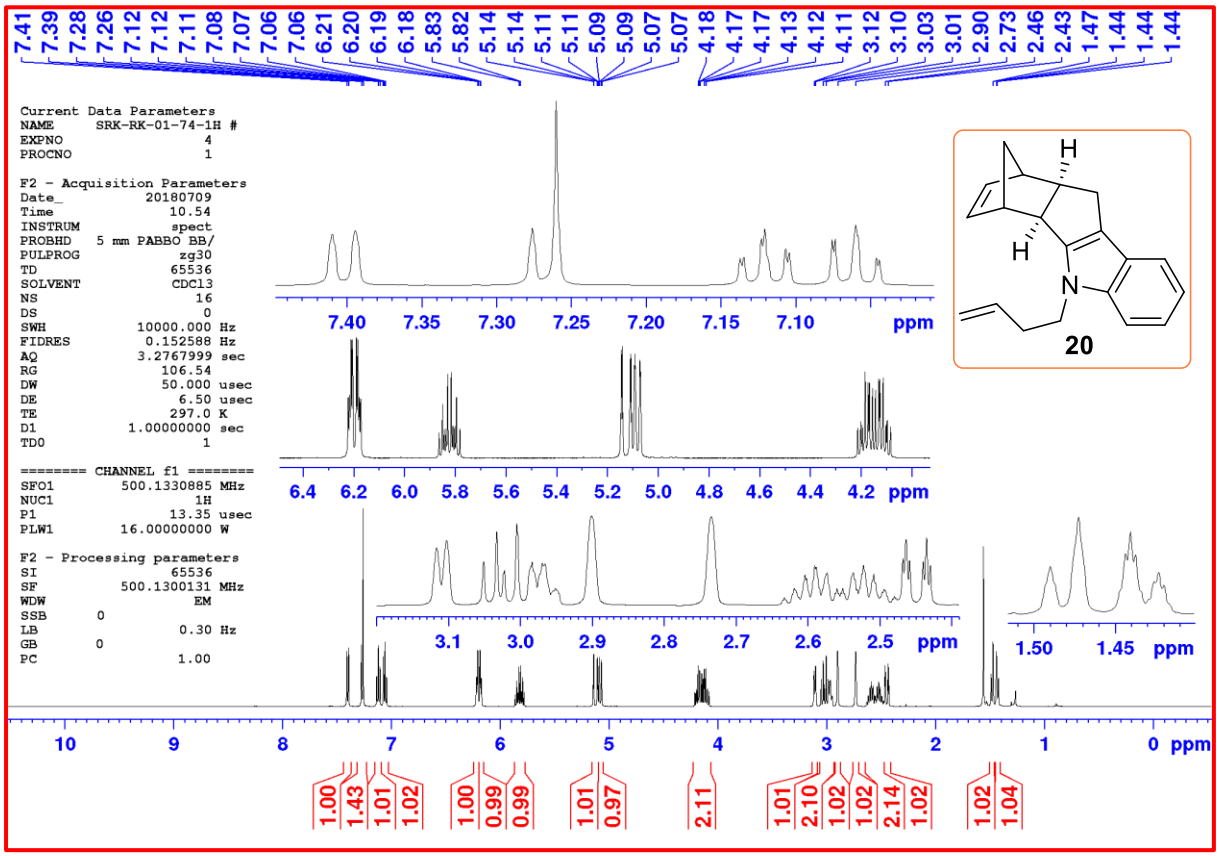
^{13}C NMR (100 MHz, $CDCl_3$) of **19c**



