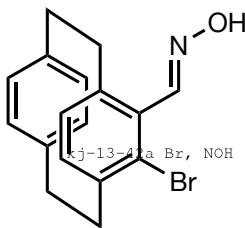


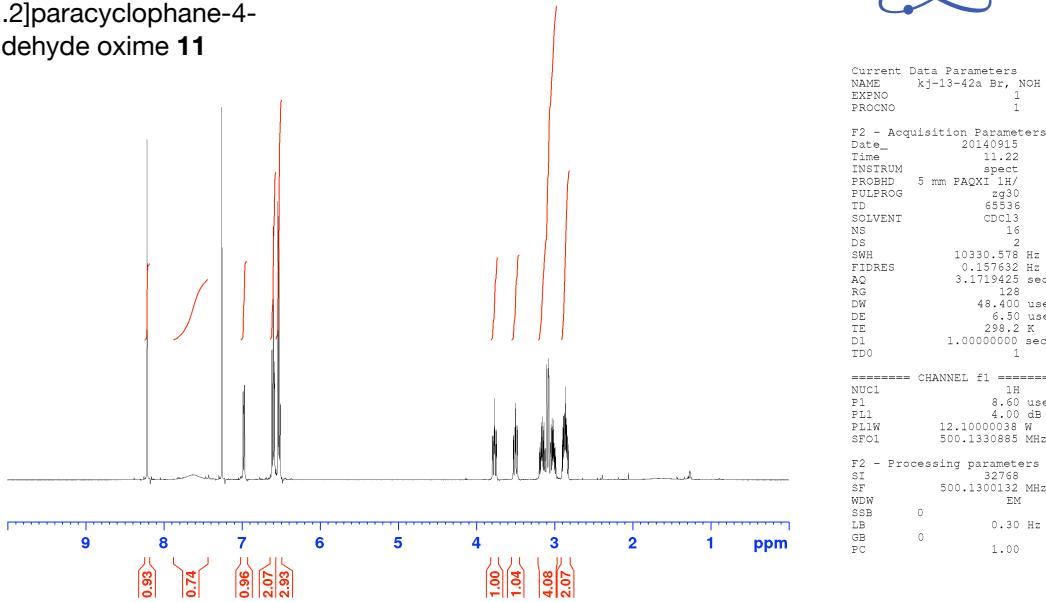
Supplementary information: The synthesis of planar chiral *pseudo-gem* aminophosphine pre-ligands based on [2.2]paracyclophane

Krishanthi P. Jayasundera, Tim G. W. Engels, David J. Lun, Maulik N. Mungalpara, Paul, G. Plieger, and Gareth J. Rowlands*

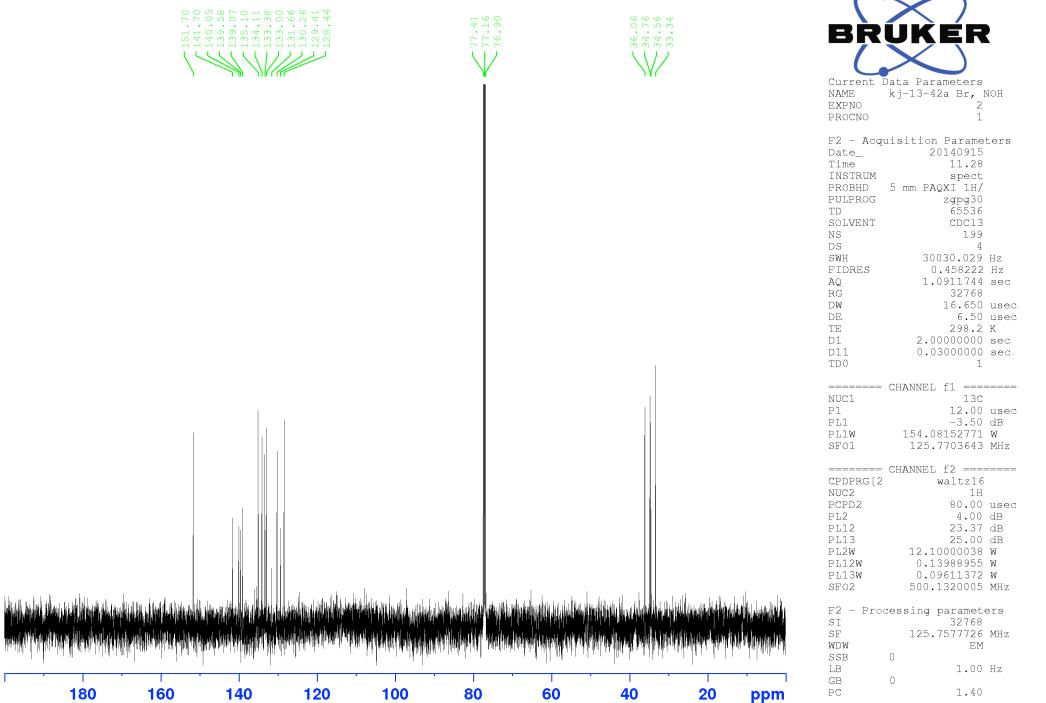


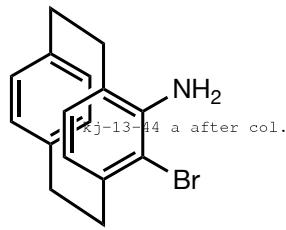
5-bromo[2.2]paracyclophane-4-carbaldehyde oxime **11**



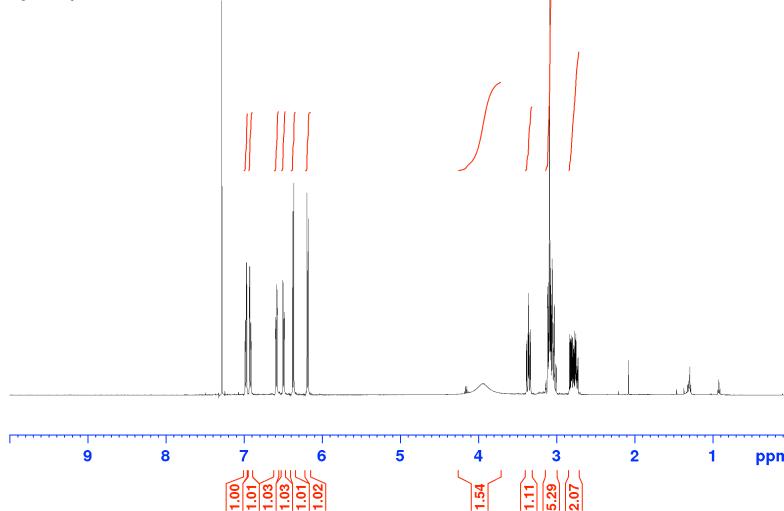


kj-13-42a Br, NOH





4-amino-5-bromo
[2.2]paracyclophane **12**





Current Data Parameter
NAME kj-13-44
EXPNO
PROCNO

```

F2 - Acquisition Parameters
Date_      20140917
Time       14.46
INSTRUM   spect
PROBHD   5 mm PAQX1 1H
PULPROG  zg30
TD        65536
SOLVENT    CDC13
NS         16
DS         2
SWH       10330.576 Hz
SFDRRES  0.157632
AQ        3.1719425 sec
RG        161.3
DW        48.400 usec
DE        6.50 usec
TE        298.2 K
D1        1.0000000 sec
TDO      1

