



Supporting Information

for

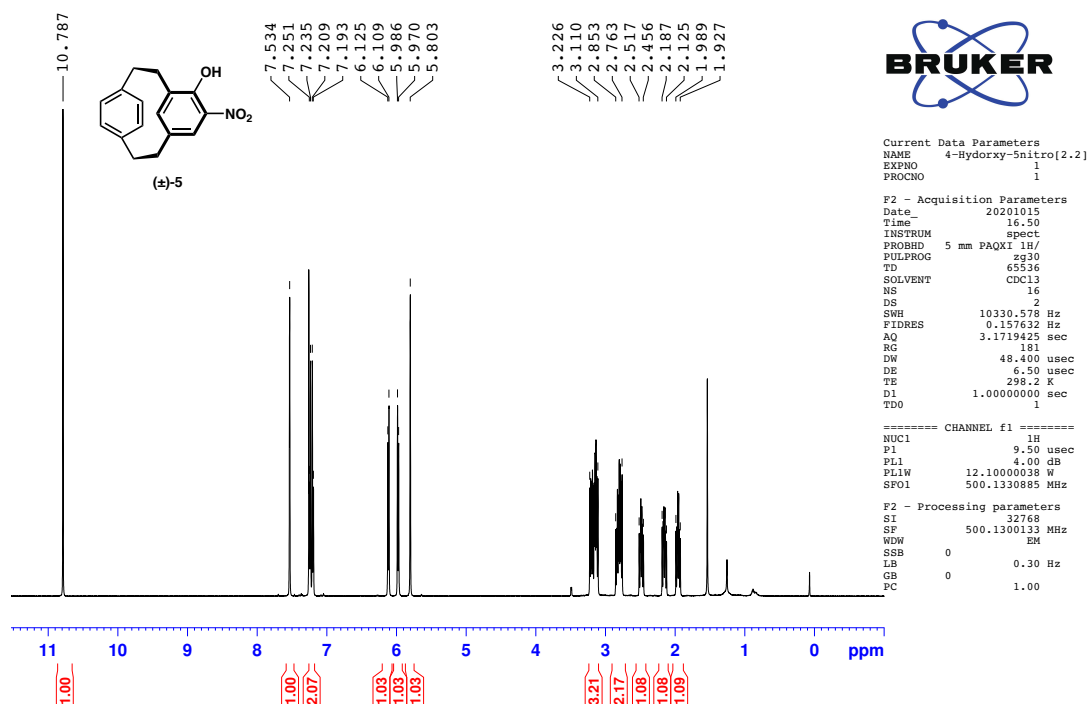
Breaking paracyclophane: the unexpected formation of non-symmetric disubstituted nitro[2.2]metaparacyclophanes

Suraj Patel, Tyson N. Dais, Paul G. Plieger and Gareth J. Rowlands

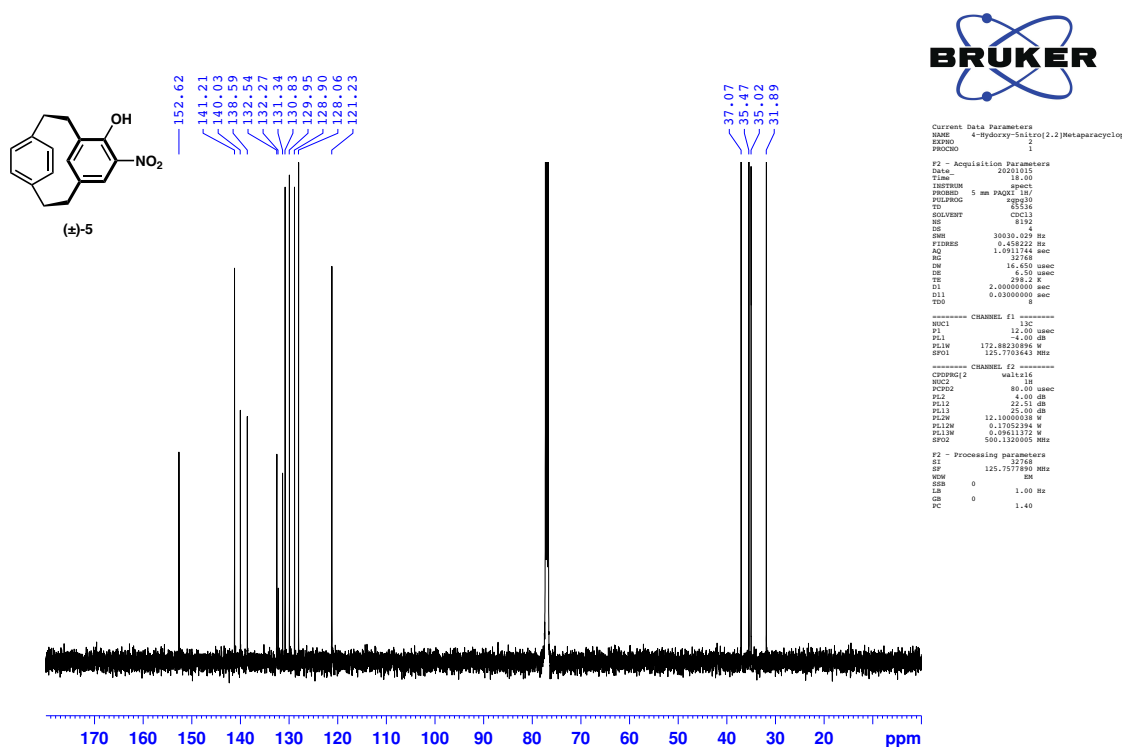
Beilstein J. Org. Chem. **2021**, *17*, 1518–1526. doi:10.3762/bjoc.17.109

Metaparacyclophane spectra

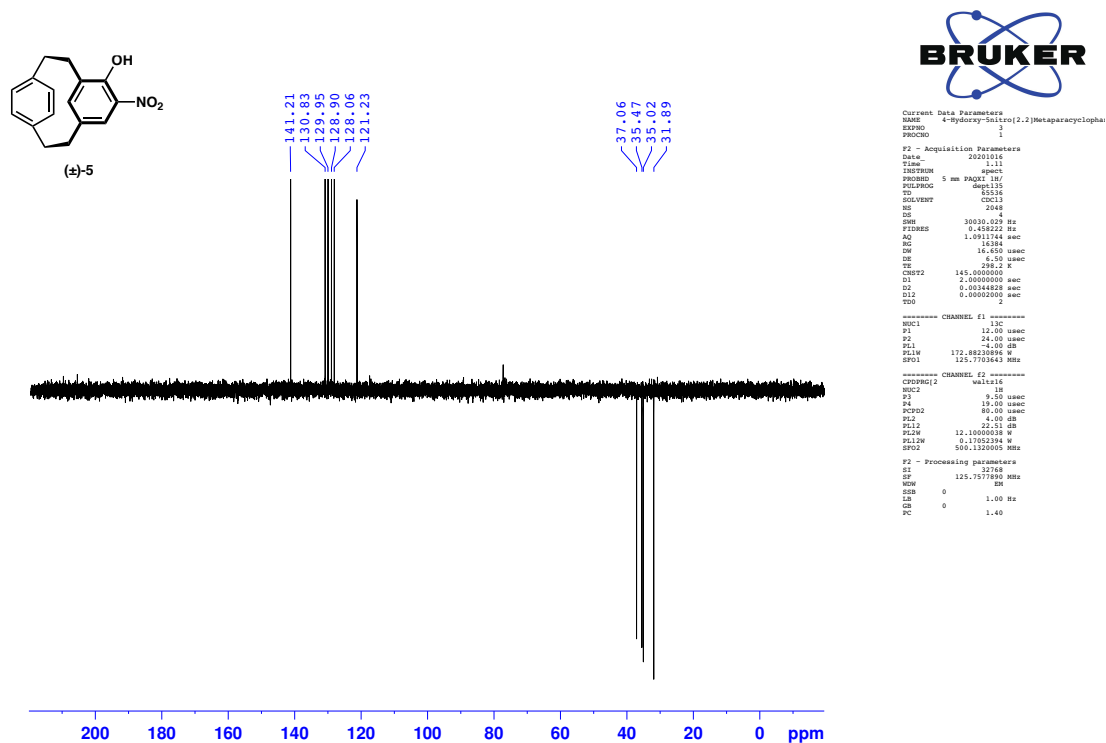
^1H NMR spectrum of ($\pm$)-**5** in CDCl_3



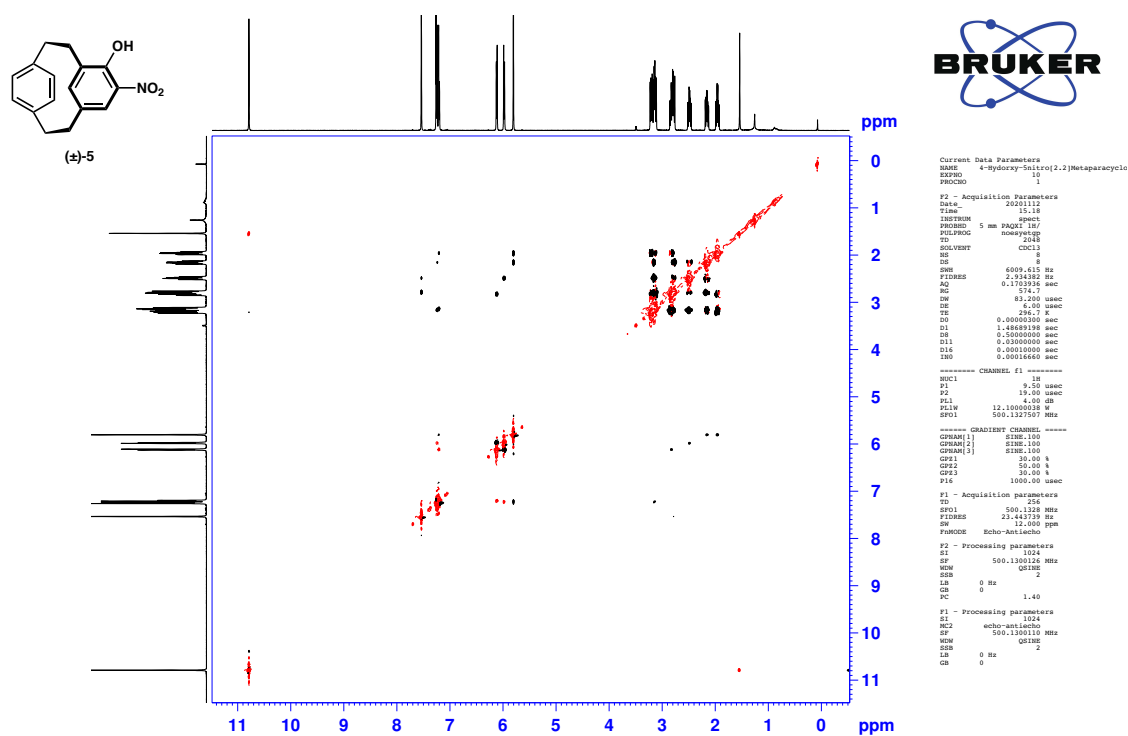
^{13}C NMR spectrum of ($\pm$)-**5** in CDCl_3