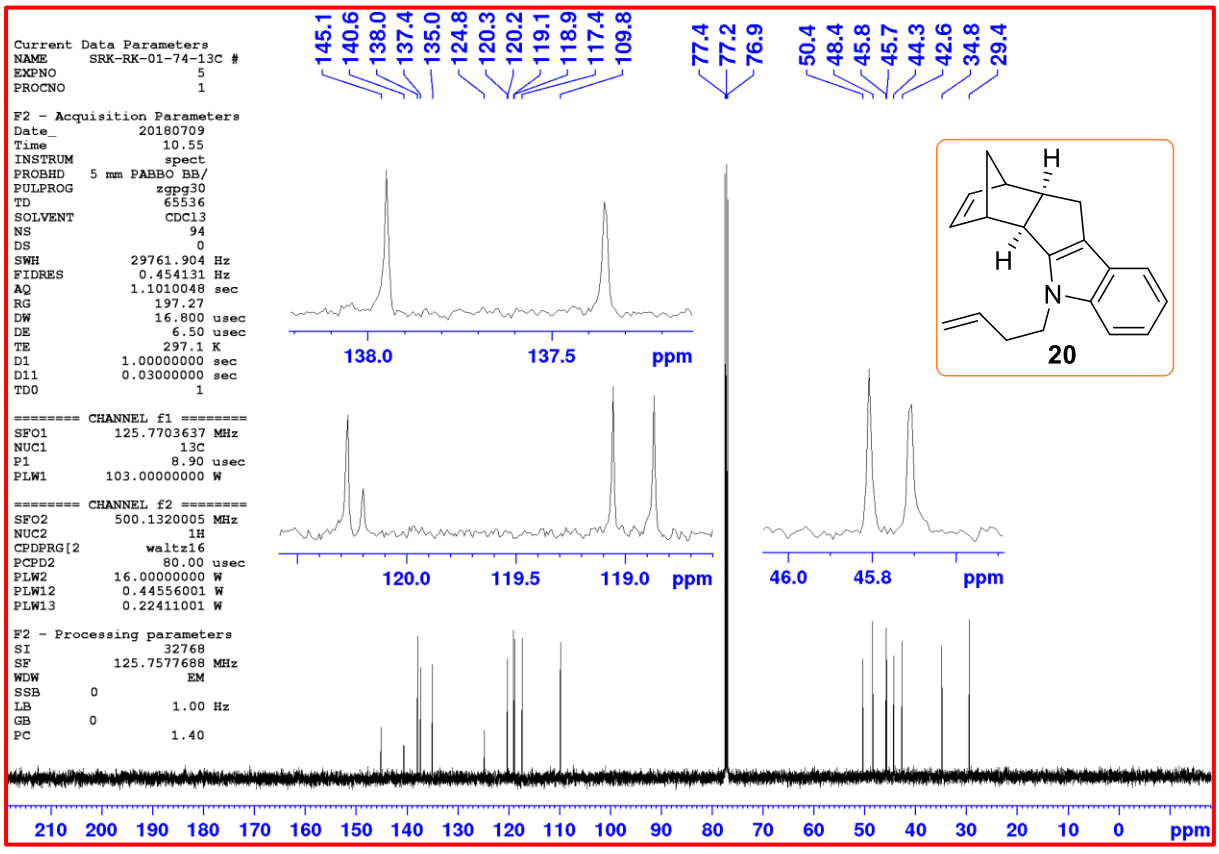
DEPT-135 (100 MHz, $CDCl_3$) of **19c**



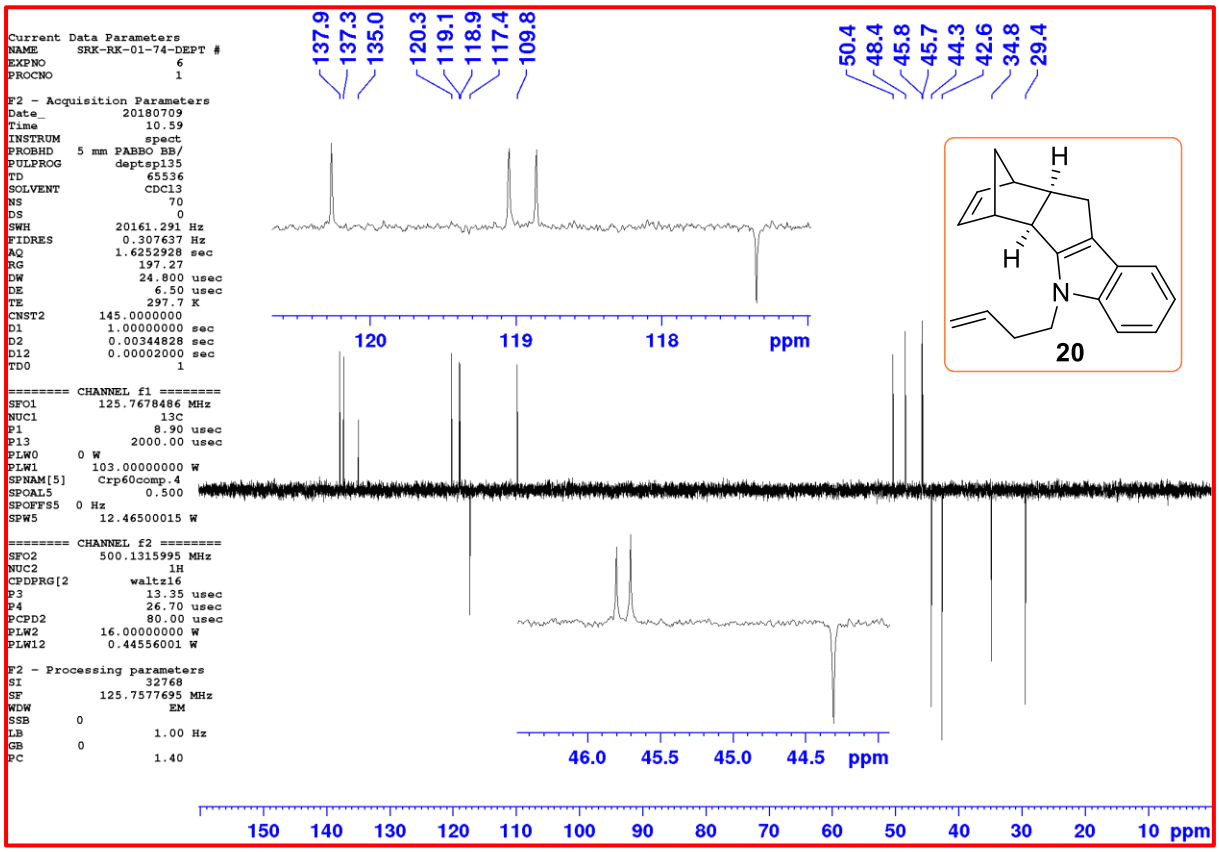
^{1}H NMR (500 MHz, $CDCl_3$) of **20**



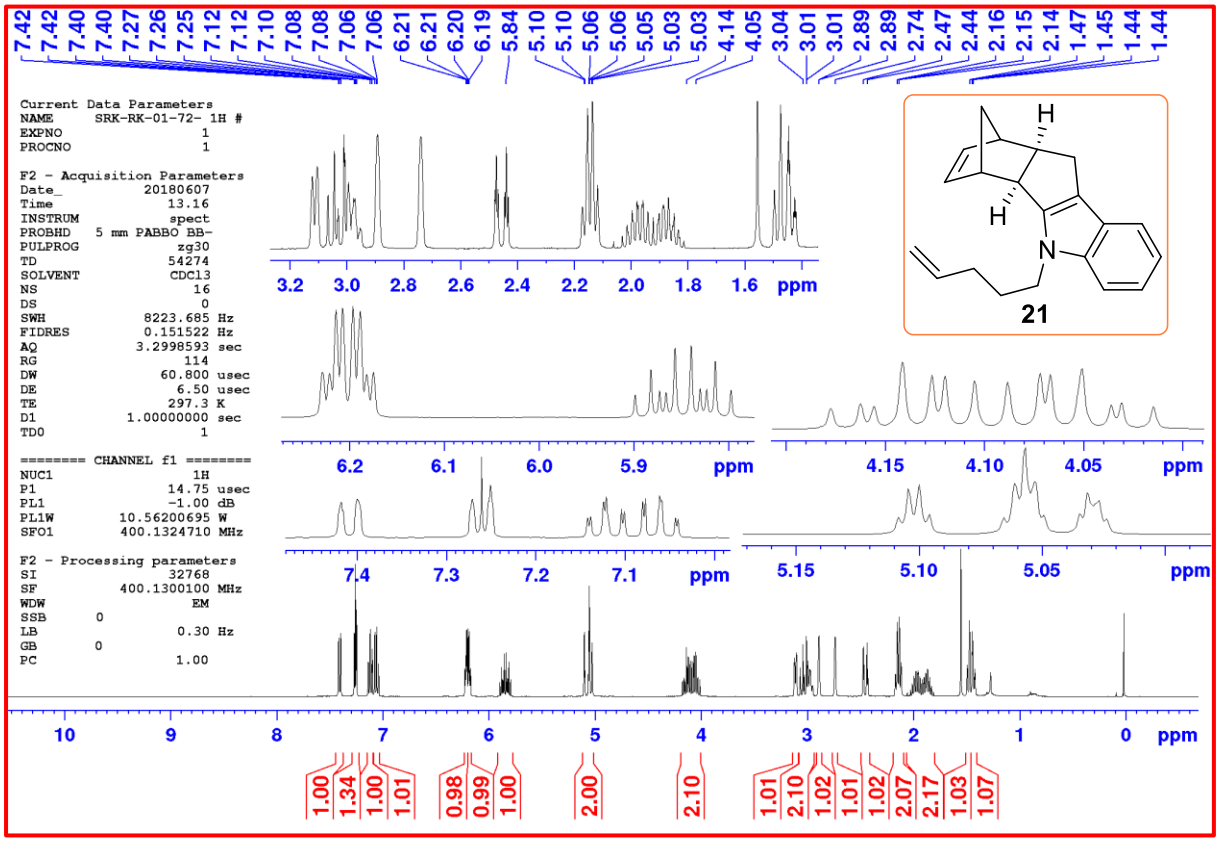
^{13}C NMR (125 MHz, $CDCl_3$) of **20**