```

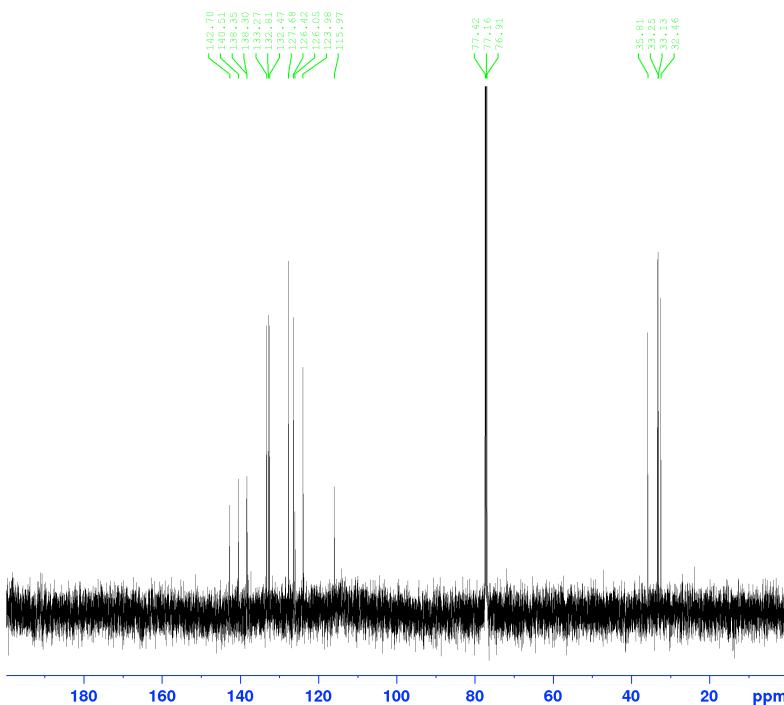
===== CHANNEL f1 =====
NUC1 1H
P1 8.60 usec
PLL 4.00 dB
PL1W 12.10000038 W
SFO1 500.1330895 MHz

```

F2 - Processing parameters
SI      32768
SF      500.1300000 MHz
WDW      EM
SSB      0
LB      0.30 Hz
GB      0
PC      1.00

```

kj-13-44 a after col



BRUKER

Current Data Parameters
NAME kj-13-44a
EXPNO 2
PROCNO 1

```

F2 - Acquisition Parameters
Date_        20140917
Time         14.58
INSTRUM     spect
PROBHD      5 mm PFGX1 1H
PULPROG    zg3sp30
TD          65536
SOLVENT     CDCl3
PROCNO     001
NS           203
DS           4
SWH         30030, 0.29 Hz
FIDRES     0.458222 Hz
AQ          1.0911744 sec
RG          32768
DW          16.00 usec
DE          6.50 usec
TE          298.2 K
D1          2.0000000 sec
D11         0.0300000 sec
TD0         1