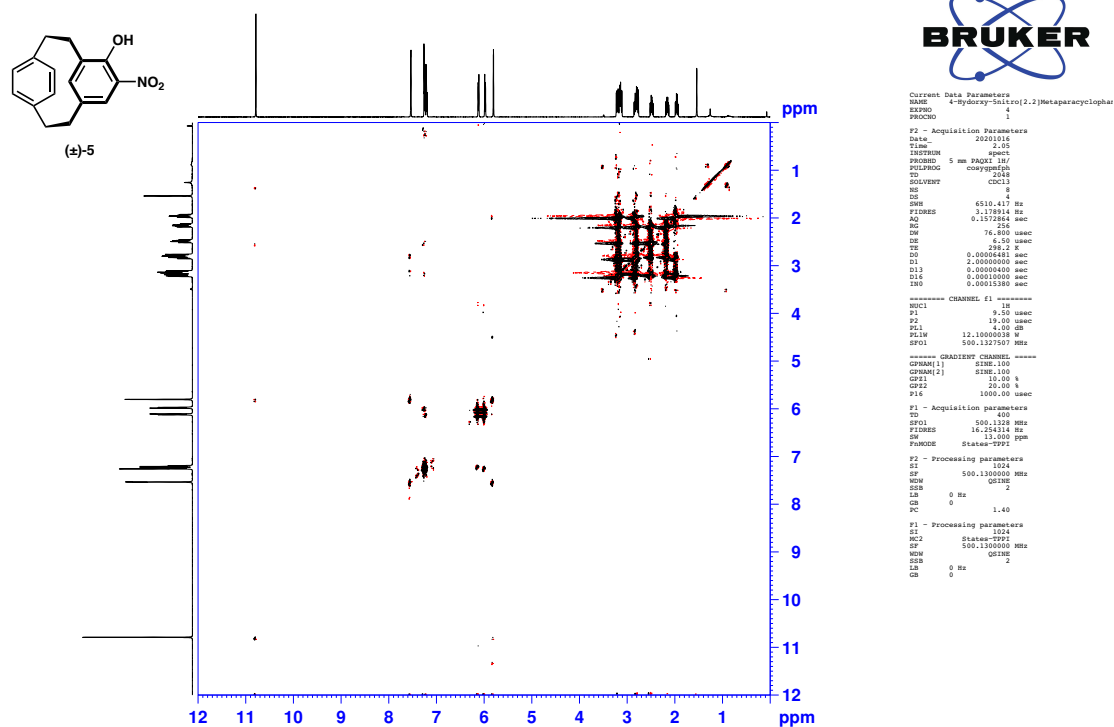
DEPT NMR spectrum of (±)-**5** in CDCl₃



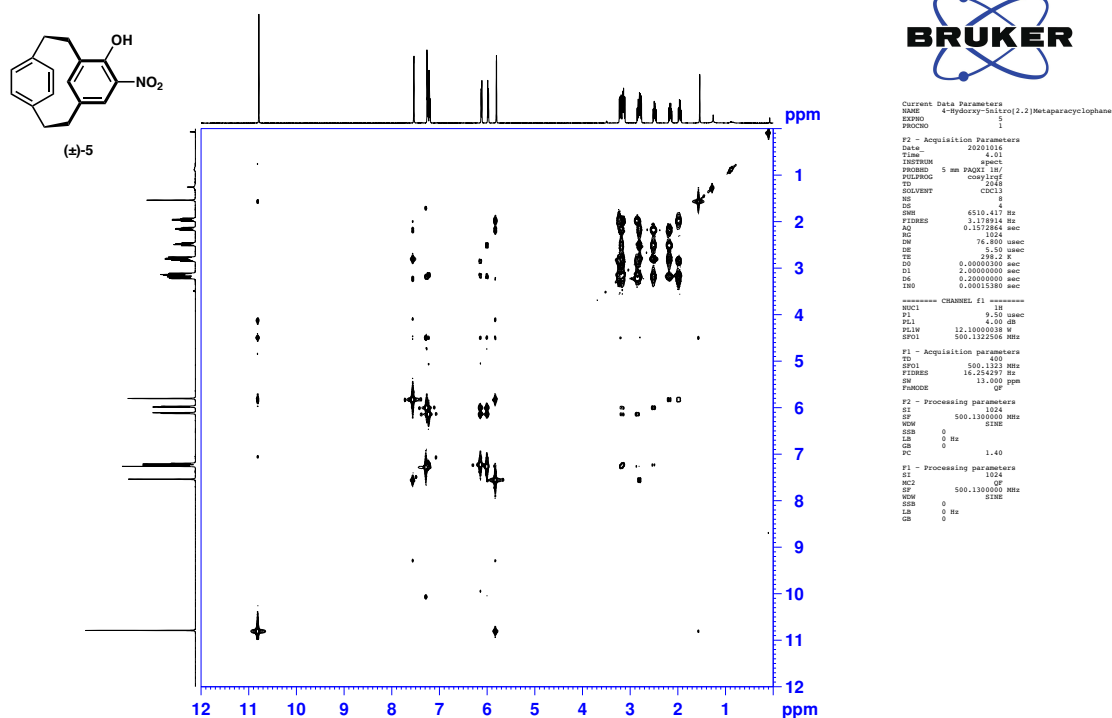
NOESY spectrum of (±)-**5** in CDCl₃



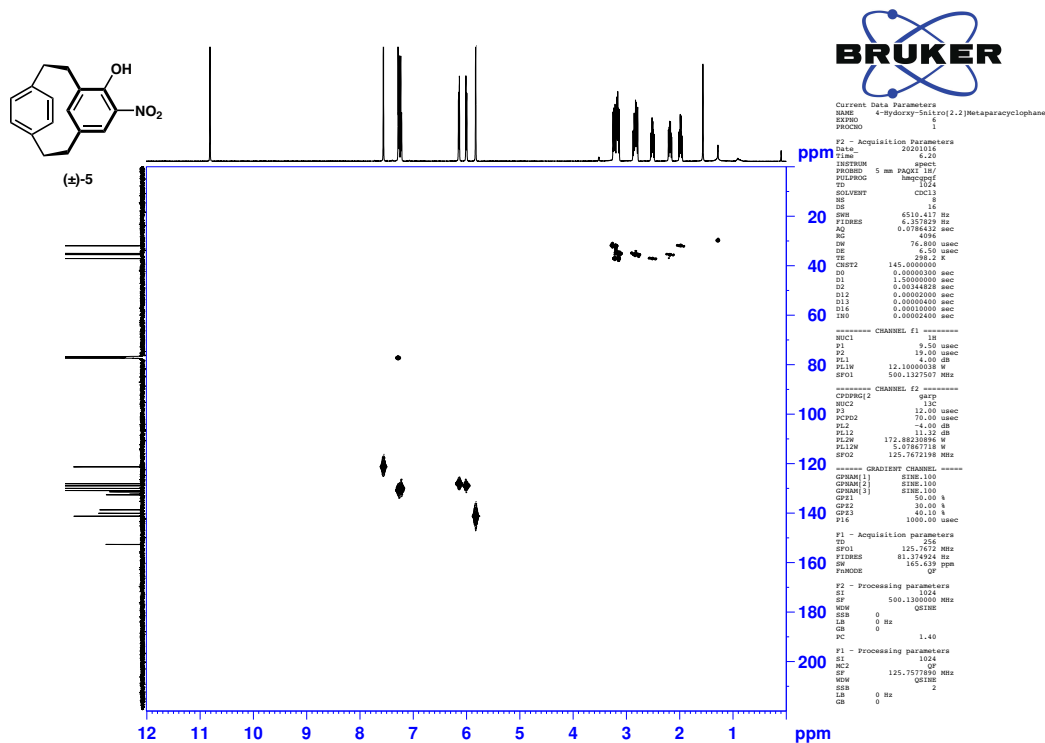
COSY NMR spectrum of ($\pm$)-**5** in CDCl₃



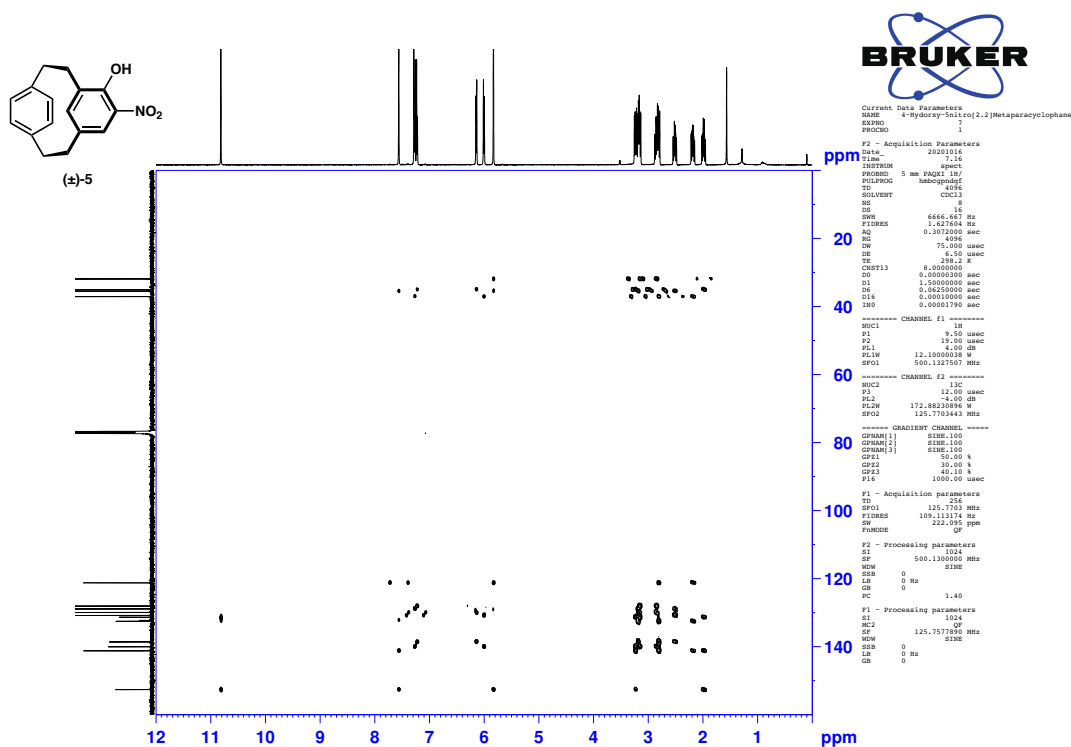
Long range COSY NMR spectrum of ($\pm$)-**5** in CDCl₃