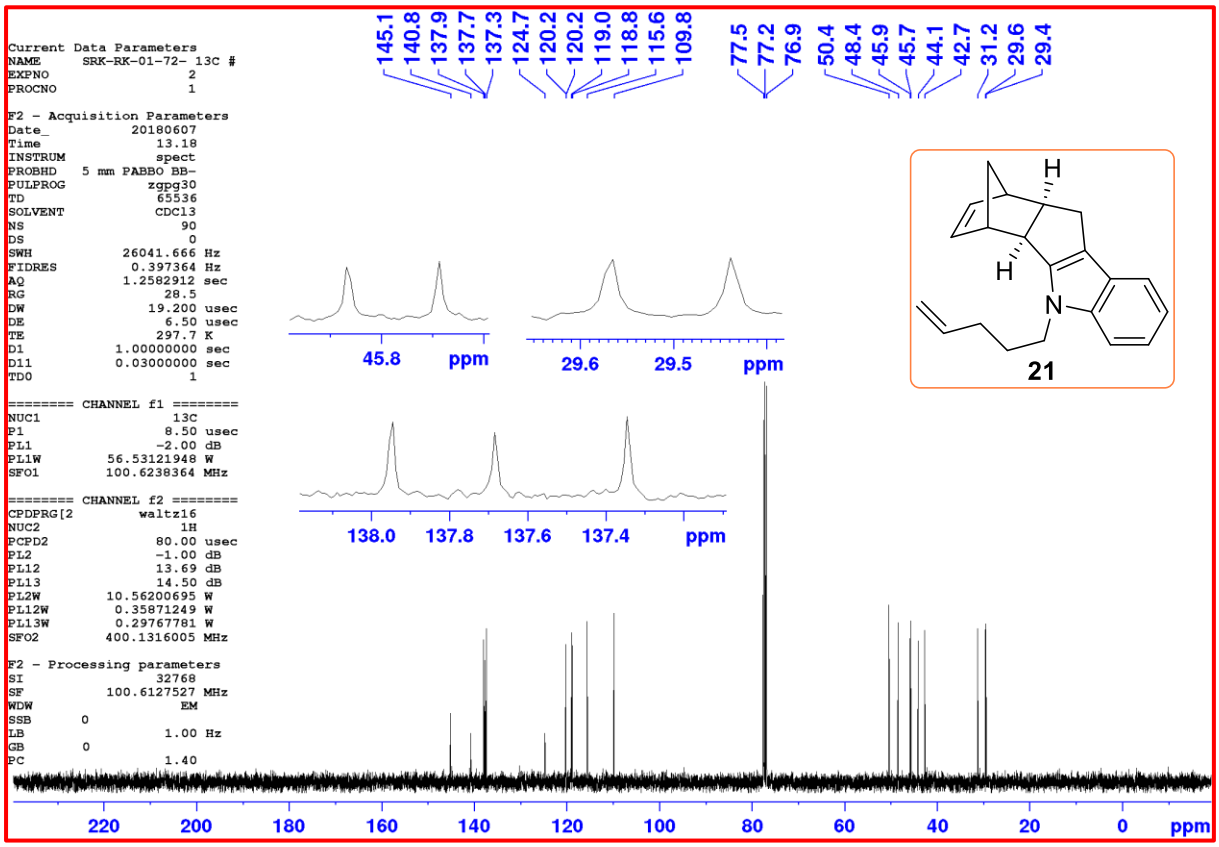
DEPT-135 (125 MHz, $CDCl_3$) of **20**



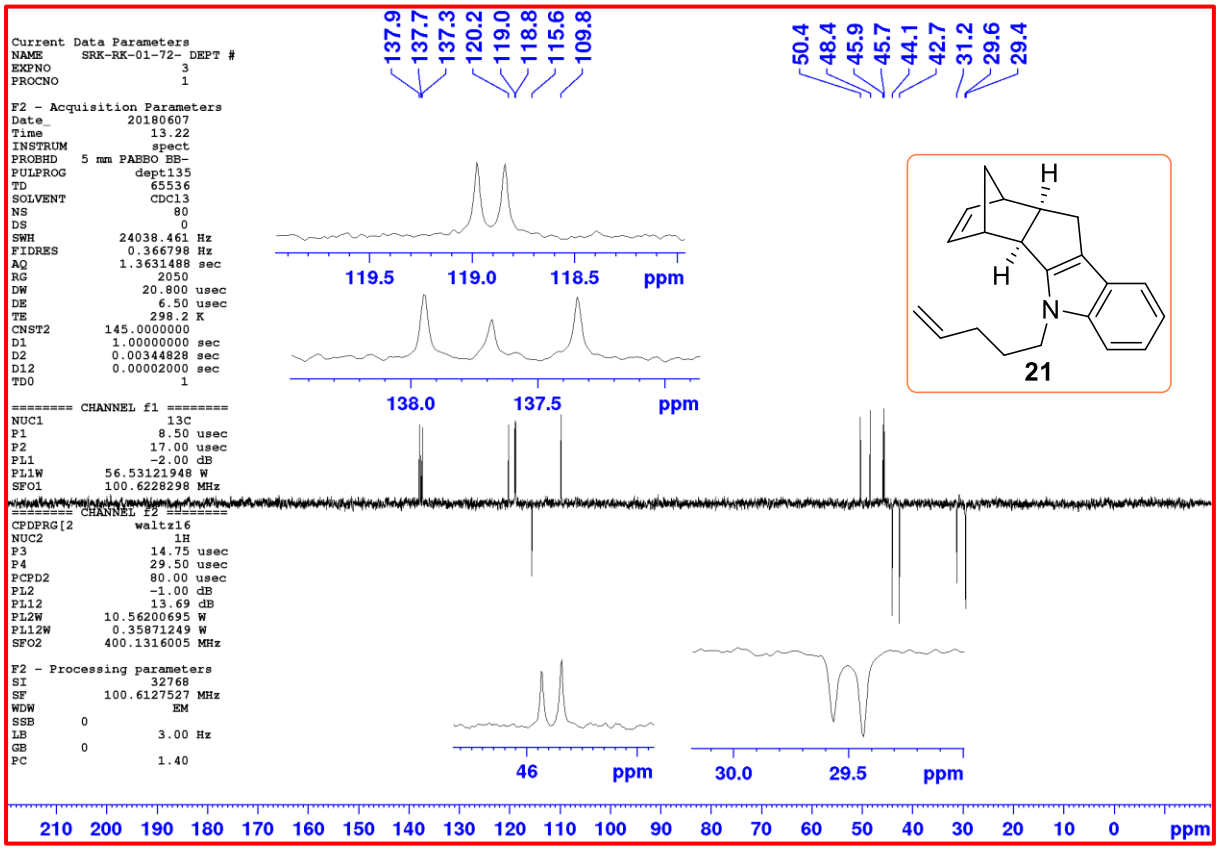
^{1}H NMR (400 MHz, $CDCl_3$) of **21**



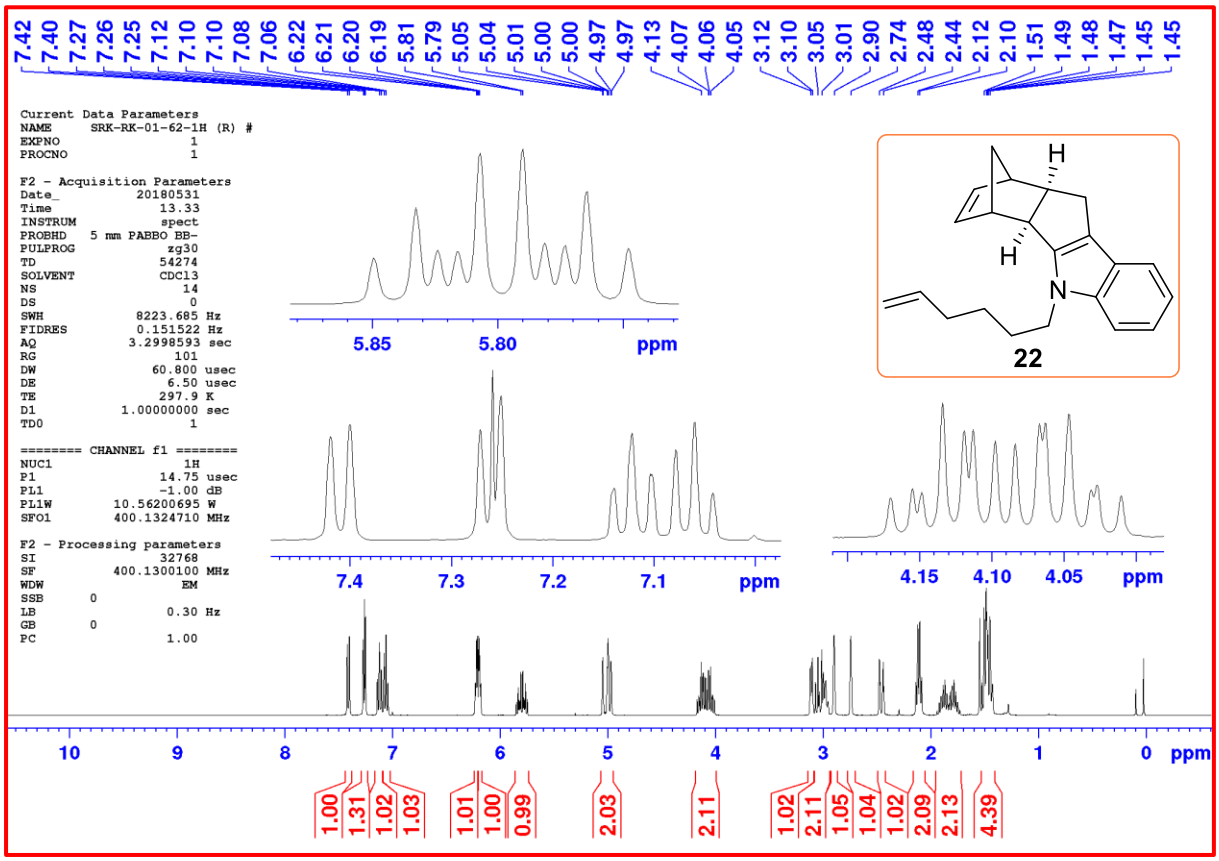
^{13}C NMR (100 MHz, $CDCl_3$) of **21**