```

```
===== CHANNEL f1 =====
NUC1          13C
P1           12.00 usec
PL1          -3.50 dB
PL1W         154.08152771 W
SFO1        125.7703643 MHz
```

```

----- CHANNEL f2 -----
CPDPRG[2]          waltz16
NUC2                1H
PCPD2              80.00 used
PL2                 4.00 dB
PL12                23.37 dB
PL13                25.00 dB
PL2W               12.10000038 W
PL12W              0.13988955 W
PL13W              0.09611372 W

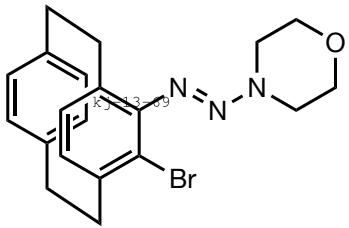
```

```

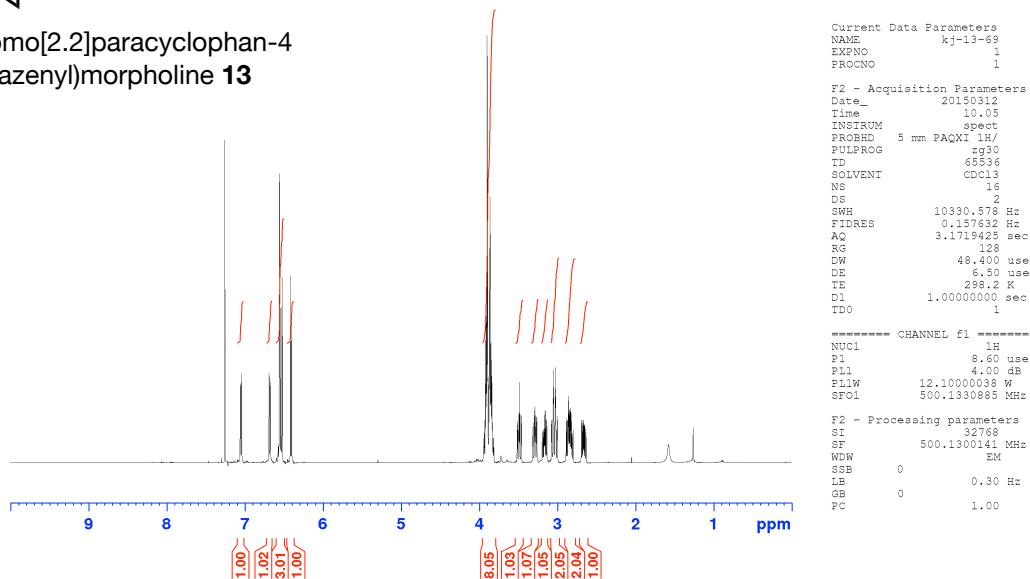
SF02      300.1320000 MHz

F2 - Processing parameters
SI          32168
SF        125.7577726 MHz
WDW          EM
SSB          0
LB          1.00 Hz
GB          0
PC          1.40

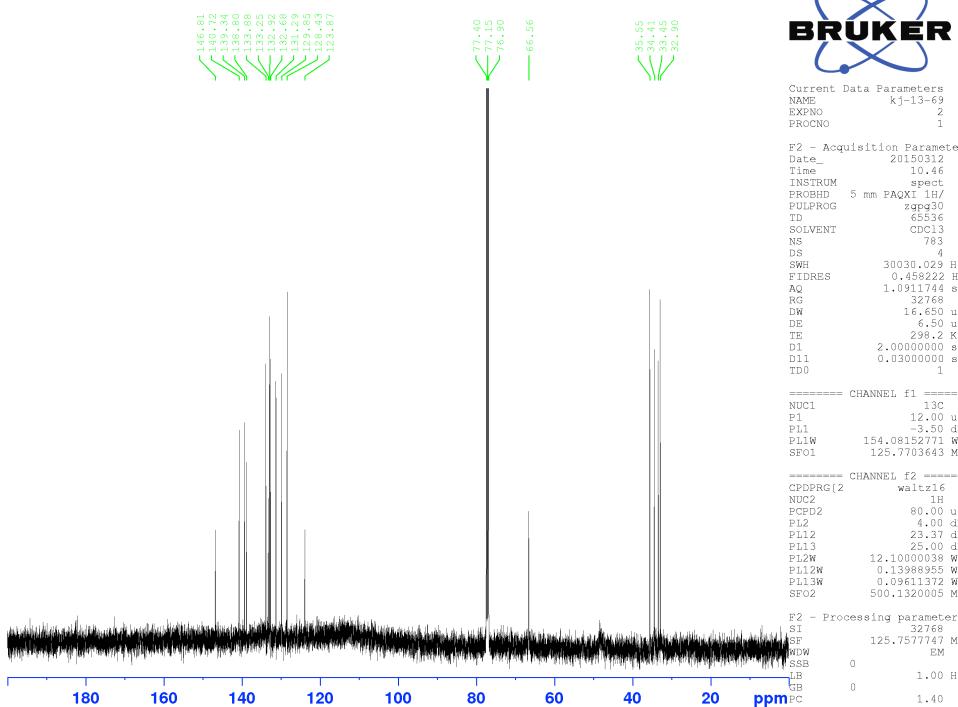
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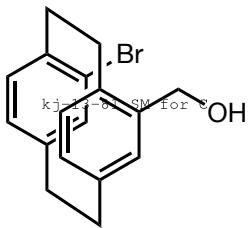


4-(5-bromo[2.2]paracyclophan-4-ylidaz恒)morpholine **13**



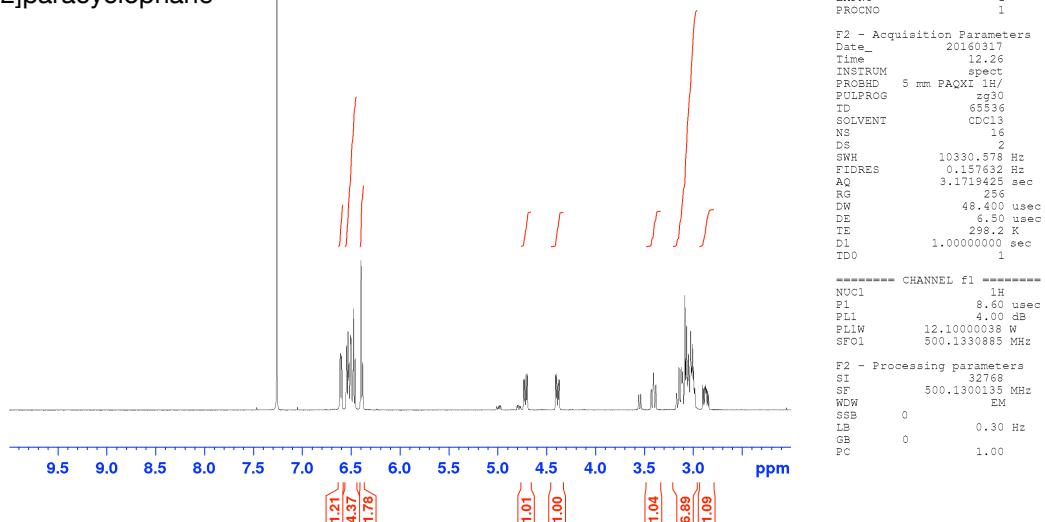
kj-13-69



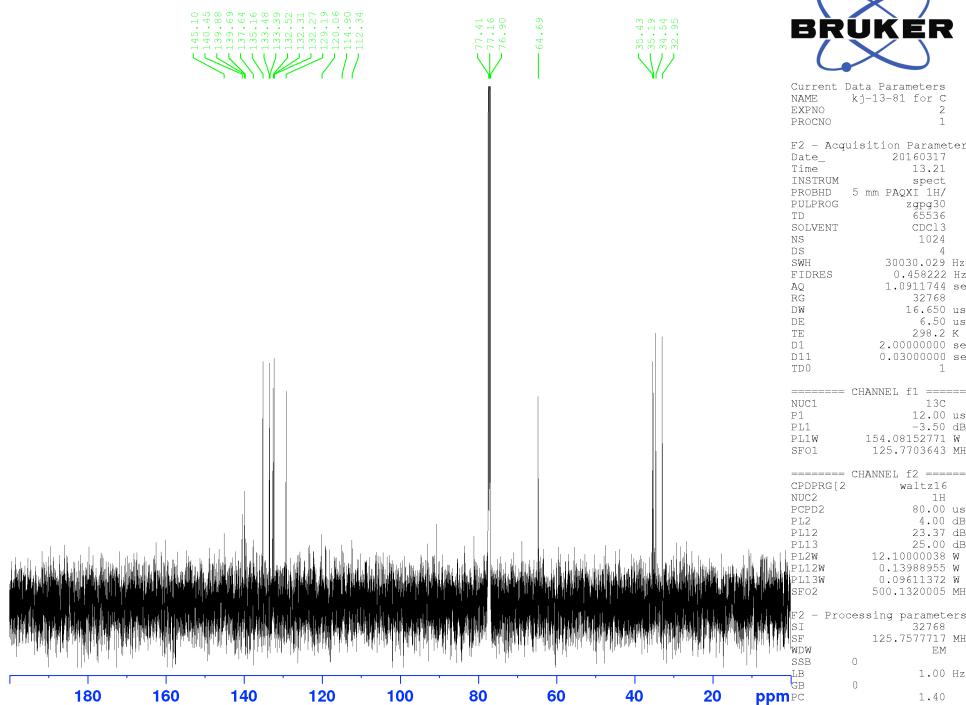


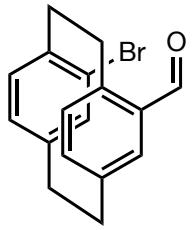
13-bromo-4-hydroxymethyl [2.2]paracyclophane





kj-13-81 for C

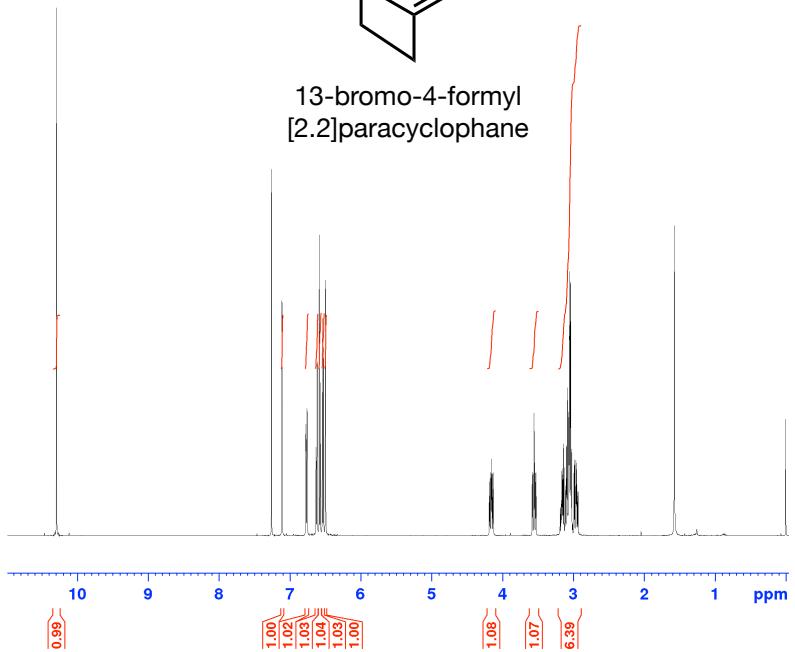




13-25 CHO, Br



13-bromo-4-formyl [2.2]paracyclophane