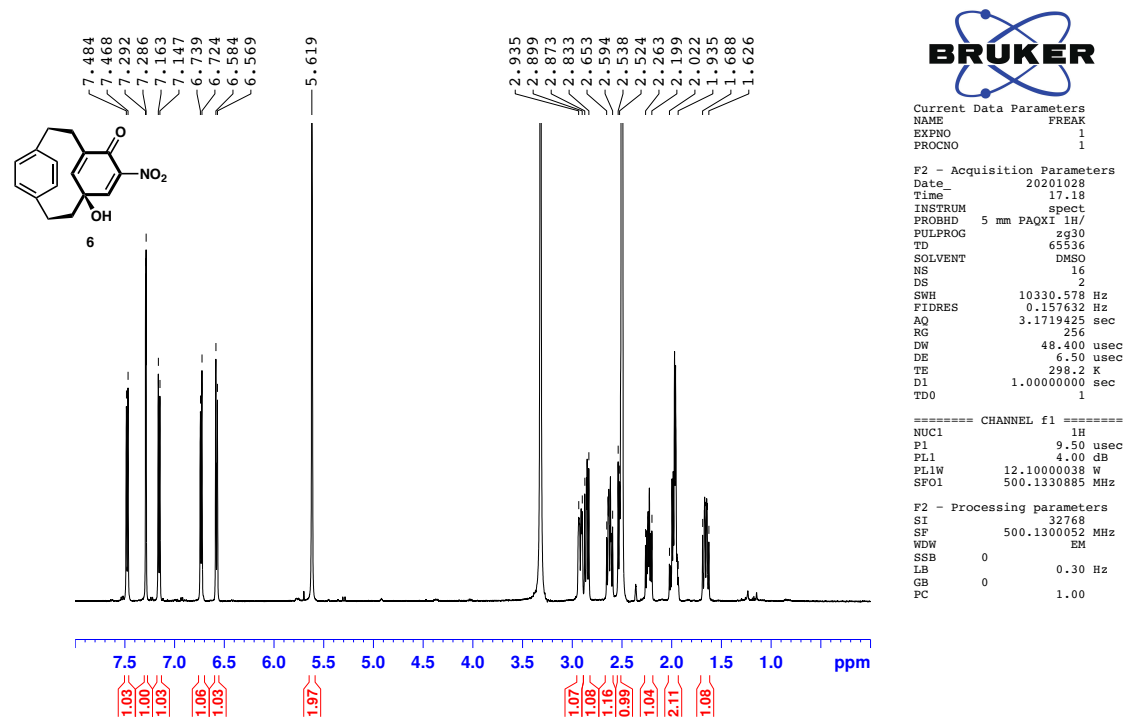
HMQC NMR spectrum of (±)-**5** in CDCl₃



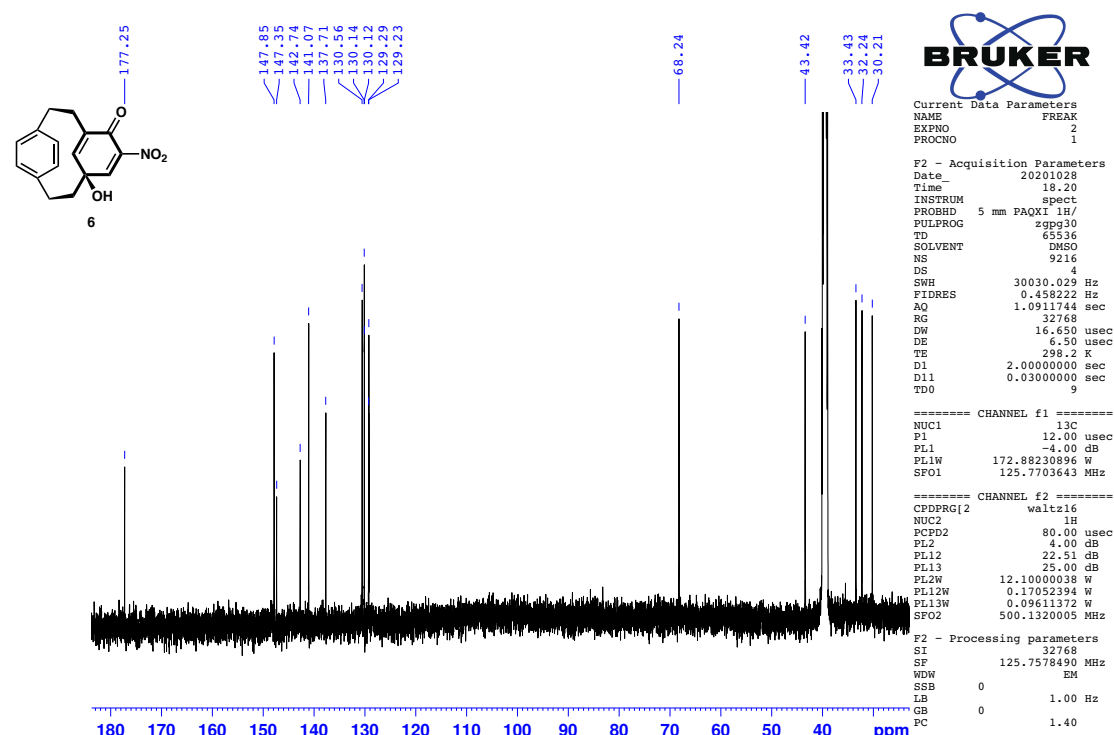
HMBC spectrum of (±)-**5** in CDCl₃



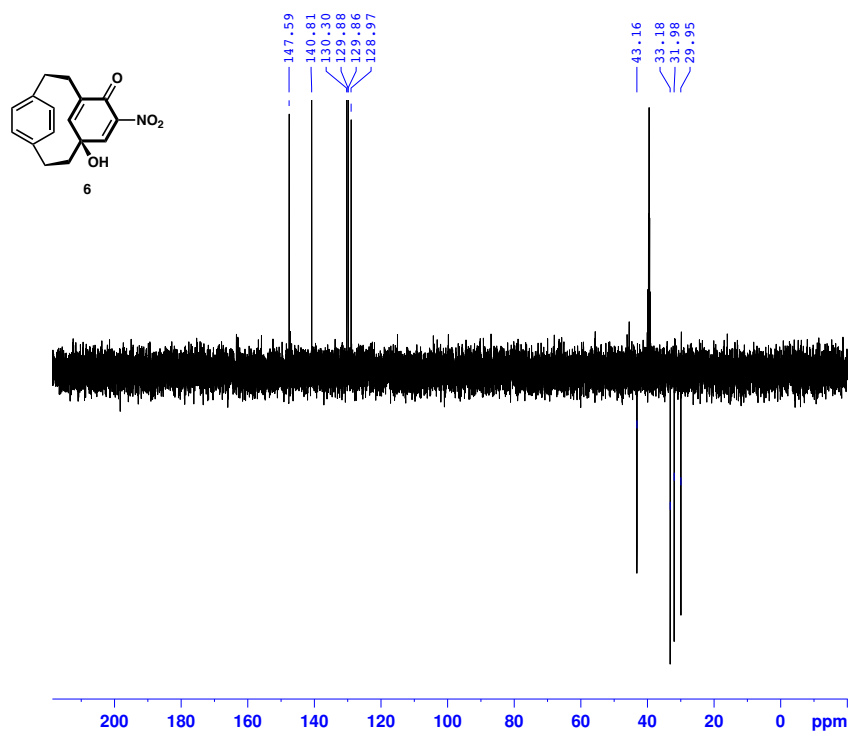
¹H NMR spectrum of **6** in DMSO-*d*₆



¹³C NMR spectrum of **6** in DMSO-*d*₆



DEPT NMR spectrum of **6** in DMSO-*d*₆



Current Data Parameters
NAME FREAK
EXPNO 3
PROCNO 1

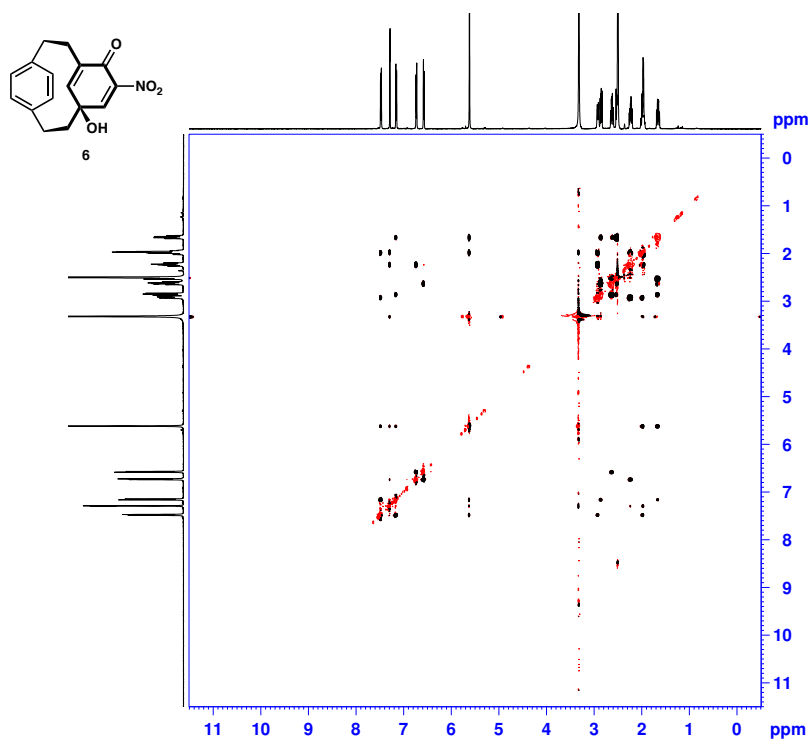
F2 - Acquisition Parameters
Date_ 20201029
Time 2.24
INSTRUM spect
PROBHD 5 mm PAQXI 1H/
PULPROG dept135
TD 65536
SOLVENT DMSO
NS 1024
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0911744 sec
RG 16384
DW 16.650 usec
DE 6.50 usec
TE 298.2 K
CHST2 145.0000000
D1 2.00000000 sec
D2 0.00344828 sec
D12 0.00002000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
P2 24.00 usec
PL1 4.00 dB
PL1W 172.88230896 W
SFO1 125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
P3 9.50 usec
P4 19.00 usec
PCPD2 80.00 usec
PL2 4.00 dB
PL12 22.51 dB
PL2W 12.10000038 W
PL12W 0.17052394 W
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7578814 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

NOESY spectrum of **6** in DMSO-*d*₆



Current Data Parameters
NAME FREAK
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20201029
Time 2.25
INSTRUM spect
PROBHD 5 mm PAQXI 1H/
PULPROG noesyetpg
TD 2048
SOLVENT DMSO
NS 16
DS 8
SWH 6009.615 Hz
FIDRES 2.534382 Hz
AQ 0.1703936 sec
RG 574.7
DW 83.200 usec
DE 6.00 usec
TE 298.2 K
DO 0.00000300 sec
D1 1.4868198 sec
D8 0.50000000 sec
D11 0.03000000 sec
D16 0.00010000 sec
IN0 0.00016660 sec

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P1 9.50 usec
P2 19.00 usec
PL1 4.00 dB
PL1W 12.10000038 W
SFO1 500.1327507 MHz

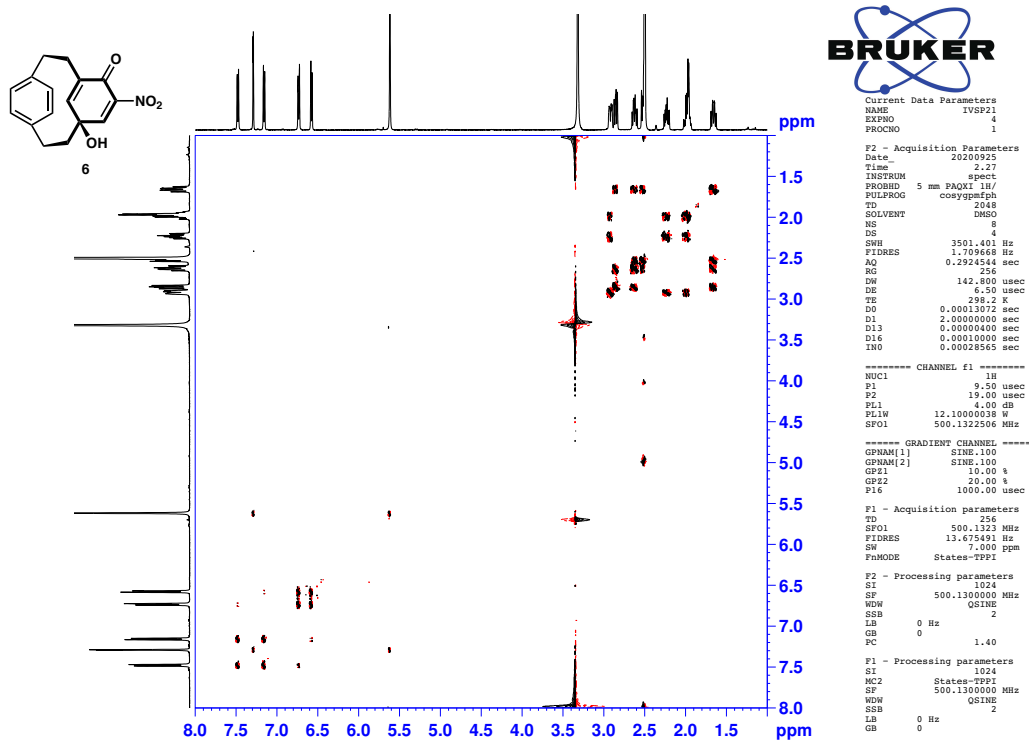
===== GRADIENT CHANNEL =====
GPRAM[1] SINE.100
GPRAM[2] SINE.100
GPRAM[3] SINE.100
GP21 30.00 %
GP22 50.00 %
GP23 30.00 %
P16 1000.00 usec

F1 - Acquisition parameters
TD 256
SFO1 500.1328 MHz
FIDRES 23.443739 Hz
SN 12.000 ppm
P16MODE Echo-Antiecho

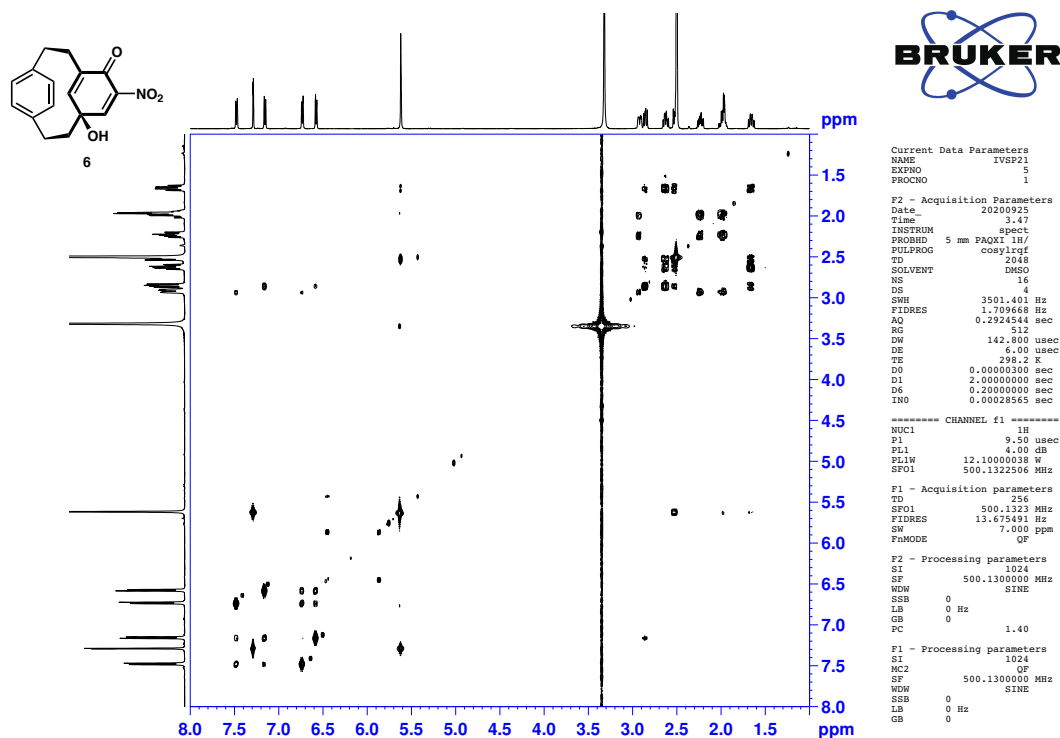
F2 - Processing parameters
SI 1024
SF 500.1300000 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 echo-antiecho
SF 500.1300000 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0