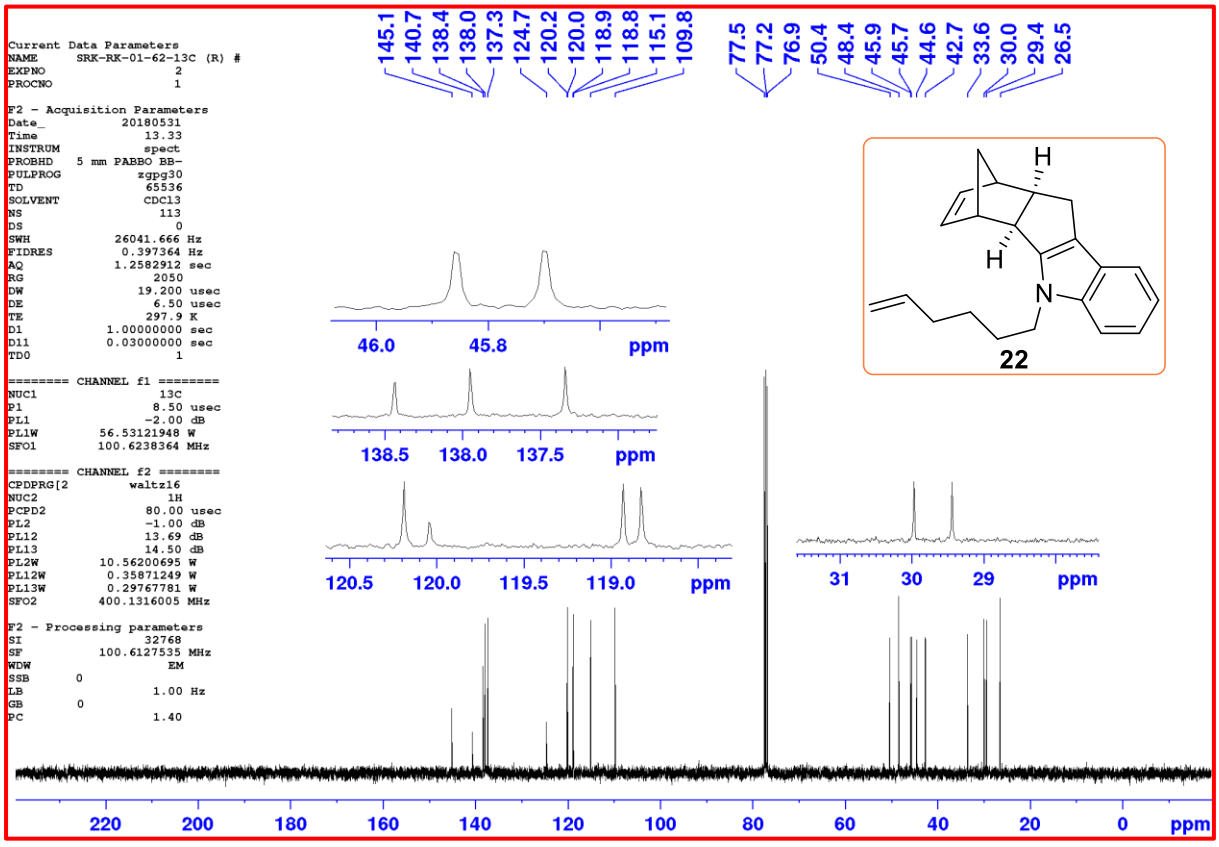
DEPT-135 (100 MHz, $CDCl_3$) of **21**



^{1}H NMR (400 MHz, $CDCl_3$) of **22**



^{13}C NMR (100 MHz, $CDCl_3$) of **22**



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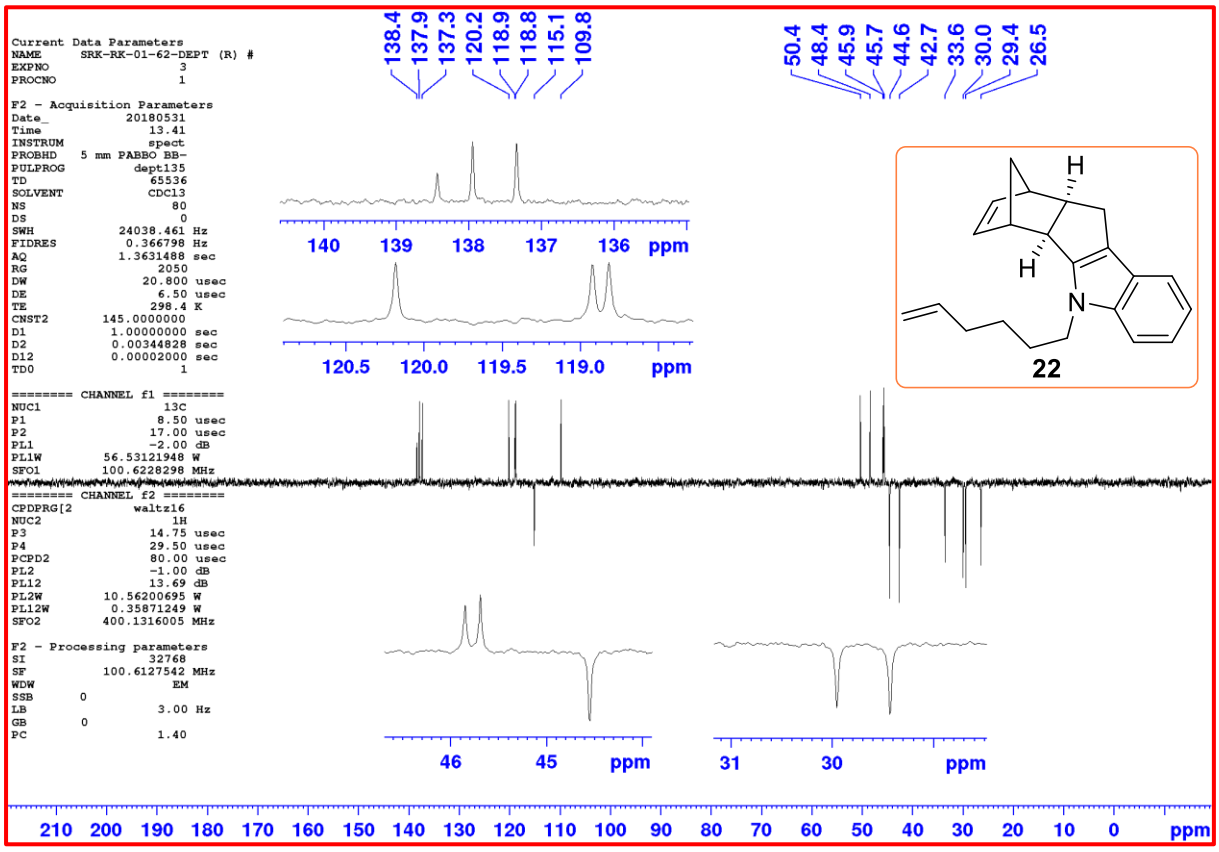
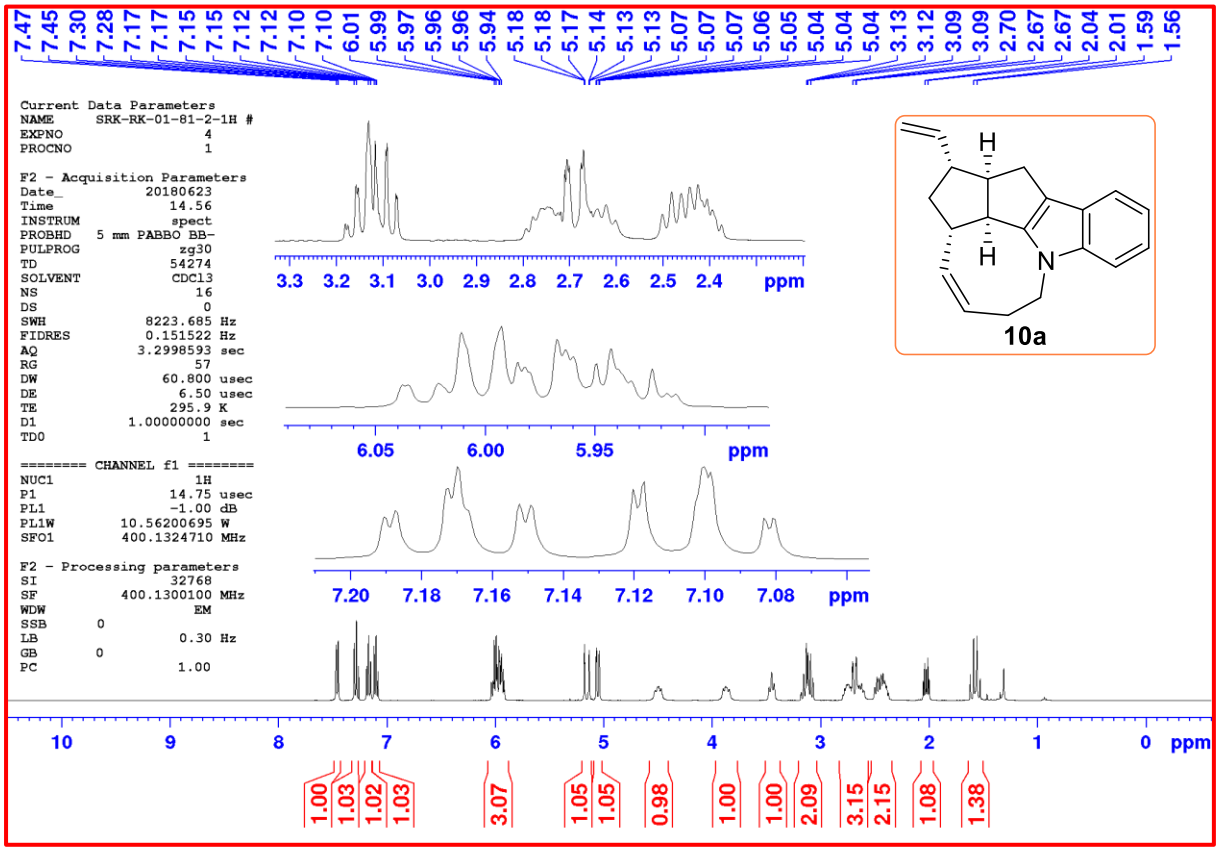
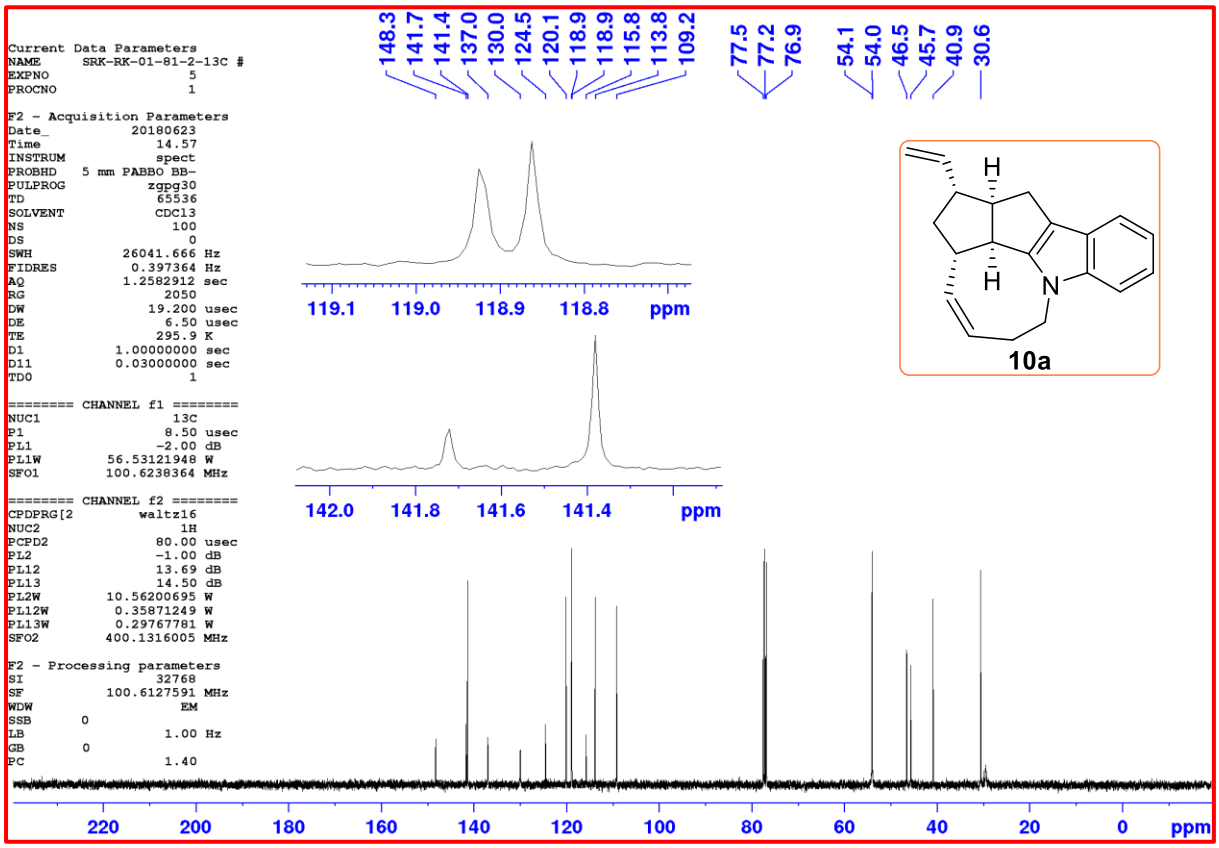