```

Current Data Parameters
NAME          kj-13-25
EXPNO         1
PROCNO        1

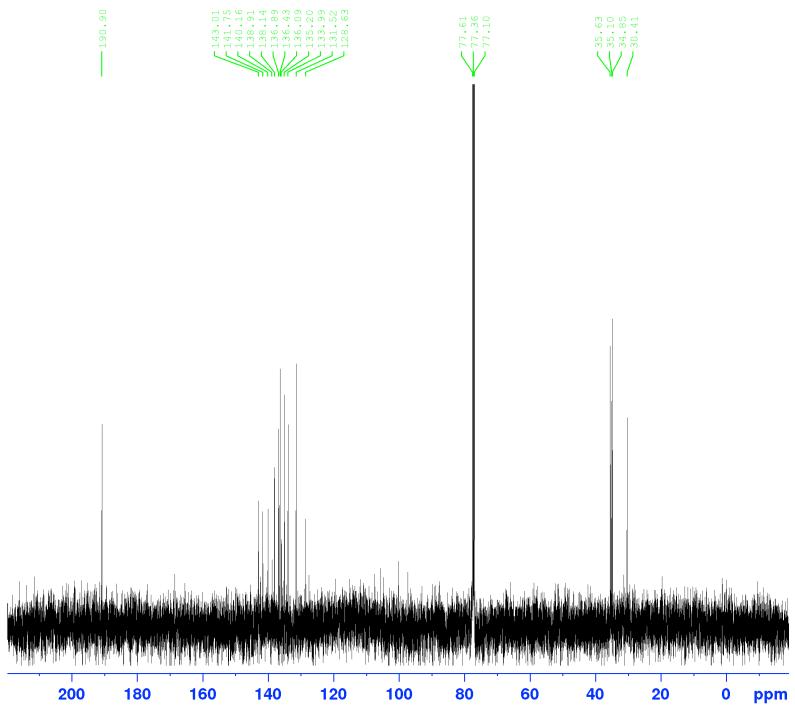
F2 - Acquisition Parameters
Date_        20140709
Time         9.50
INSTRUM     SP600
PROBOD      5 mm PAQX1 H
PULPROG    zg30
TD          65536
SOLVENT      CDC13
NS           16
DS            2
SWH         10330.578 Hz
TDRES      0.157632 sec
AQ          3.17111 sec
RG           181
DW           48.00 usec
DE           6.50 usec
TE           298.2 K
D1          1.0000000 sec
TDO          1

===== CHANNEL f1 =====
NUC1          1H
PL1          8.60 usec
PL1          4.00 dB
PL1W        12.100000038 W
SF01        500.1330885 MHz

F2 - Processing parameters
ST           32768
SF          500.1300133 MHz
NDW          EM
SSB           0
LB           0.30 Hz
GB           0
PC           1.00

```

13-25 CHO, Br



BRUKER

```

Current Data Parameters
NAME          kj-13-25
EXPNO         2
PROCNO        1

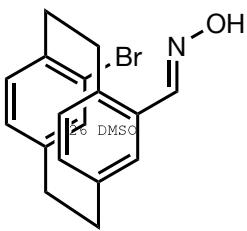
F2 - Acquisition Parameters
Date_        20140709
Time          9.55
INSTRUM     spect
PROBHD      5 mm PFG1 1H
PULPROG     zg30
TD           81920
SOLVENT      CDCl3
NS            157
DS             4
SW8            30032.02 Hz
ETR           0.458220
AQ           1.03917144 sec
RG            114
DW           16.650 usec
DE            6.500 K
TE            293.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

===== CHANNEL F2 =====
NUC1          13C
PL1           12.00 usec
PL1L          -3.50 dB
PL1W        154.08152717 MHz
SF01          125.703643 MHz

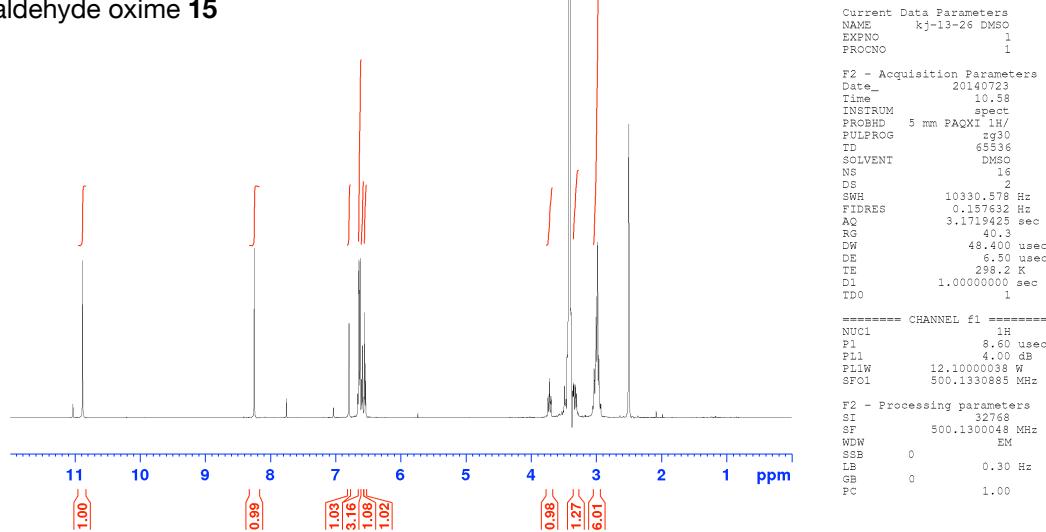
===== CHANNEL F2 =====
CPDPGR2[2       waltz16
NUC2          1H
PCPD2        80.00 usec
PL2           4.00 dB
PL2L          25.00 usec
PL2W        12.10000038 dB
PL12W        0.13988955 W
PL13W        0.09611372 W
SF02          500.13200000 MHz