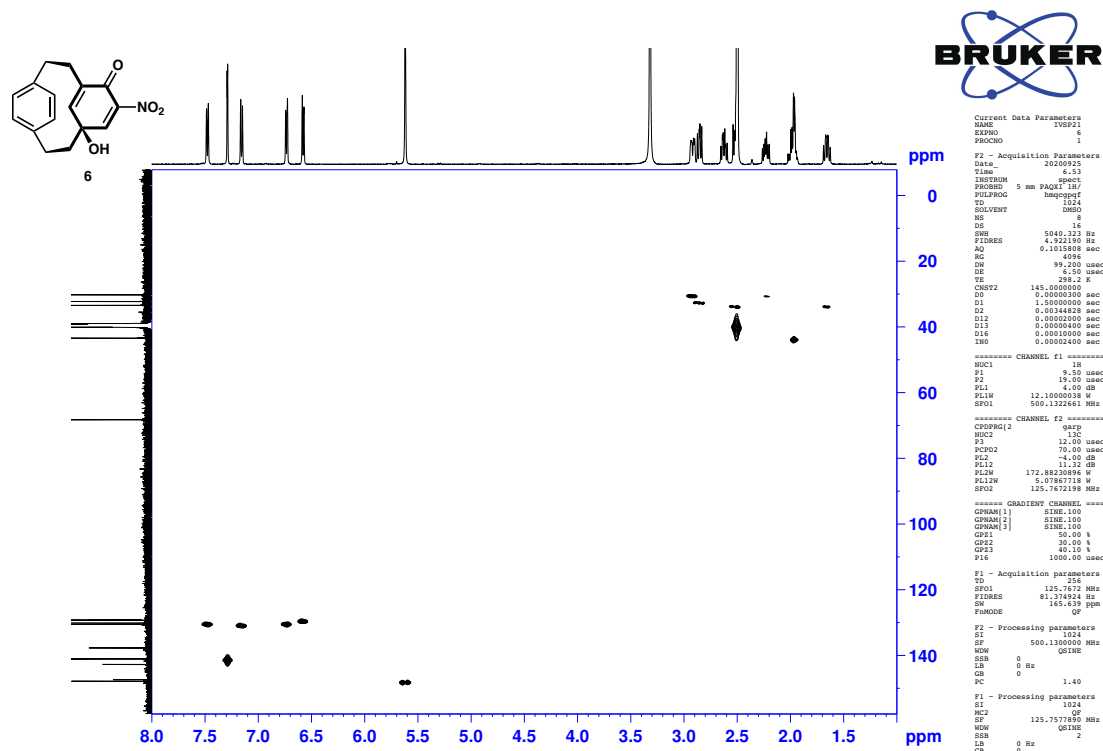
COSY NMR spectrum of **6** in DMSO-*d*₆



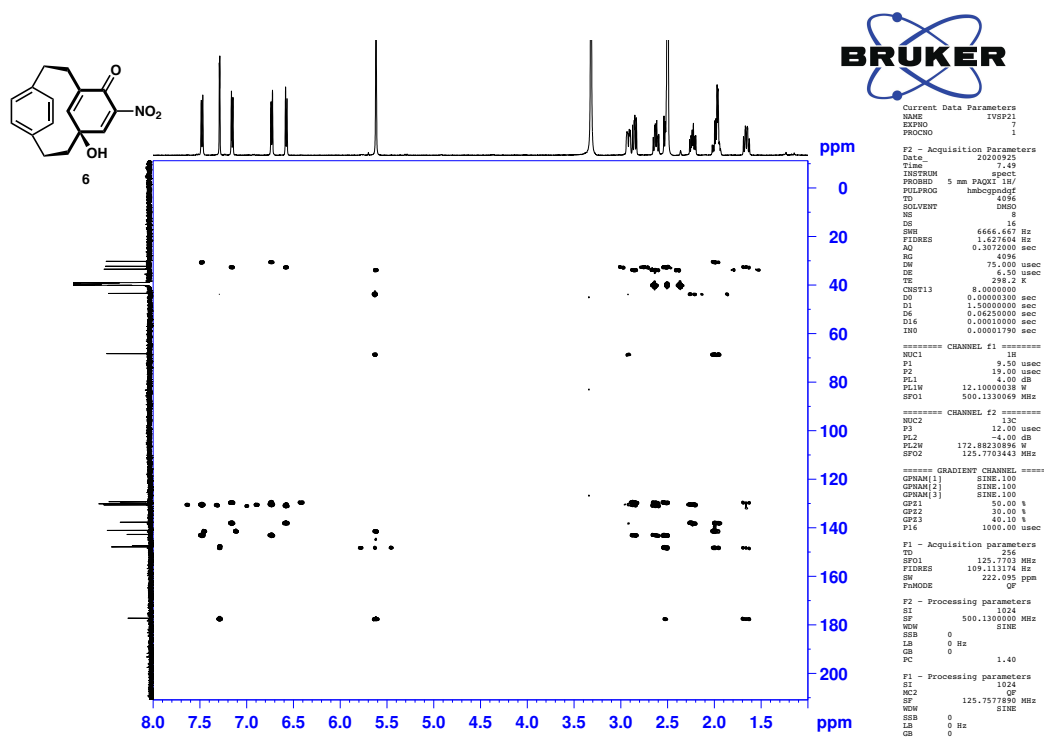
Long range COSY NMR spectrum of **6** in DMSO-*d*₆



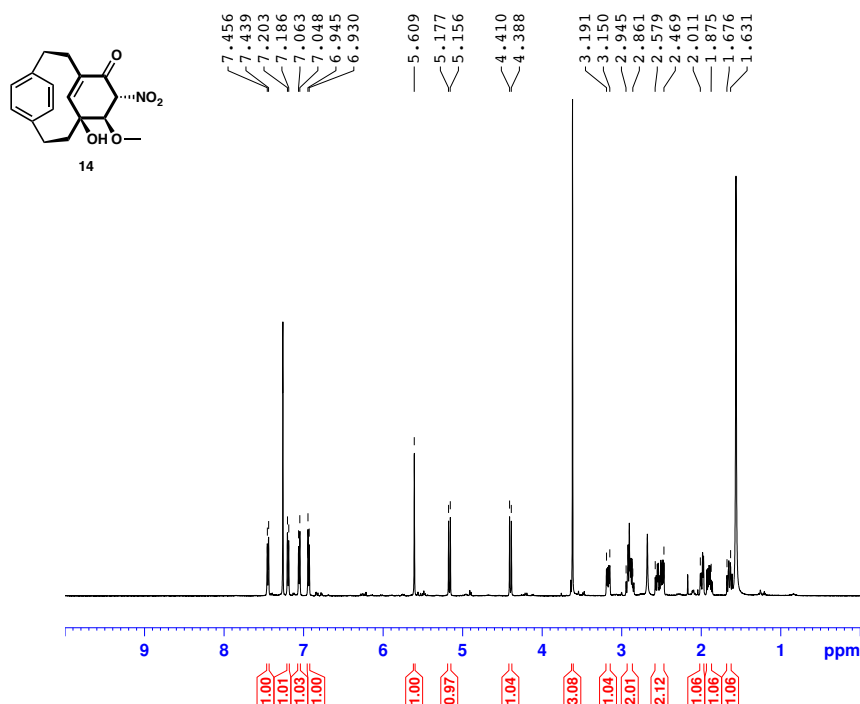
HMQC NMR spectrum of **6** in DMSO-*d*₆



HMBC NMR spectrum of **6** in DMSO-*d*₆



¹H NMR spectrum of **14** in CDCl₃



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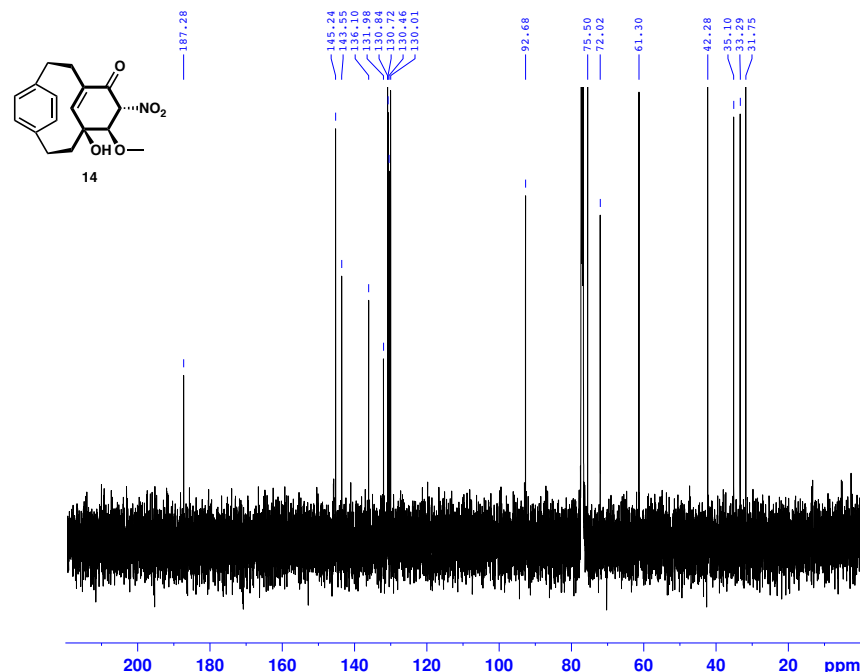
Current Data Parameters
NAME IVSP106
EXPNO 1
PROCNO 1

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Time 16.45
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PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719425 sec
RG 456.1
DW 48.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 9.50 usec
PL1 4.00 dB
PL1W 12.10000038 W
SFO1 500.1330885 MHz

F2 - Processing parameters
SI 32768
SF 500.1300138 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C NMR spectrum of **14** in CDCl₃



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Current Data Parameters
NAME IVSP106
EXPNO 2
PROCNO 1

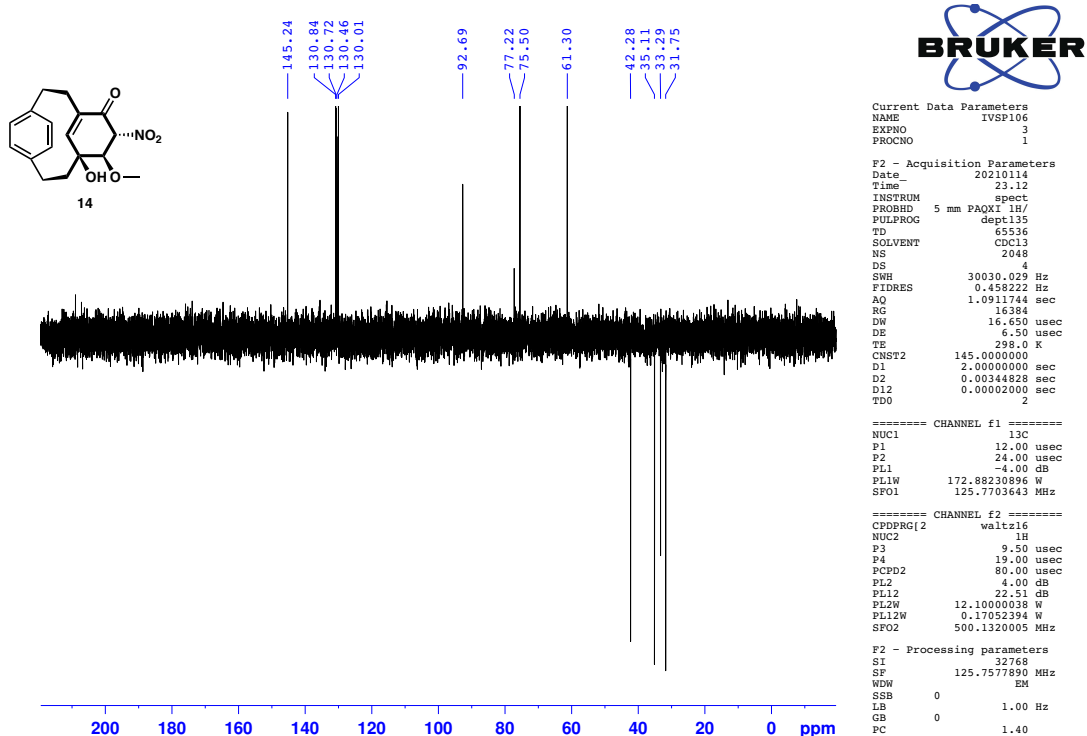
F2 - Acquisition Parameters
Date 20210114
Time 17.49
INSTRUM spect
PROBHD 5 mm PAQXI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 6144
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0911744 sec
RG 32768
DW 16.650 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 6

===== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 -4.00 dB
PL1W 172.88230896 W
SFO1 125.7703643 MHz