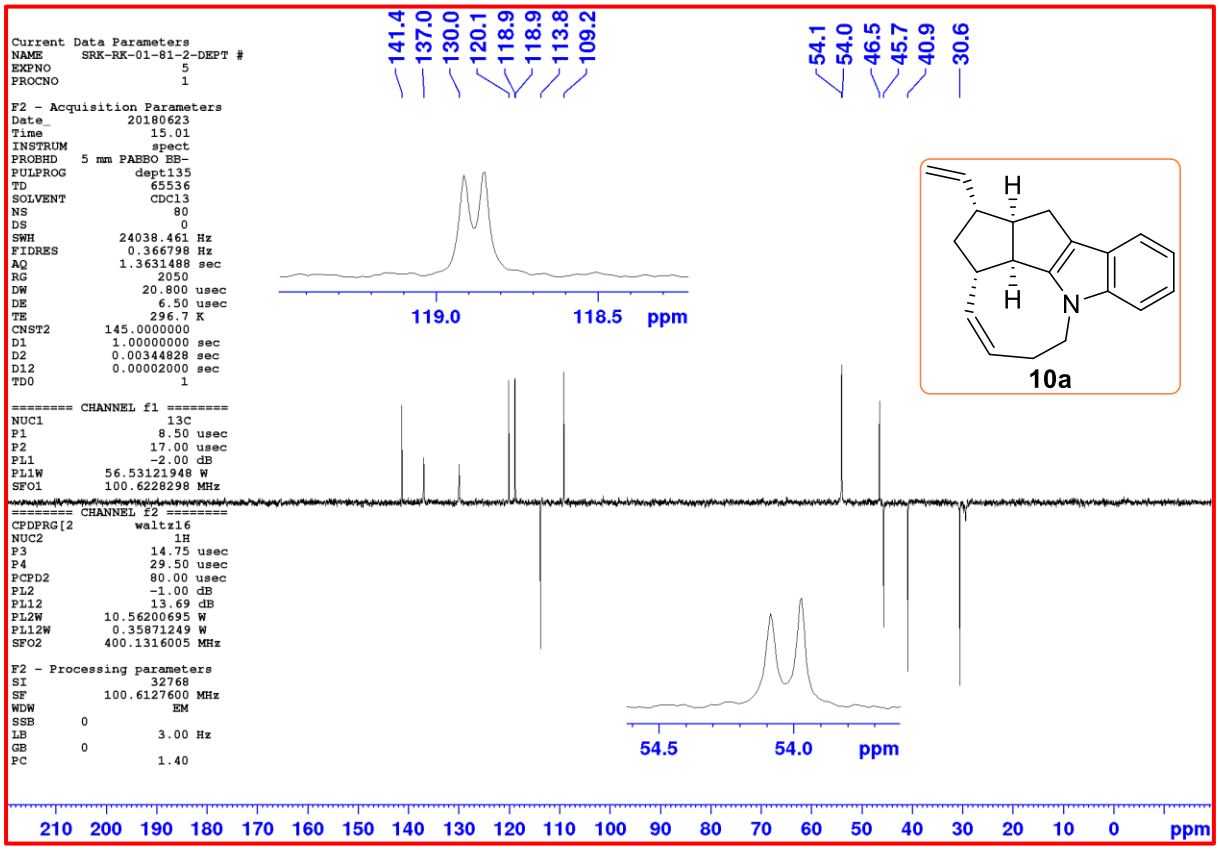
^1H NMR (400 MHz, $CDCl_3$) of **10a**



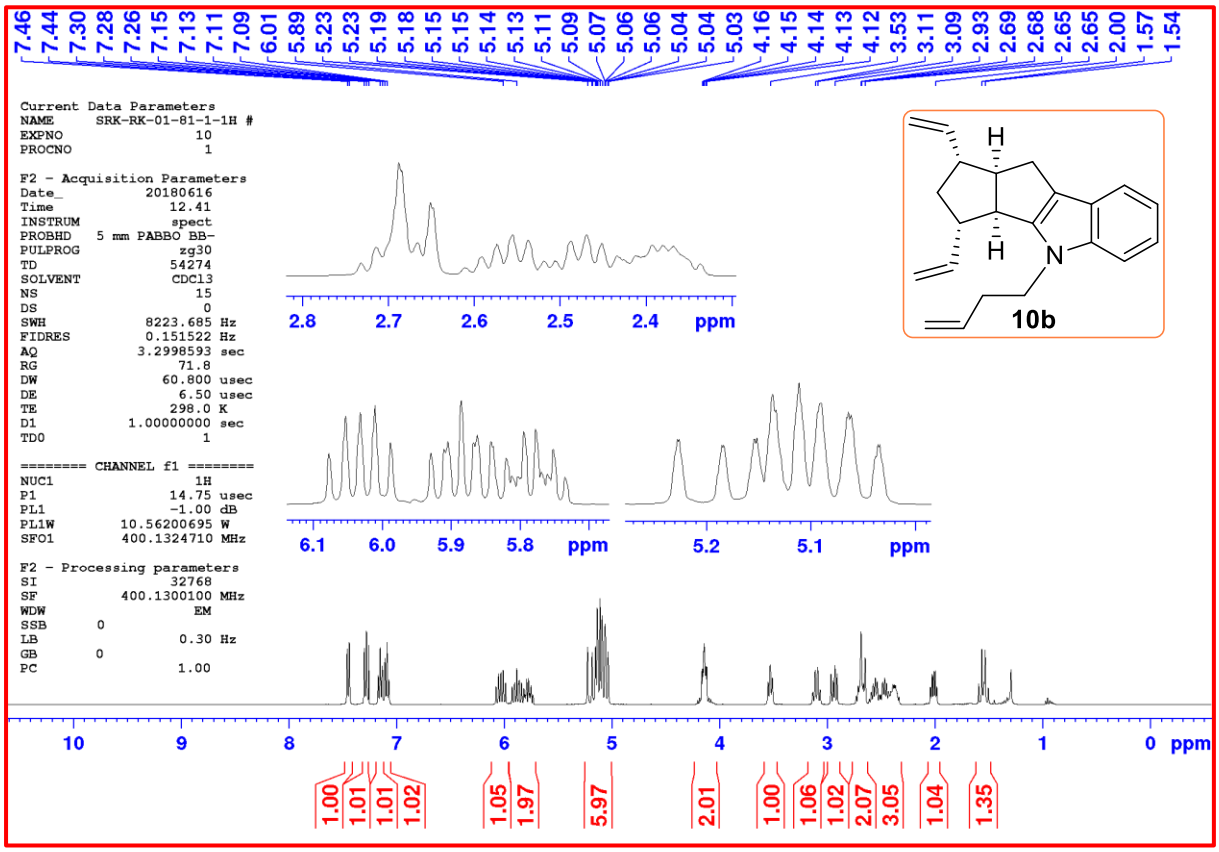
^{13}C NMR (100 MHz, $CDCl_3$) of **10a**