F2 - Processing parameters
SI            32768
SF          125.7577475 MHz
WDW           EM
SSB            0
LB           1.00 Hz
GB            0
PC            1.40

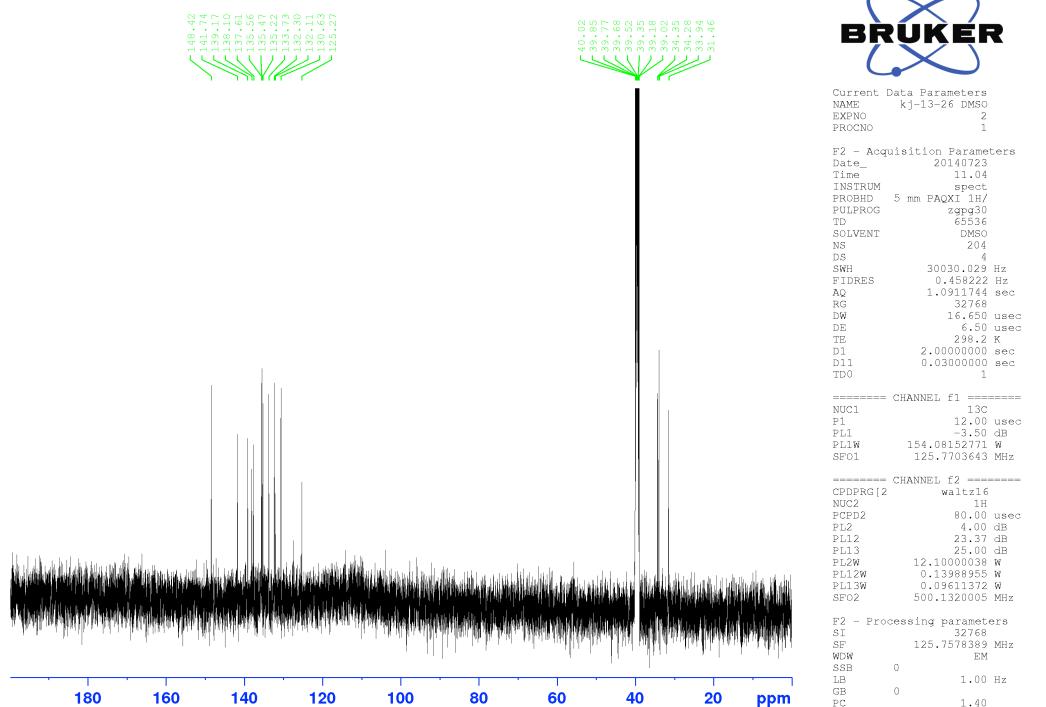
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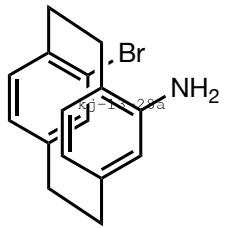