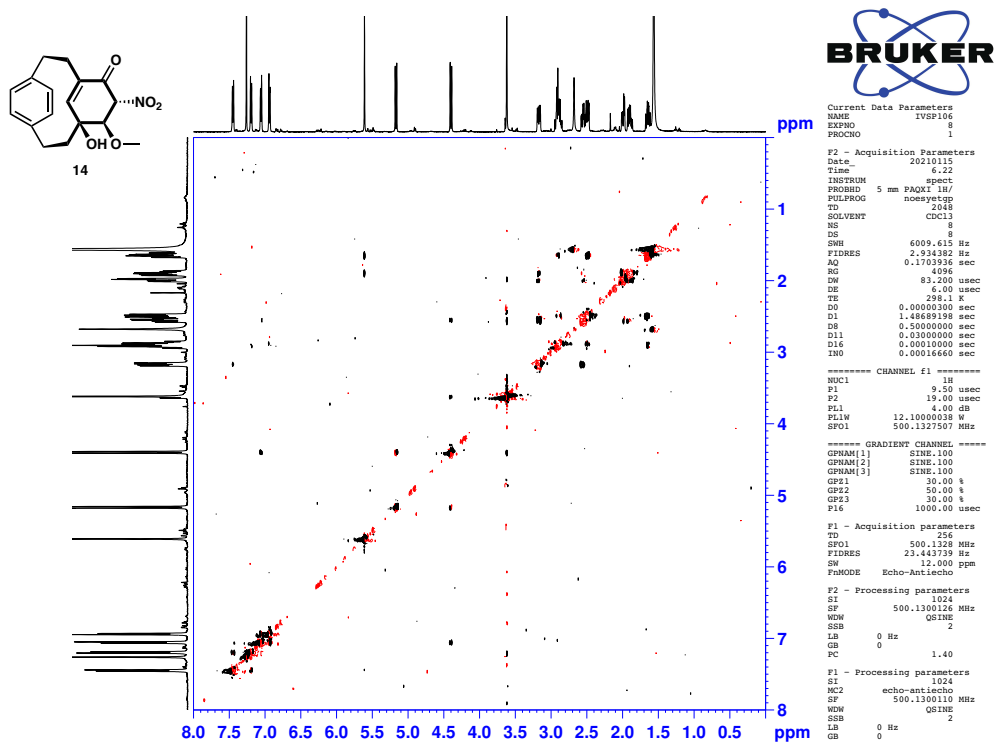
===== CHANNEL f2 =====
CPDPRG[2] waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 22.51 dB
PL13 25.00 dB
PL2W 12.10000038 W
PL12W 0.17052394 W
PL13W 0.09611372 W
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

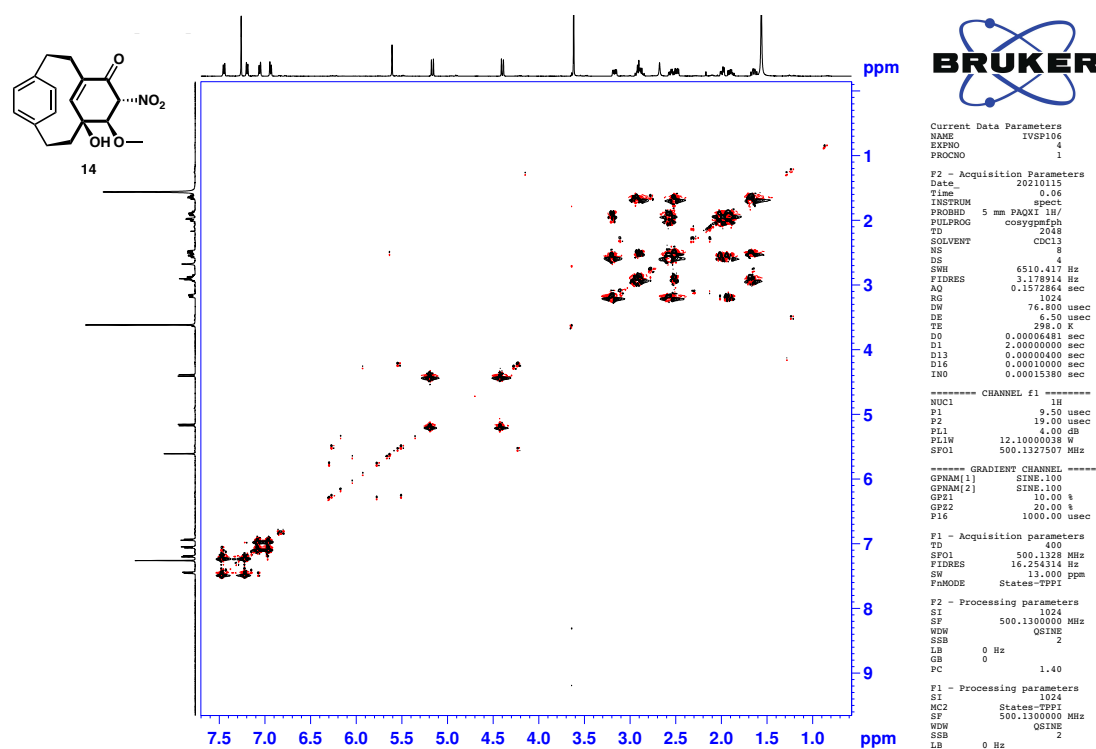
DEPT spectrum of **14** in CDCl₃



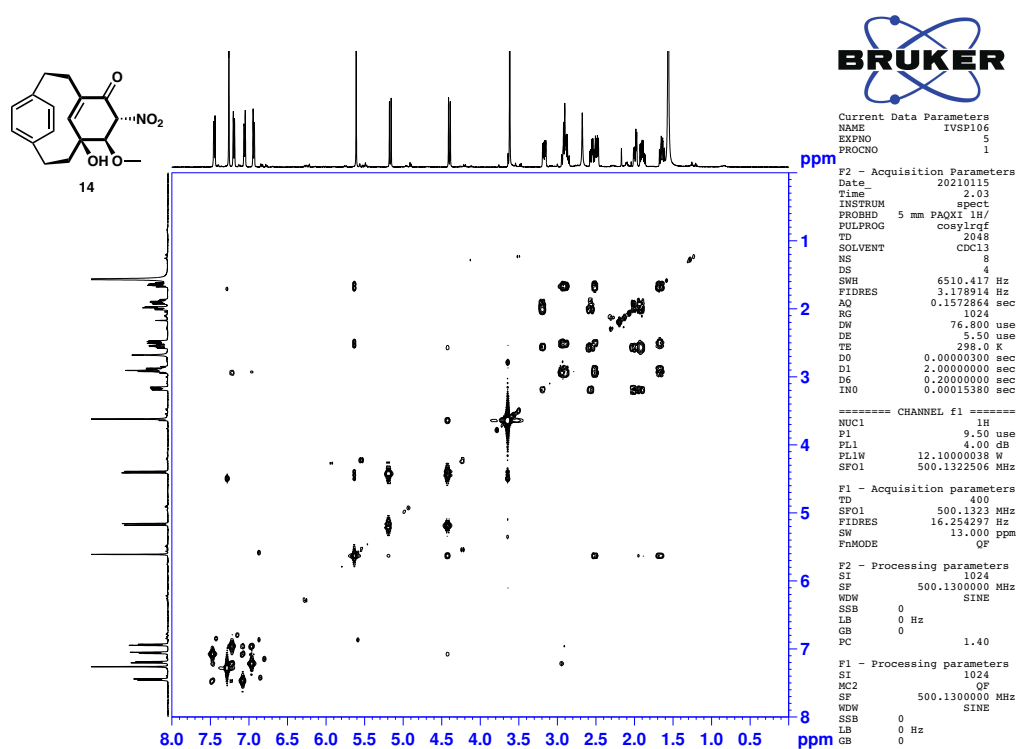
NOESY spectrum of **14** in CDCl₃



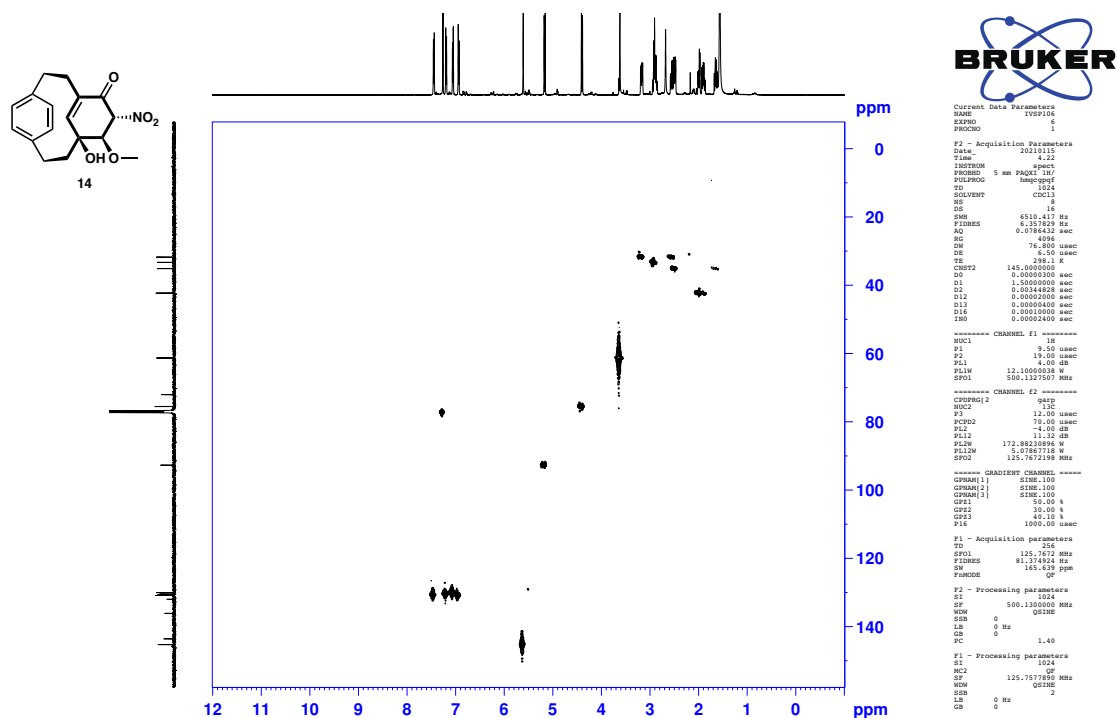
COSY spectrum of **14** in CDCl₃



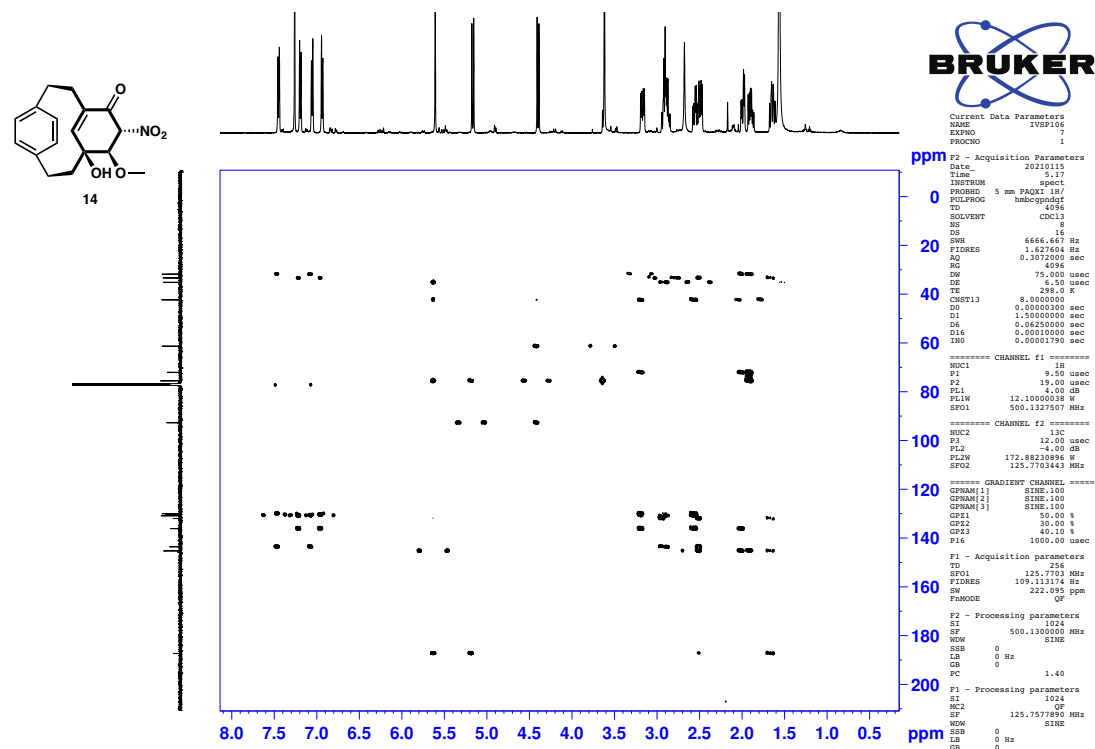
Long range COSY spectrum of **14** in CDCl₃