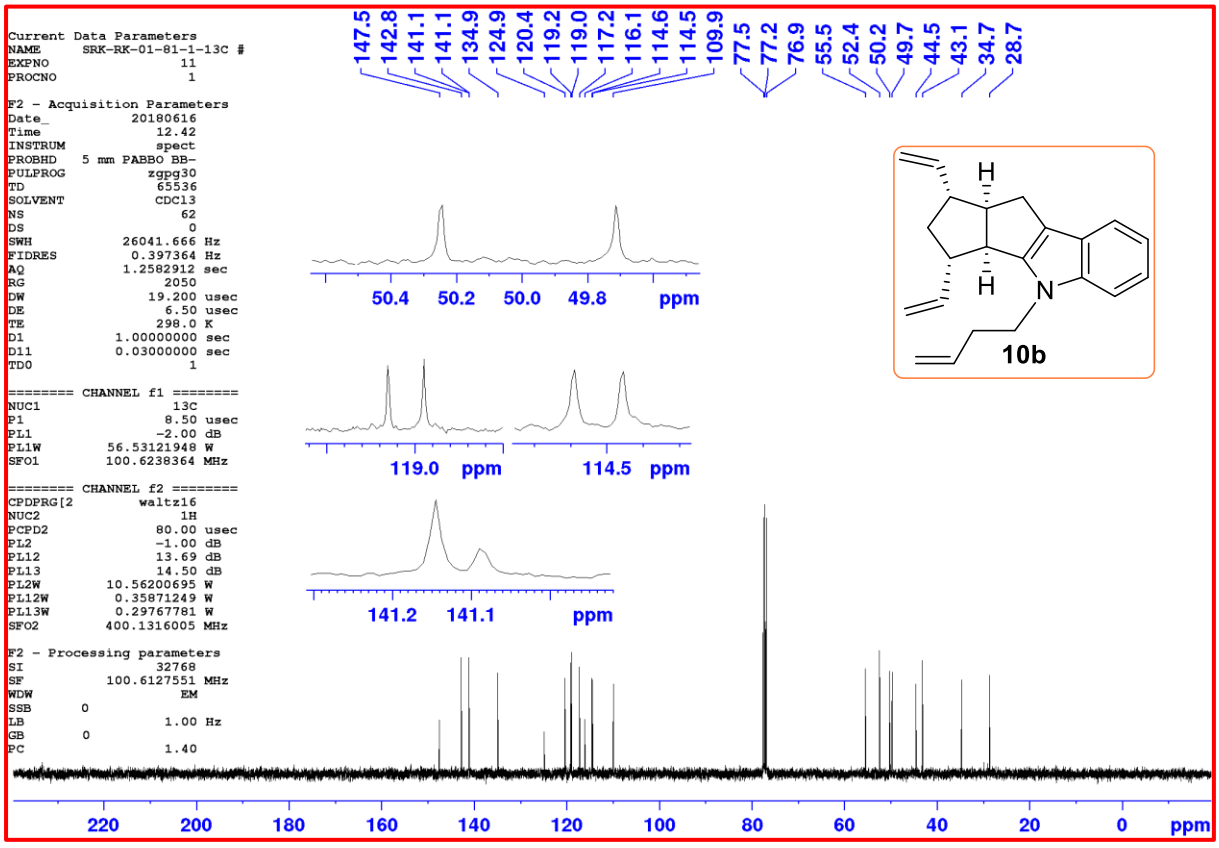
DEPT-135 (100 MHz, $CDCl_3$) of **10a**



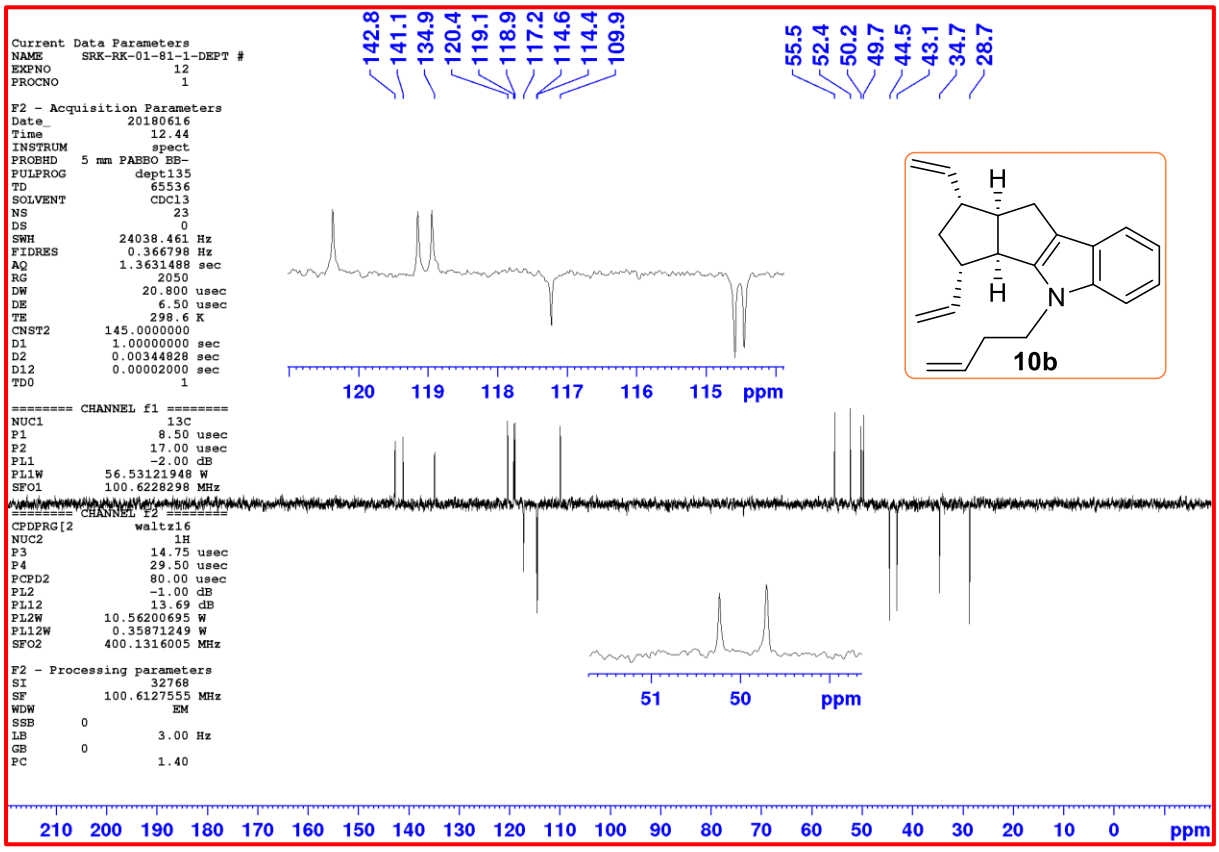
^{1}H NMR (400 MHz, $CDCl_3$) of **10b**



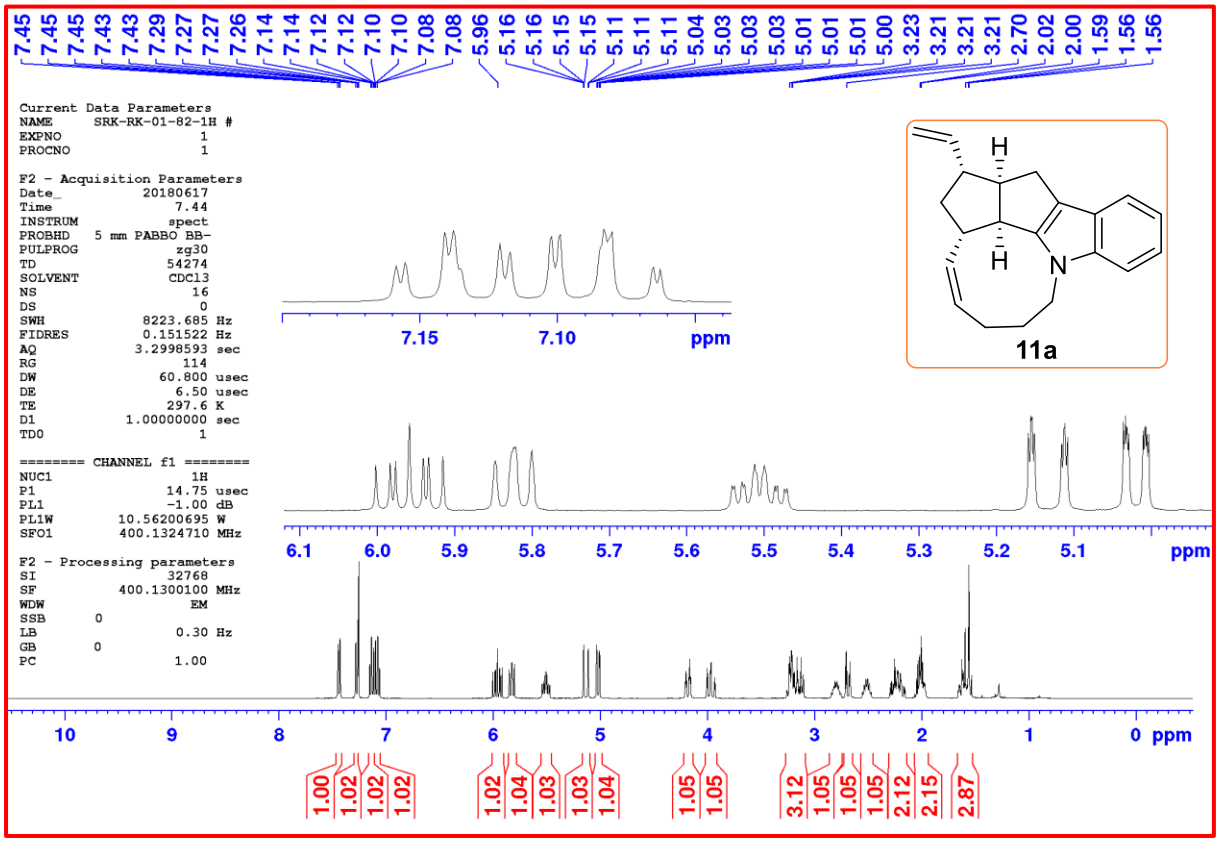
^{13}C NMR (100 MHz, $CDCl_3$) of **10b**