13-bromo[2.2]paracyclophane-4-carbaldehyde oxime **15**

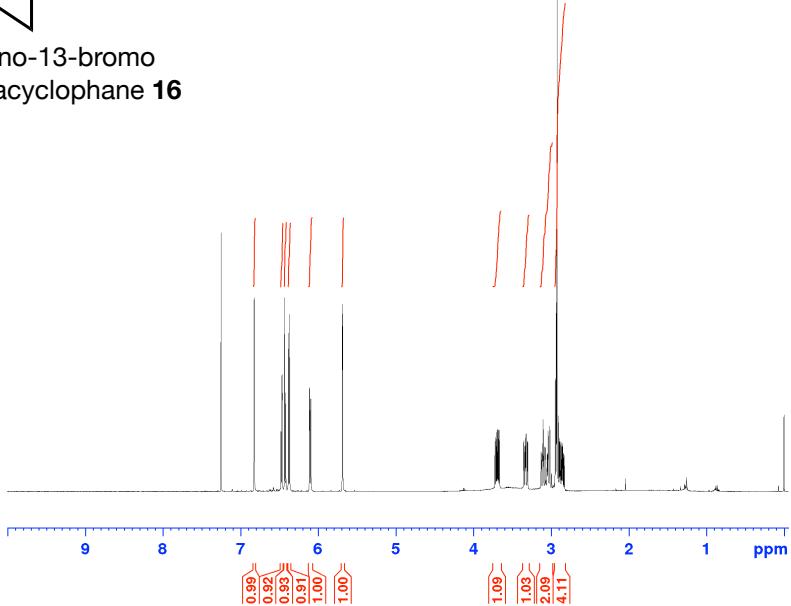


26 DMSO

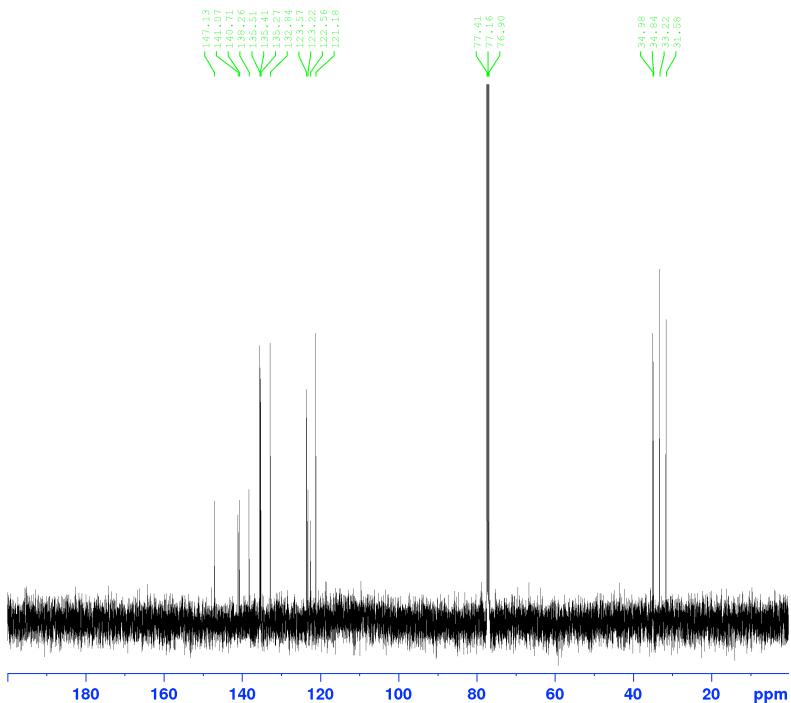


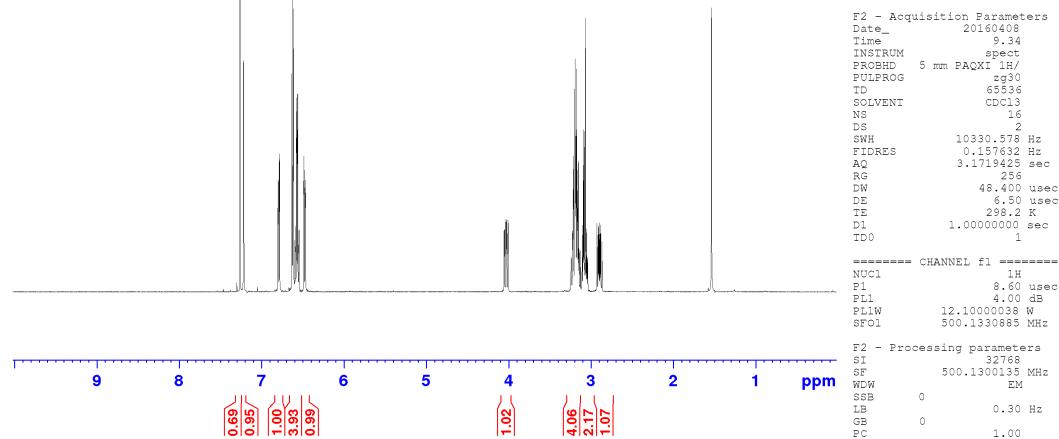
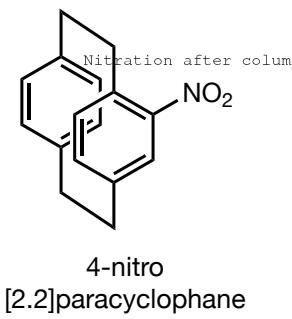