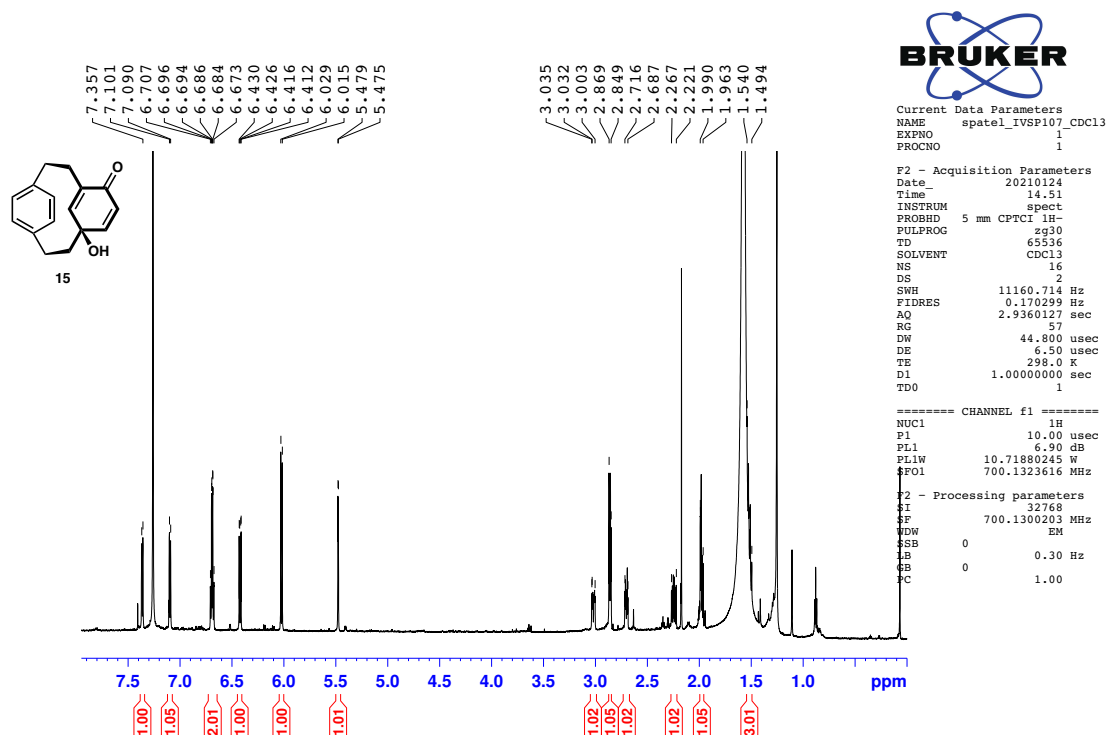
HMQC spectrum of **14** in CDCl₃



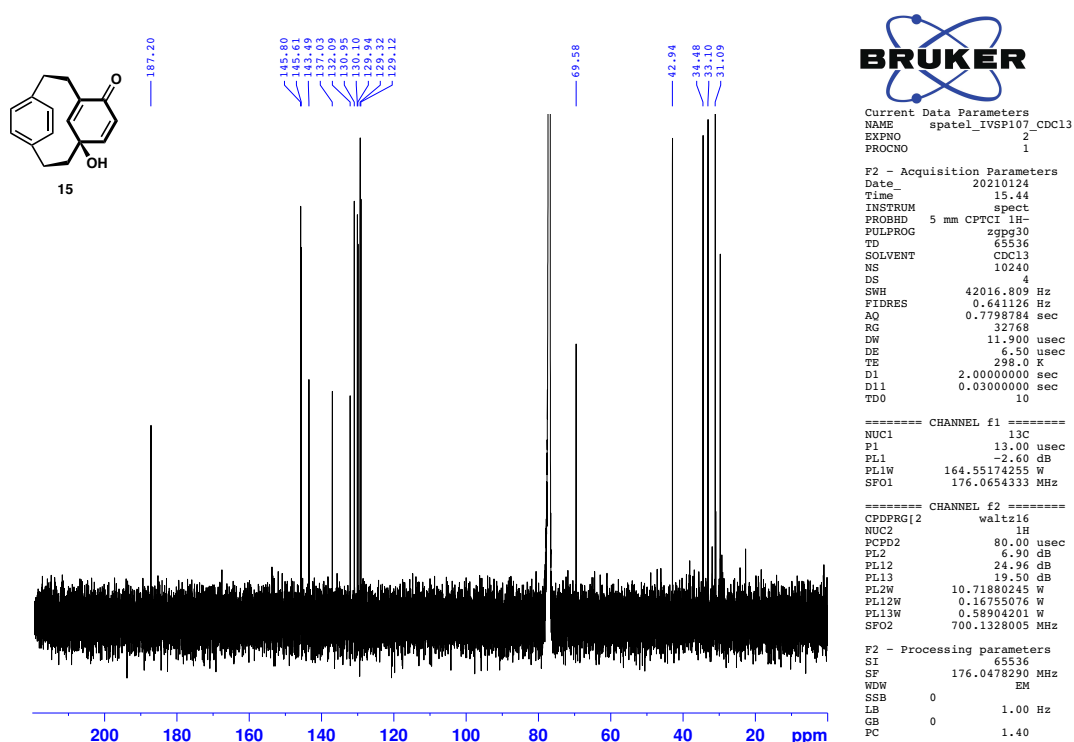
HMBC spectrum of **14** in CDCl₃



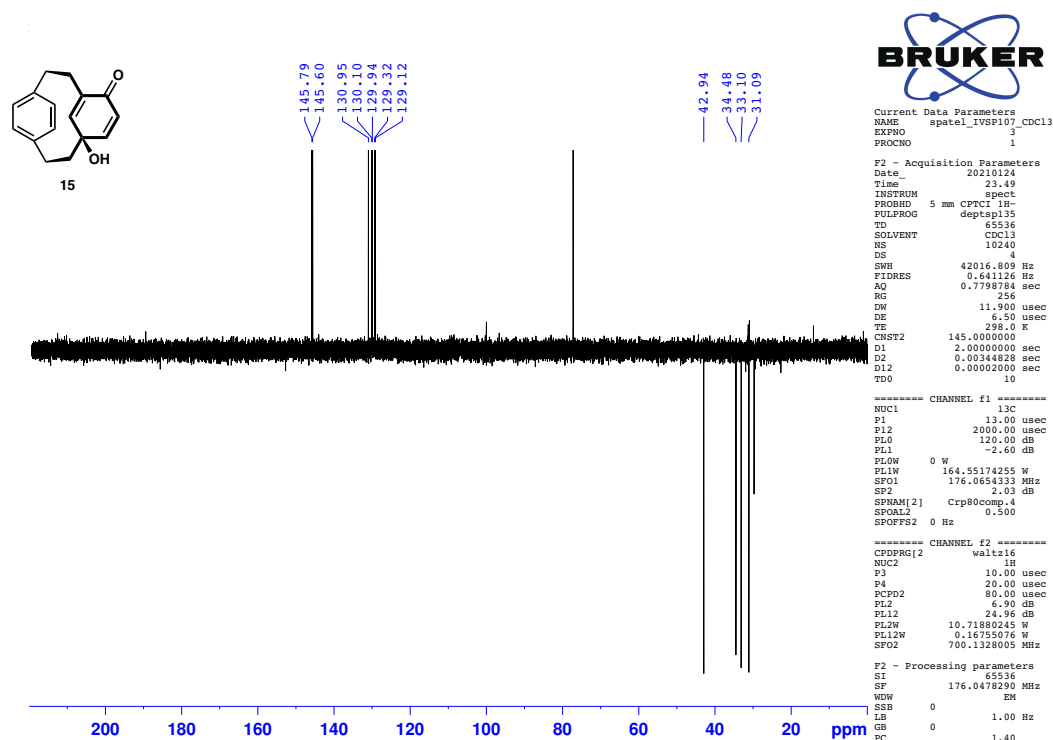
^1H NMR spectrum of **15** in CDCl_3



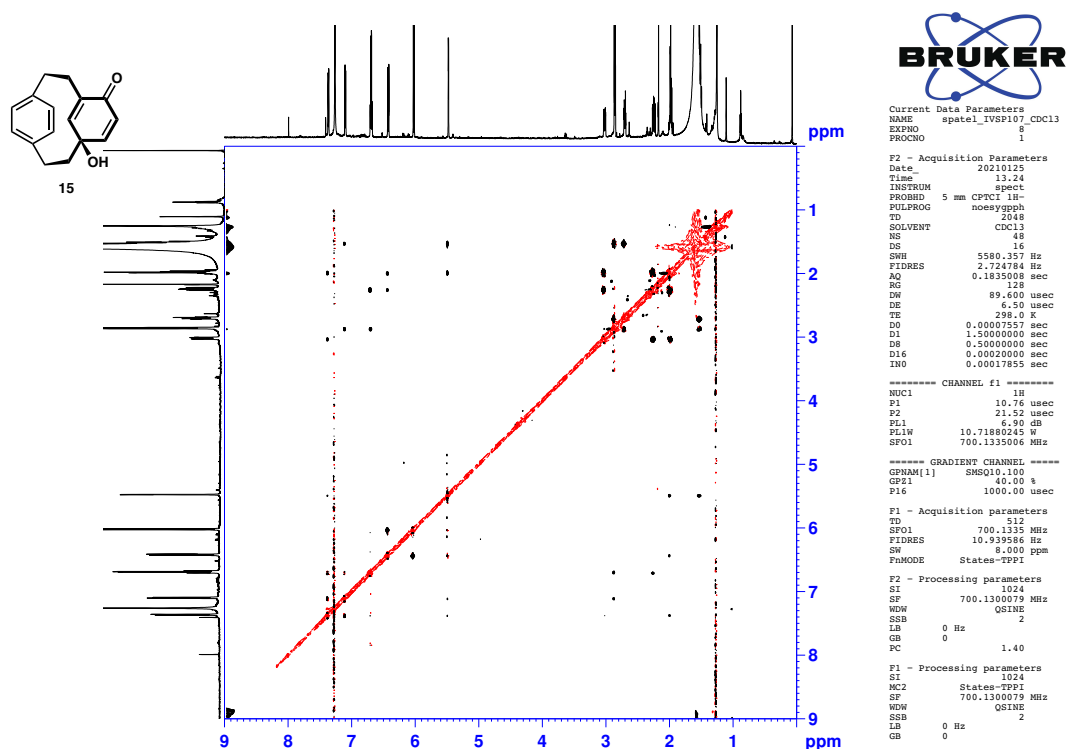
^{13}C NMR spectrum of **15** in CDCl_3