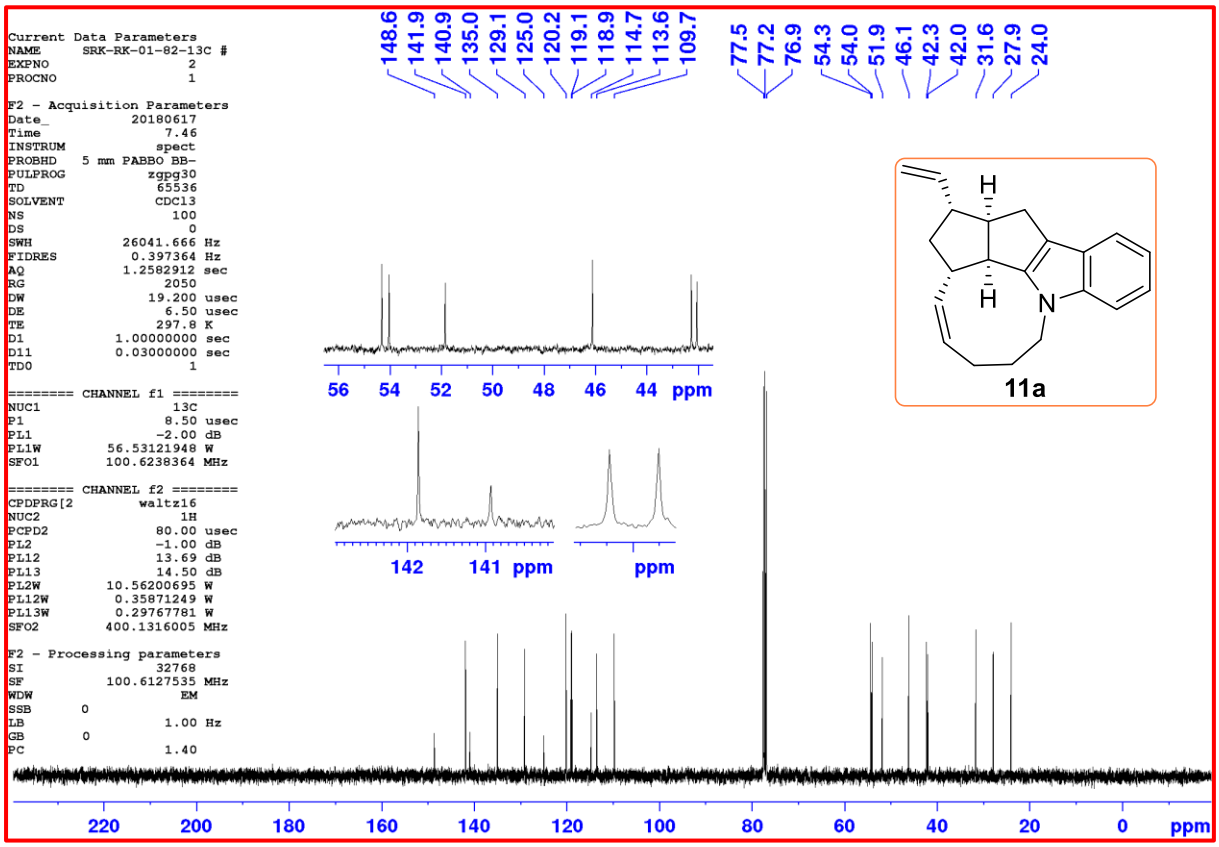
DEPT-135 (100 MHz, $CDCl_3$) of **10b**



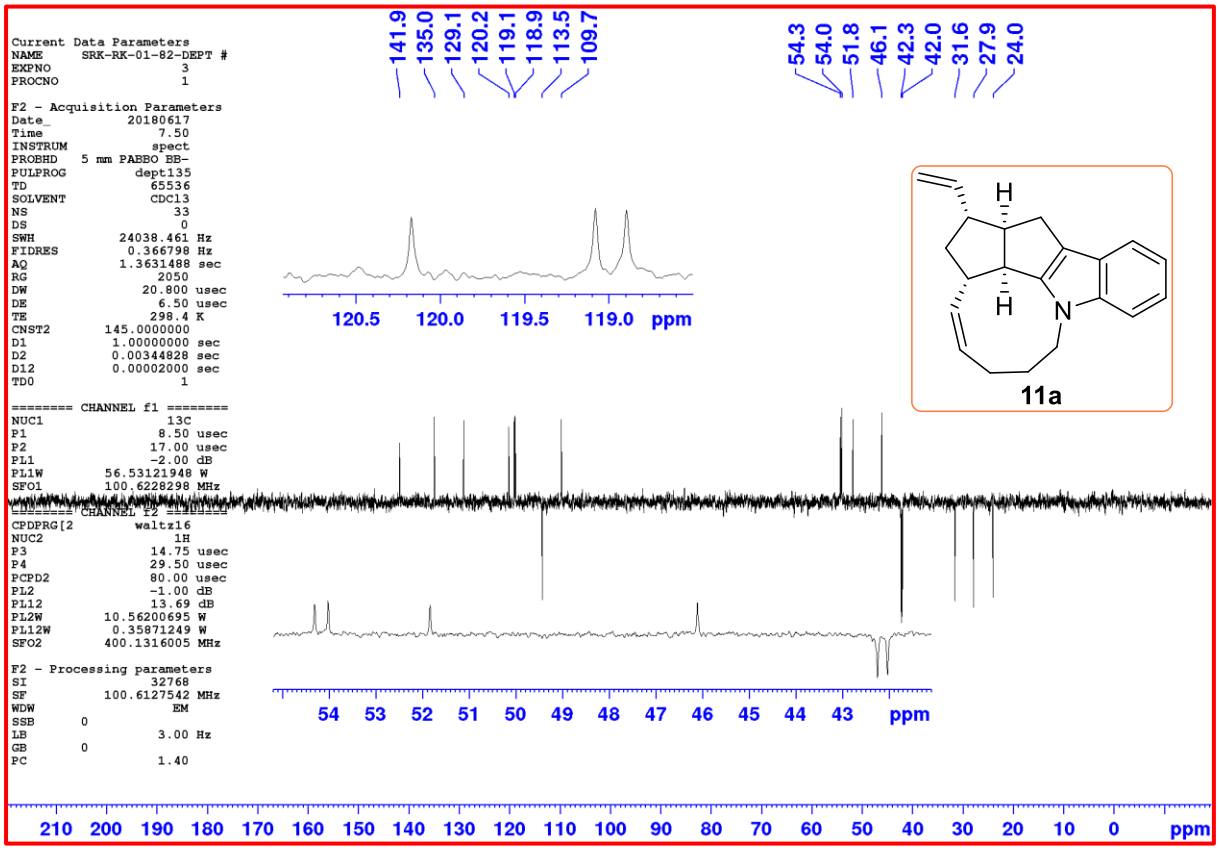
^1H NMR (400 MHz, $CDCl_3$) of **11a**



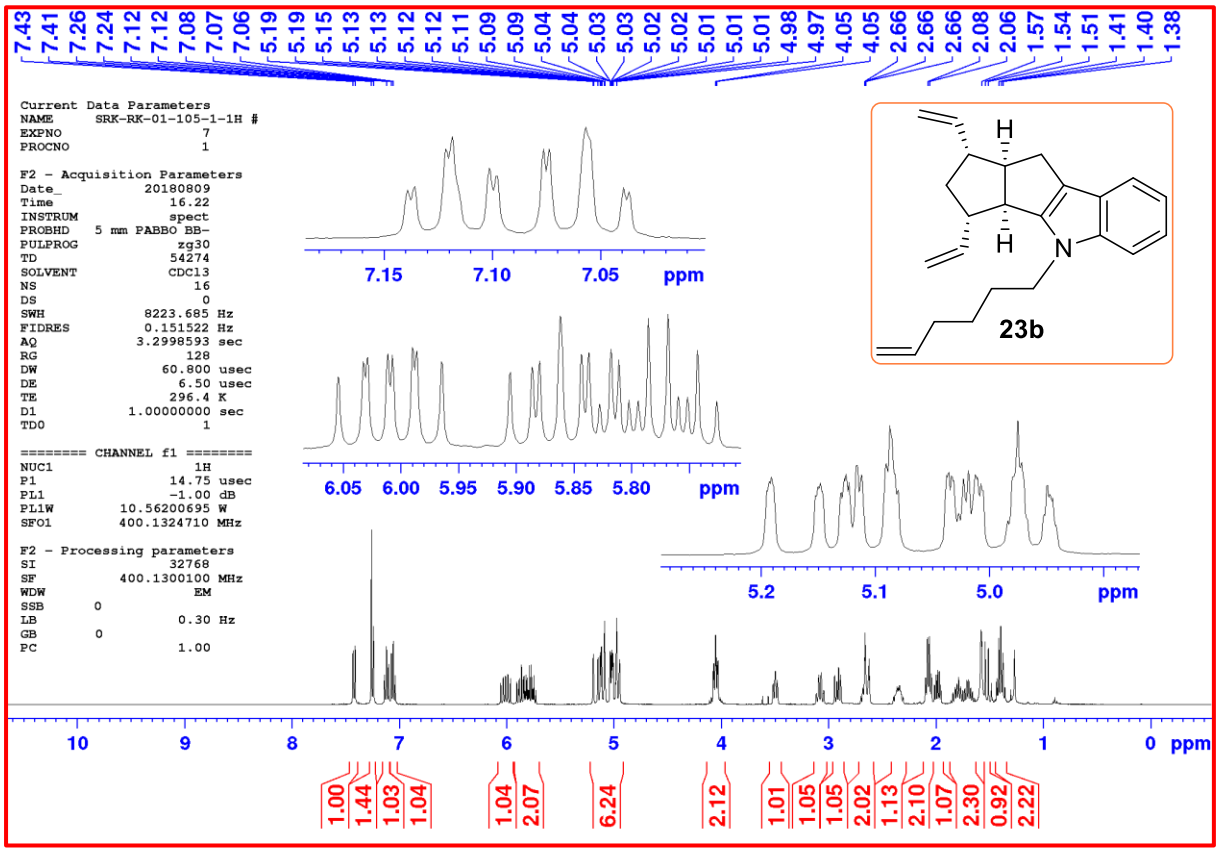
^{13}C NMR (100 MHz, $CDCl_3$) of **11a**