4-amino-13-bromo
[2.2]paracyclophane **16**



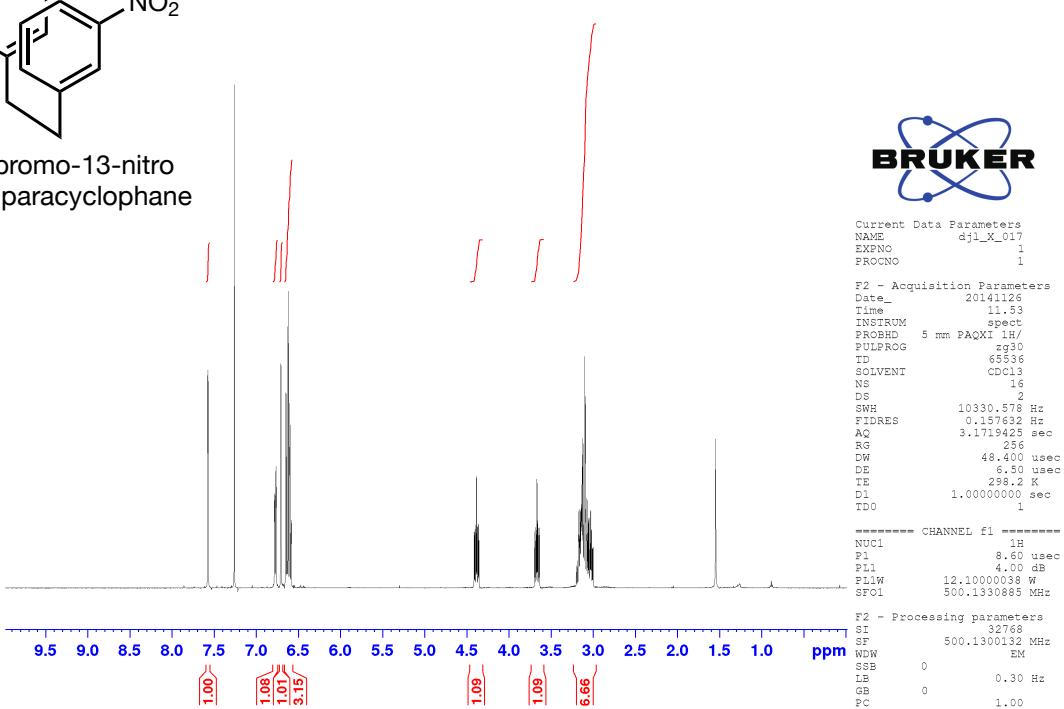
kj-13-28a

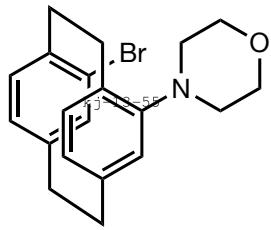




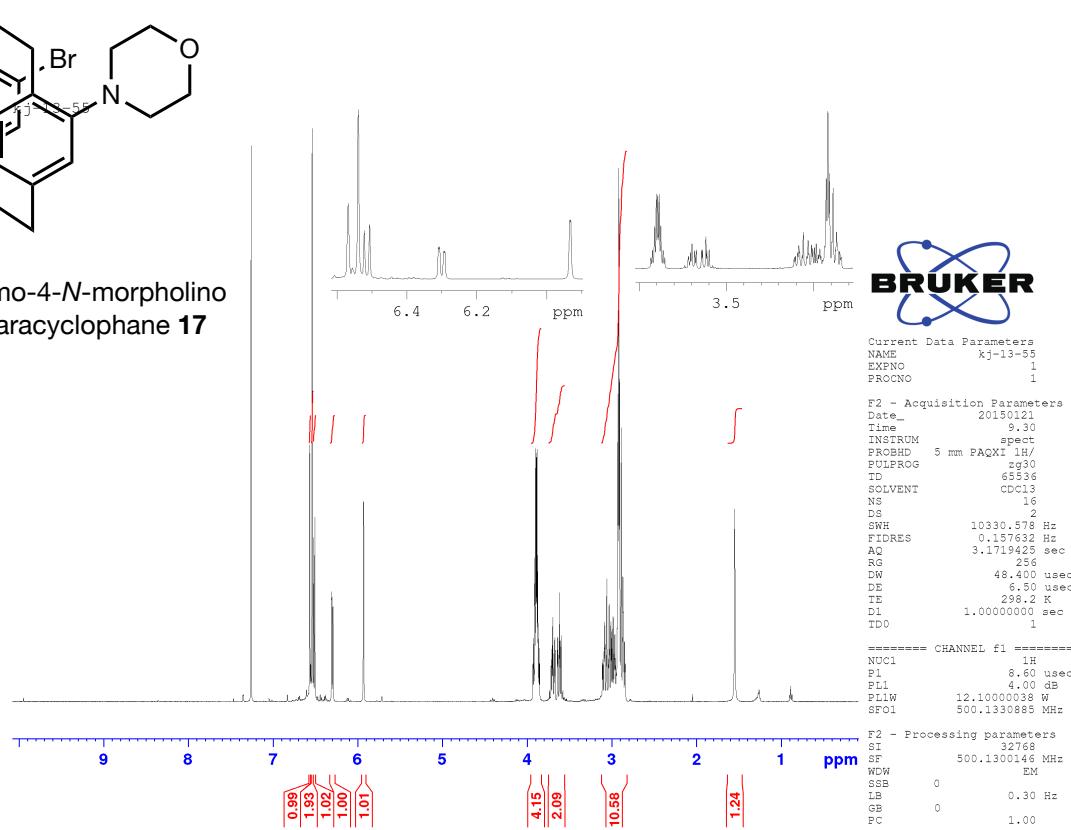


column of bromination reaction

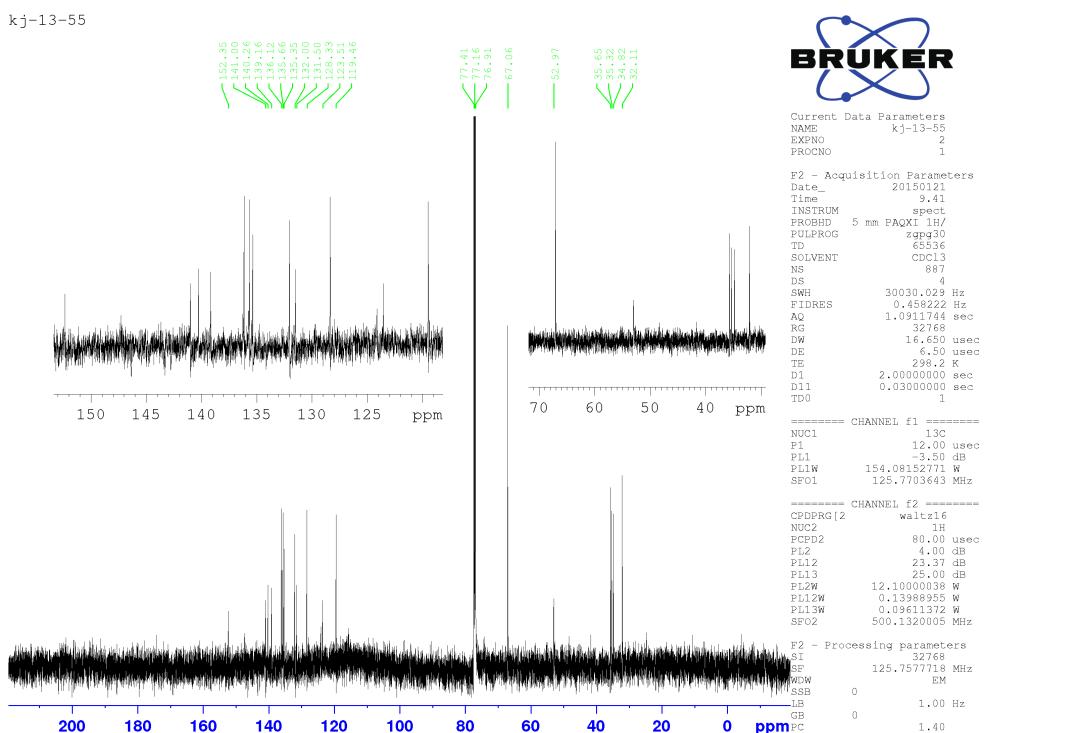


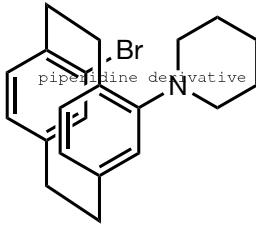


13-bromo-4-N-morpholino
[2.2]paracyclophane **17**

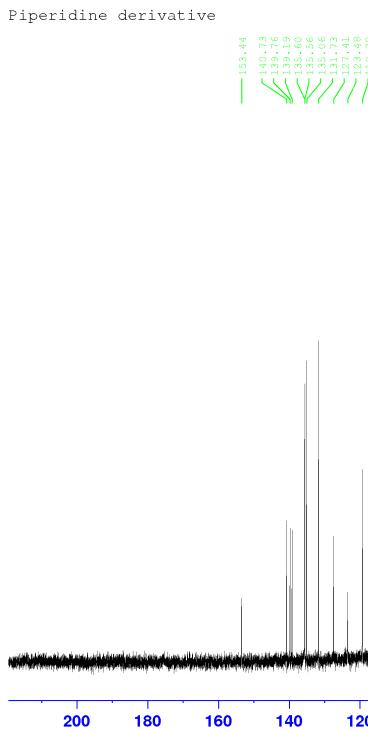
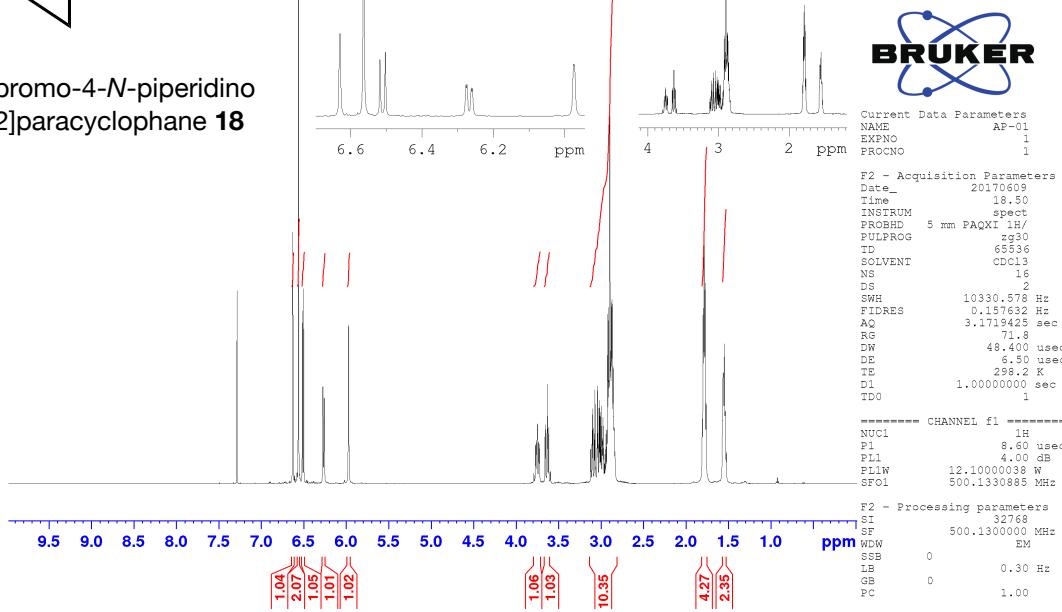