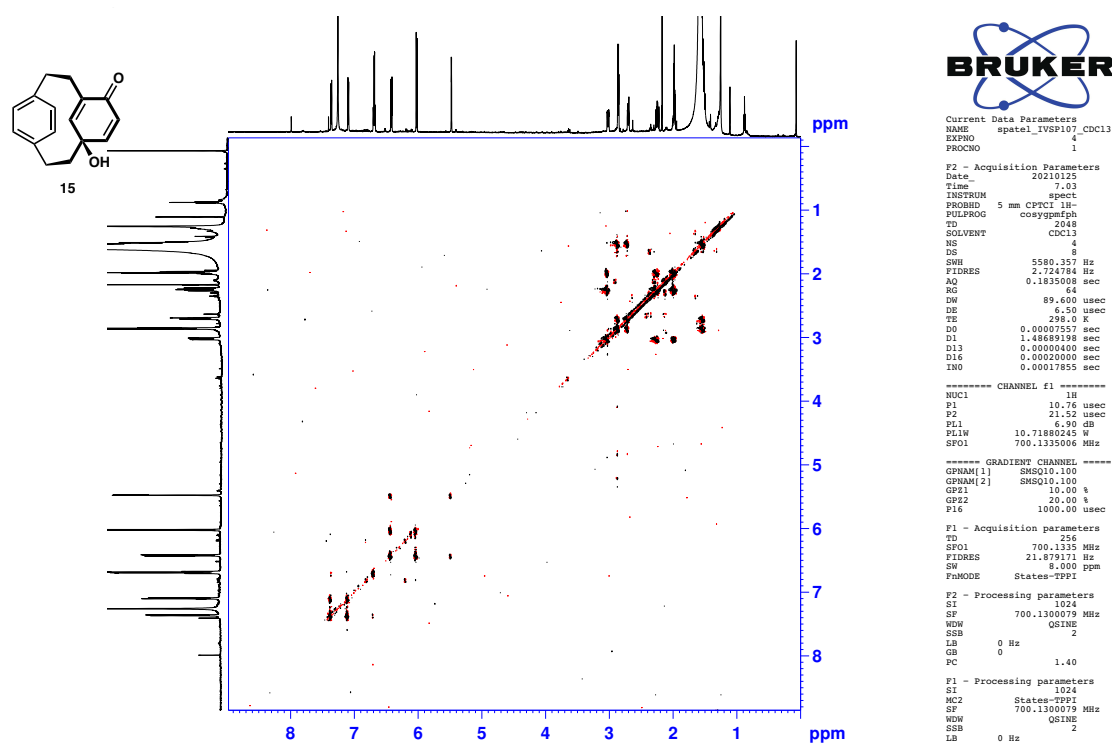
DEPT spectrum of **15** in CDCl₃



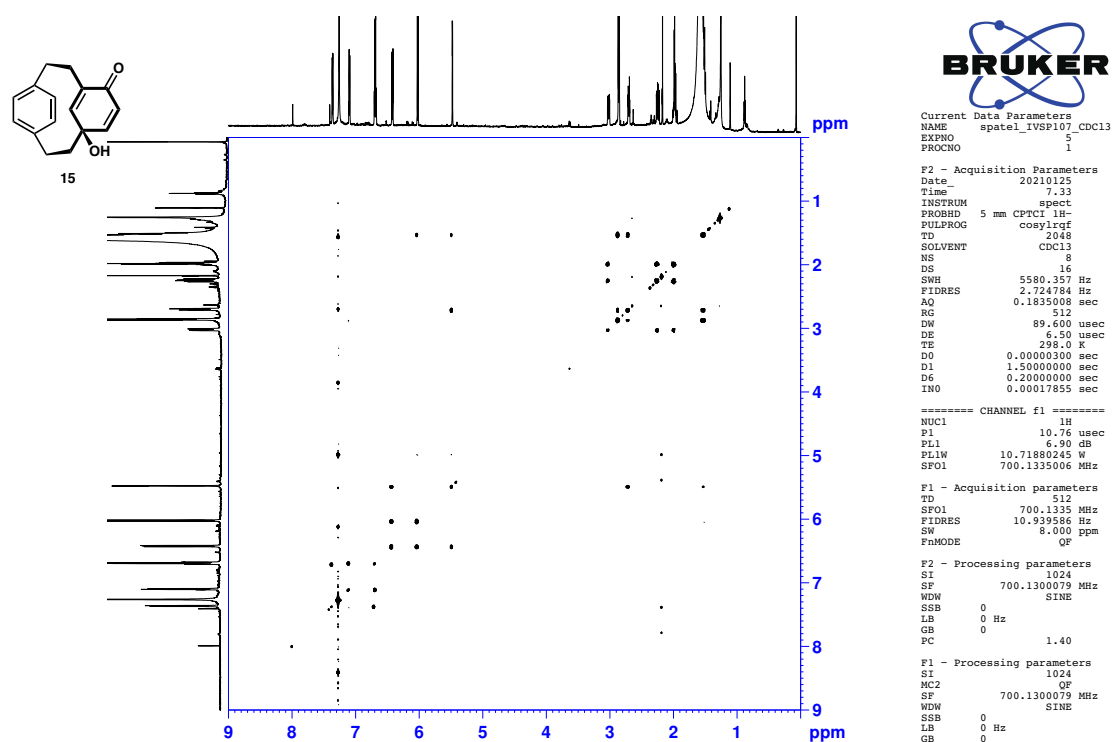
NOESY spectrum of **15** in CDCl₃



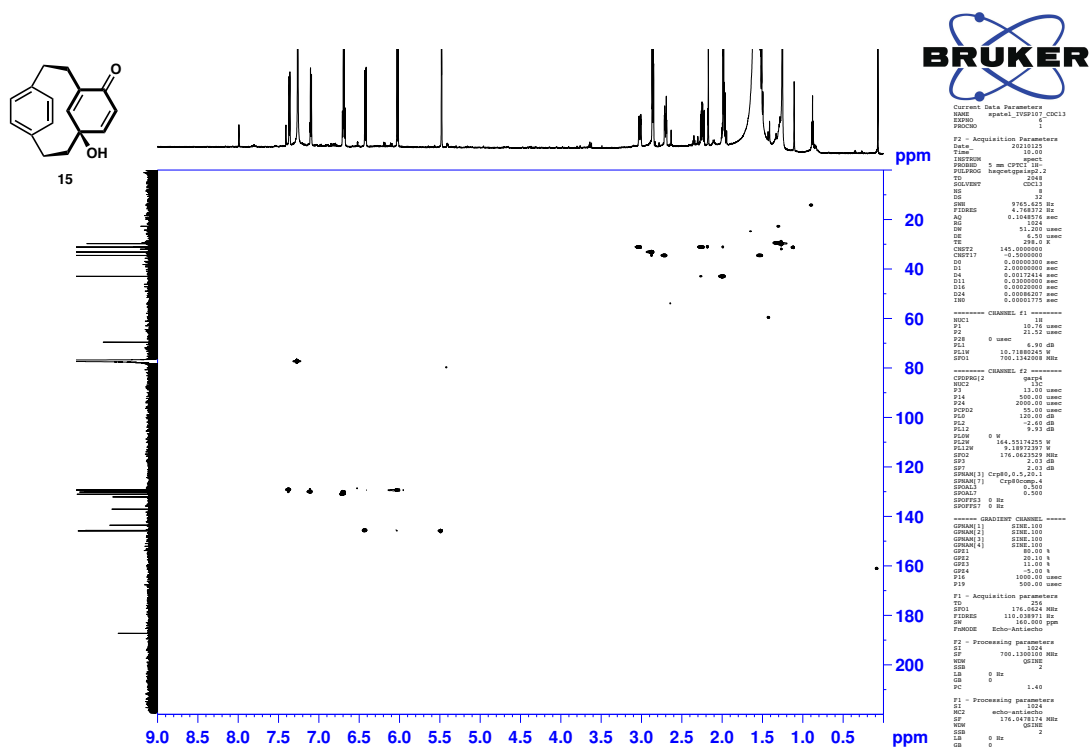
COSY spectrum of **15** in CDCl₃



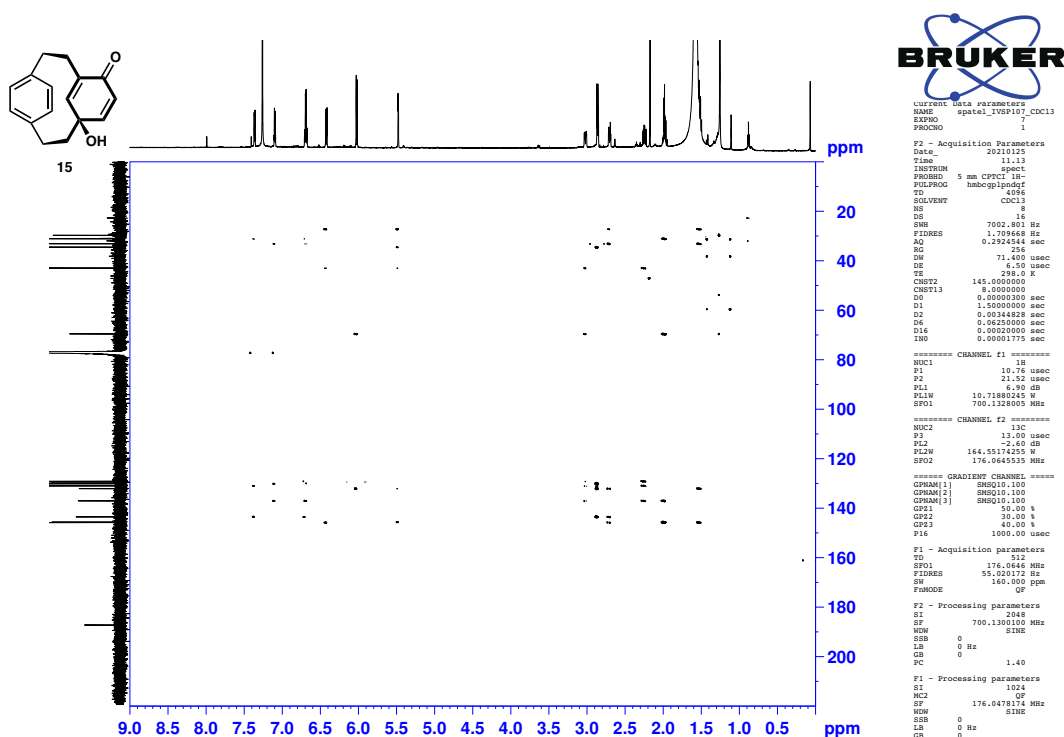
Long range COSY spectrum of **15** in CDCl₃