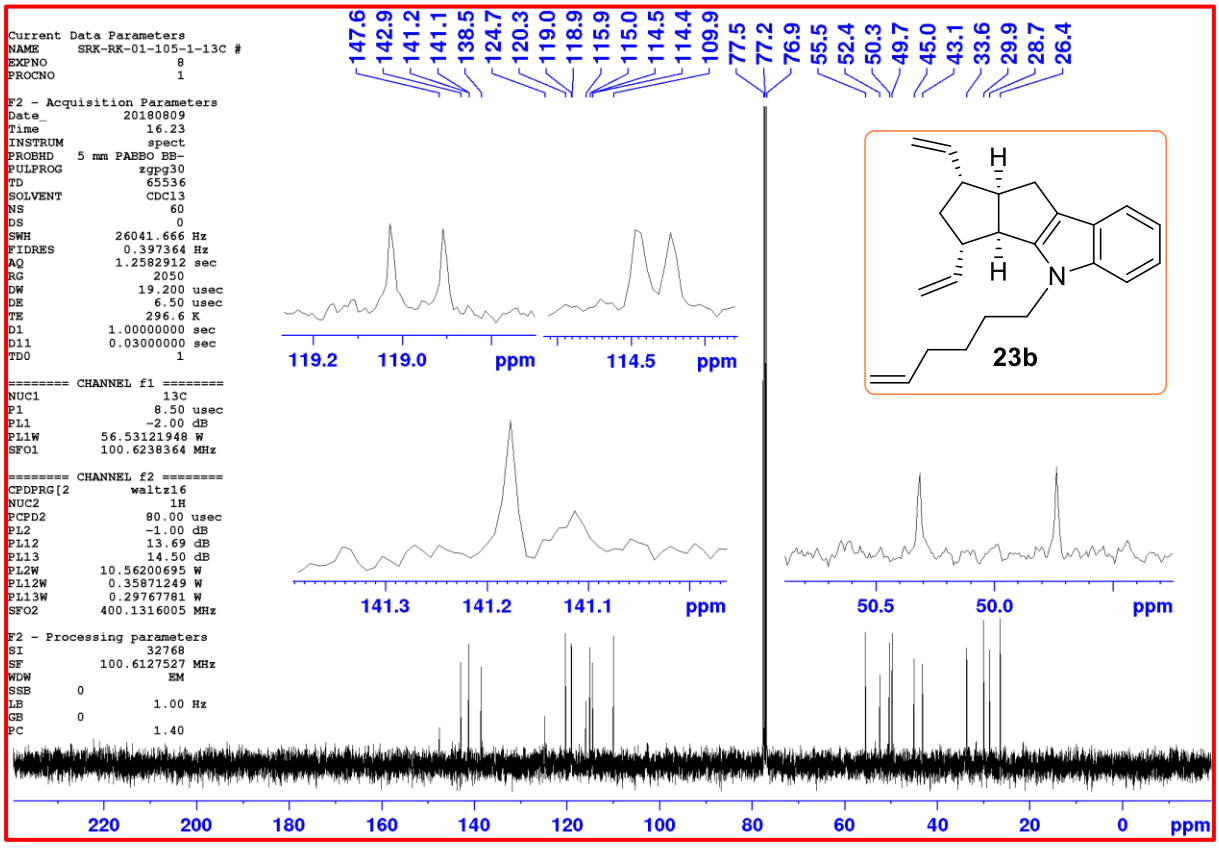
DEPT-135 (100 MHz, $CDCl_3$) of **11a**



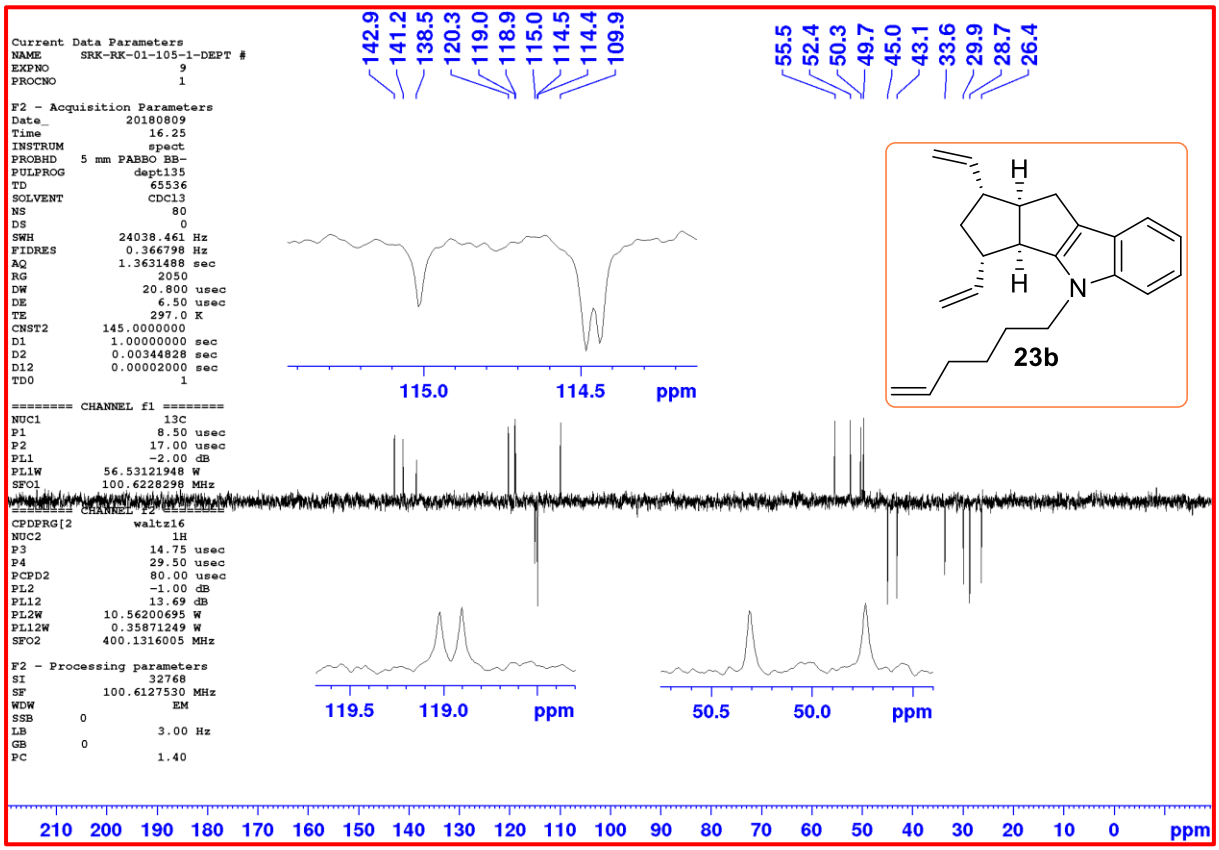
^{1}H NMR (400 MHz, $CDCl_3$) of **23b**



^{13}C NMR (100 MHz, $CDCl_3$) of **23b**



DEPT-135 (100 MHz, $CDCl_3$) of **23b**

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Current Data Parameters
NAME      SRK-RK-01-105-1-DEPT #
EXPNO                  9
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180809
Time              16.25
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG         dept135
TD                65536
SOLVENT           CDCl3
NS                   80
DS                    0
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG                 2050
DW               20.800 usec
DE                6.50 usec
TE               297.0 K
CNST2        145.0000000
D1             1.00000000 sec
D2             0.00344828 sec
D12            0.00002000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                  8.50 usec
P2                 17.00 usec
PL1                -2.00 dB
PL1W        56.53121948 W
SFO1       100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                 1H
P3                 14.75 usec
P4                 29.50 usec
PCPD2              80.00 usec
PL2                -1.00 dB
PL12               13.69 dB
PL2W        10.56200695 W
PL12W        0.35871249 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127530 MHz
WDW                  EM
SSB       0
LB                3.00 Hz
GB        0
PC                1.40
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