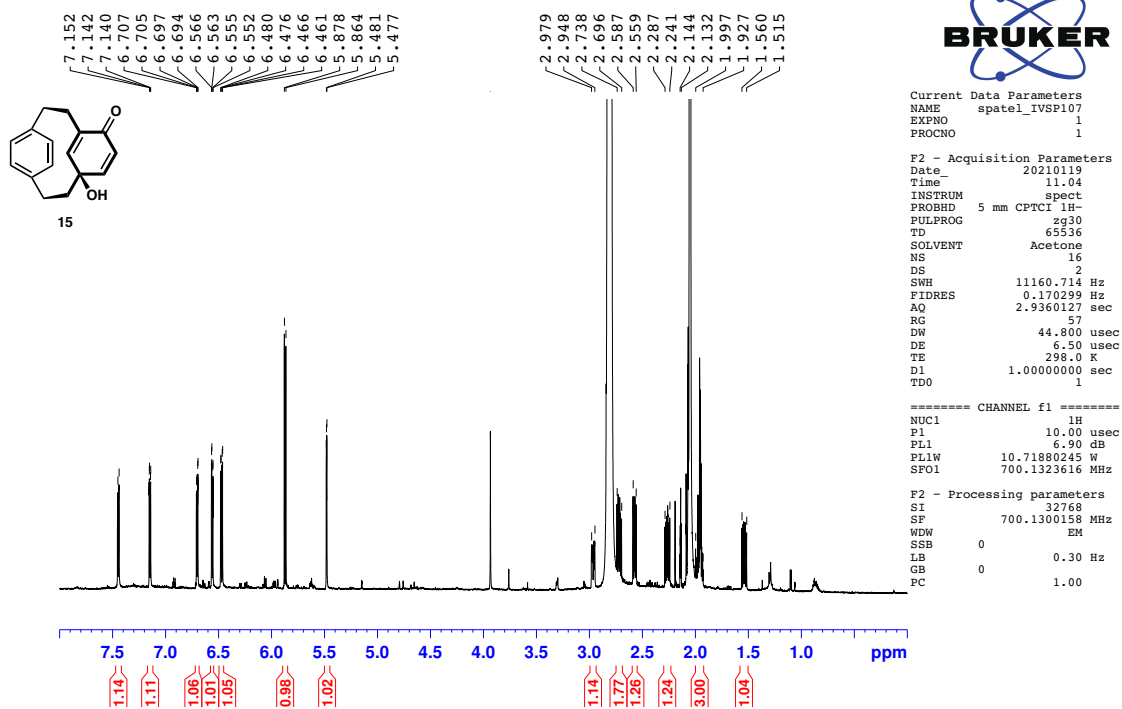
HMQC spectrum of **15** in CDCl₃



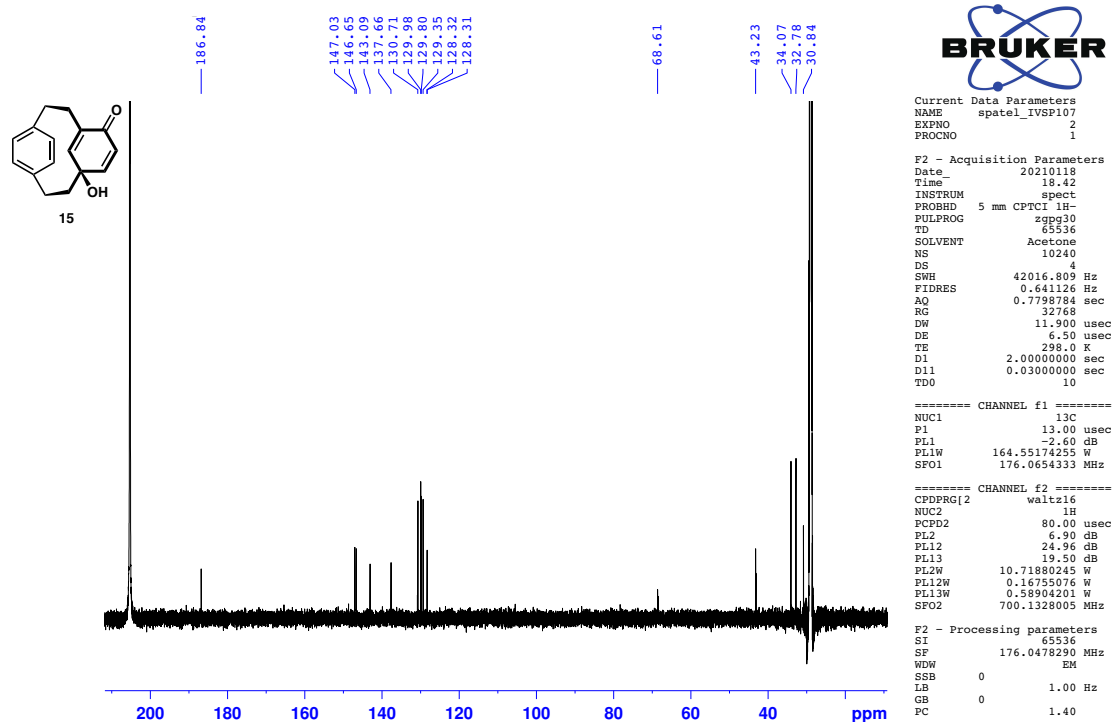
HMBC spectrum of **15** in CDCl₃



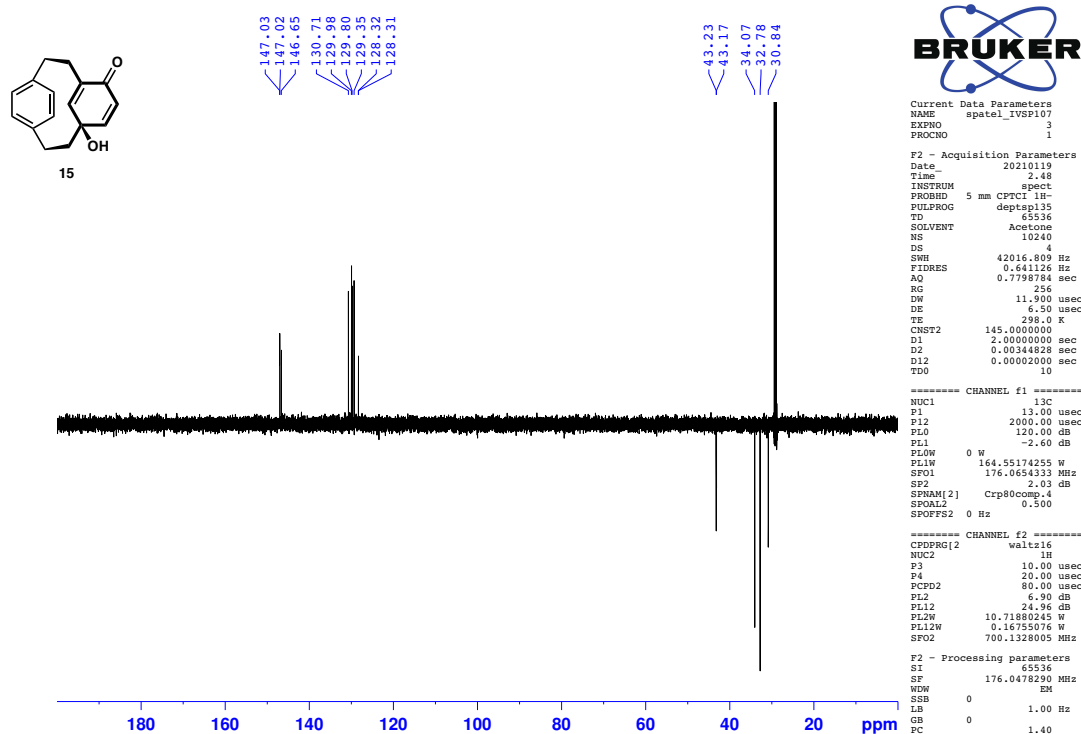
^1H NMR spectrum of **15** in $(\text{CD}_3)_2\text{CO}$



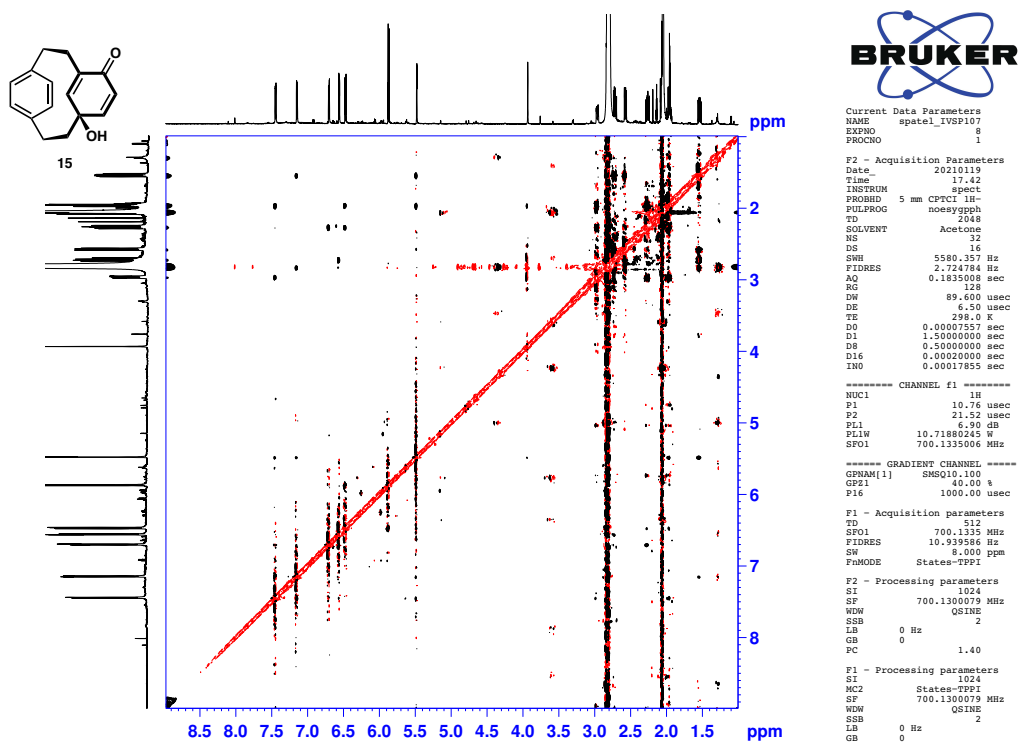
^{13}C NMR spectrum of **15** in $(\text{CD}_3)_2\text{CO}$



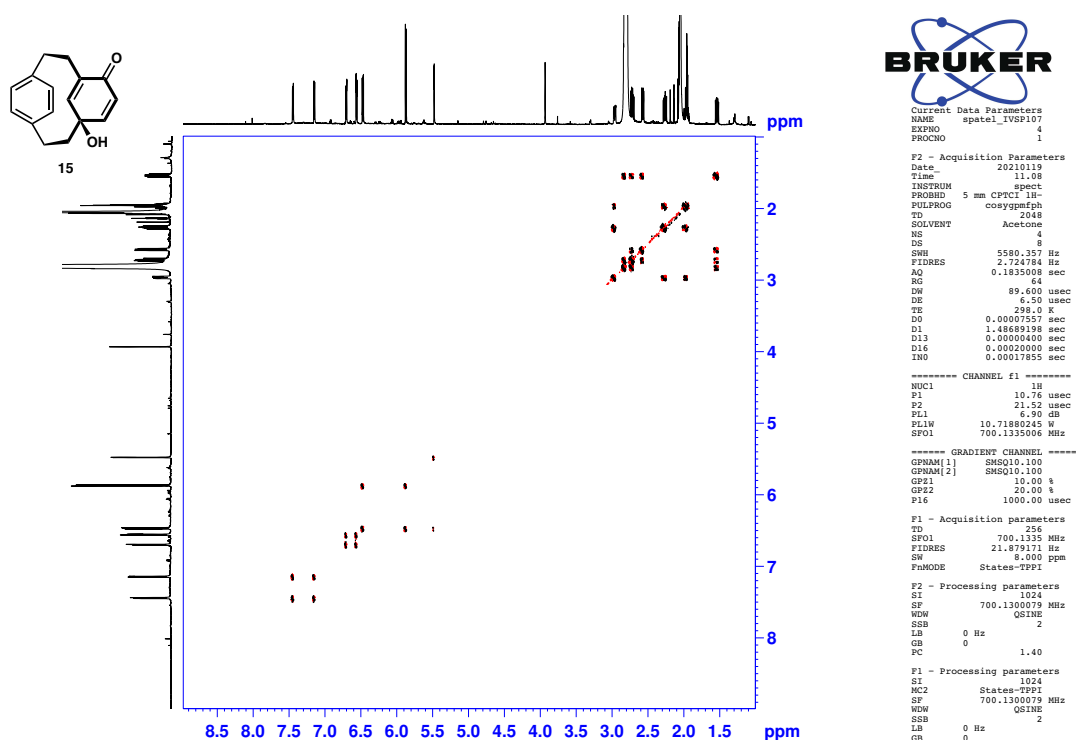
DEPT spectrum of **15** in (CD₃)₂CO



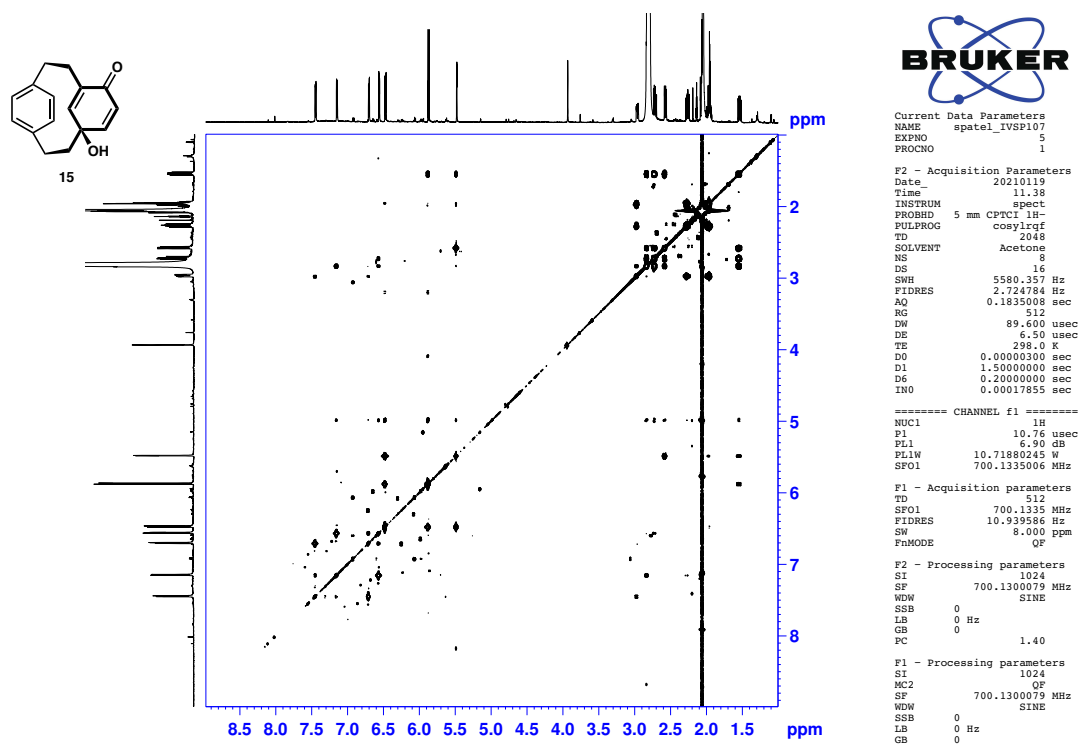
NOESY spectrum of **15** in (CD₃)₂CO



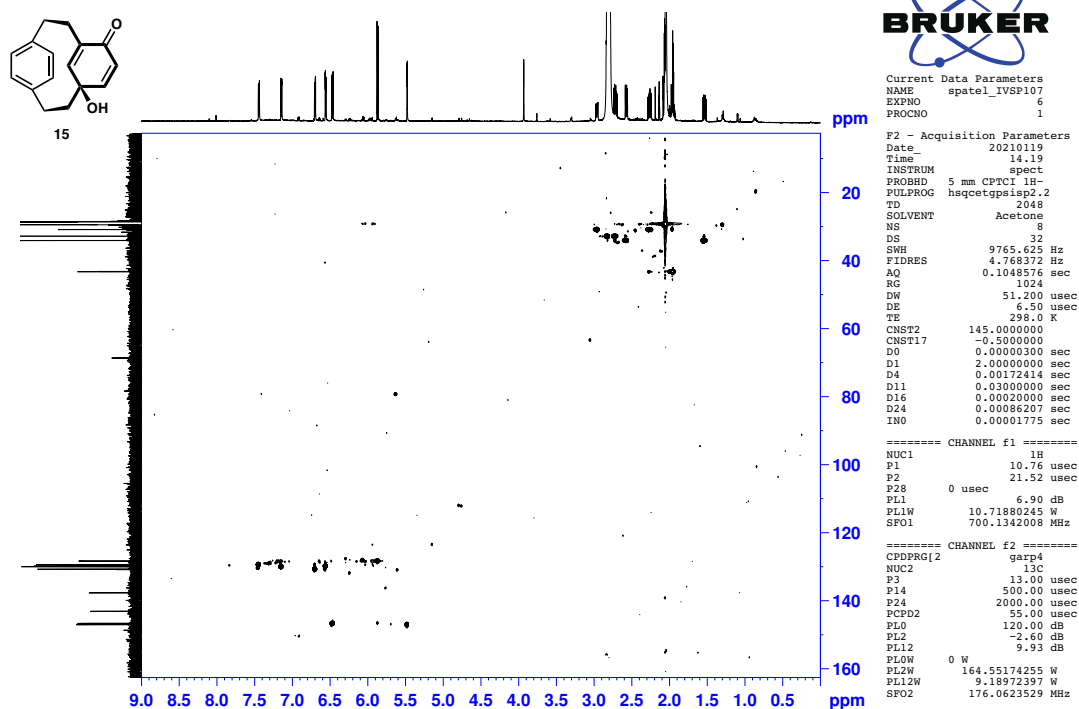
COSY spectrum of **15** in (CD₃)₂CO



Long range COSY spectrum of **15** in (CD₃)₂CO



HMQC spectrum of **15** in (CD₃)₂CO



HMBC spectrum of **15** in (CD₃)₂CO