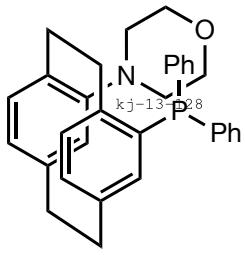
kj-13-55



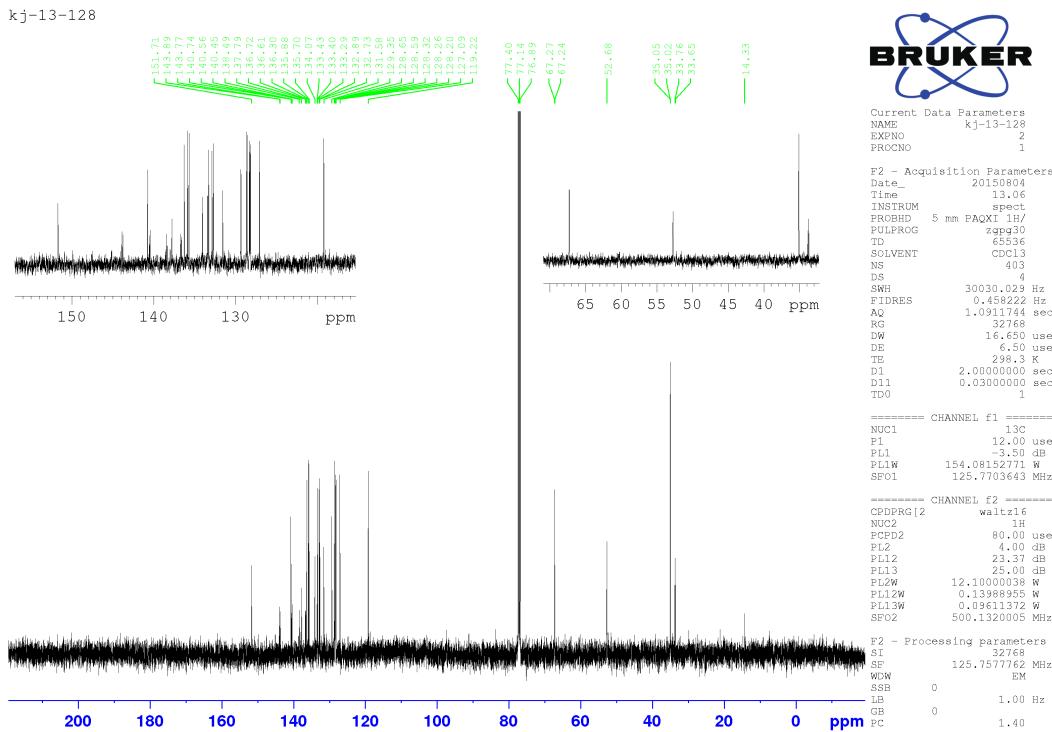
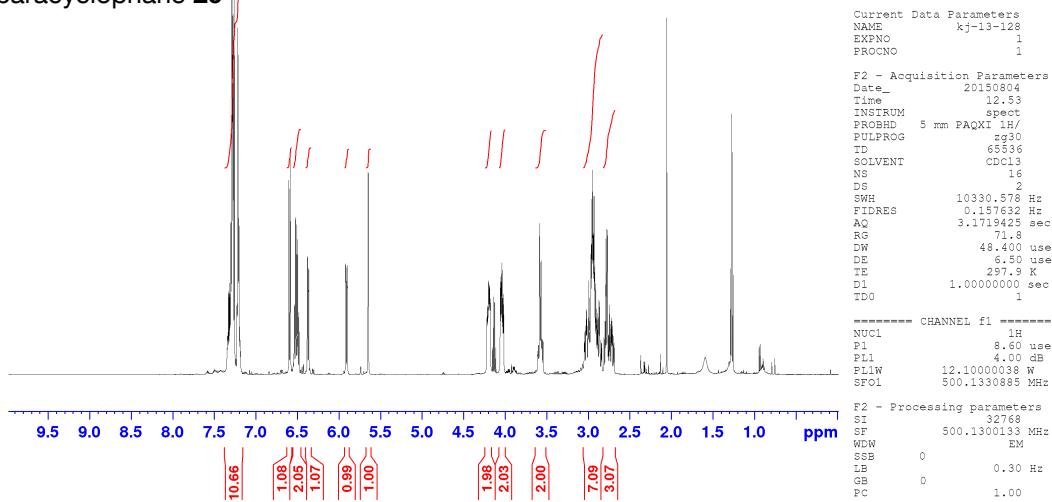


13-bromo-4-N-piperidino [2.2]paracyclophane **18**





4-diphenylphosphino-13-N-morpholino[2.2]paracyclophane **20**



kj-13-128



Current Data Parameters
NAME kj-13-128
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters

DTime 20150804
Time 11.19
INSTRUM spect
PROBHD 5 mm PAQXI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 12
DS 4
SWH 80645.164 Hz
FIDRES 1.230548 Hz
AQ 0.4063232 sec
RG 20642.5
DW 6.50 usec
DE 6.50 usec
TE 298.2 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

===== CHANNEL f1 =====

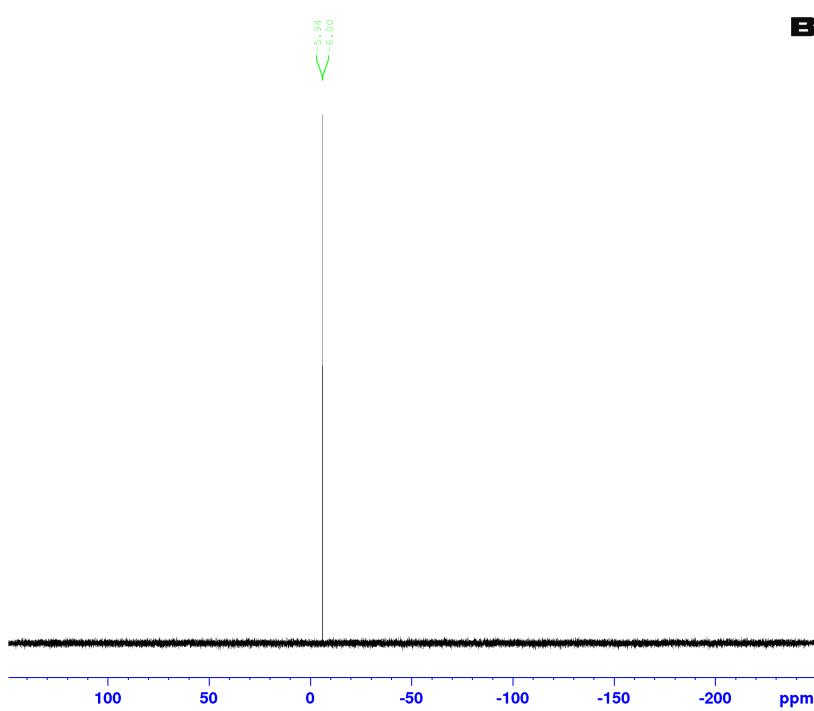
NUC1 31P
P1 20.00 usec
PL1 -2.40 dB
PL1W 131.39126587 W
SF01 202.4462122 MHz

===== CHANNEL f2 =====

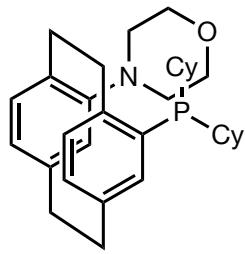
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 23.37 dB
PL13 25.00 dB
PL2W 12.10000038 W
PL12W 0.1398955 W
PL13W 0.09611372 W
SF02 500.1320005 MHz

F2 - Processing parameters

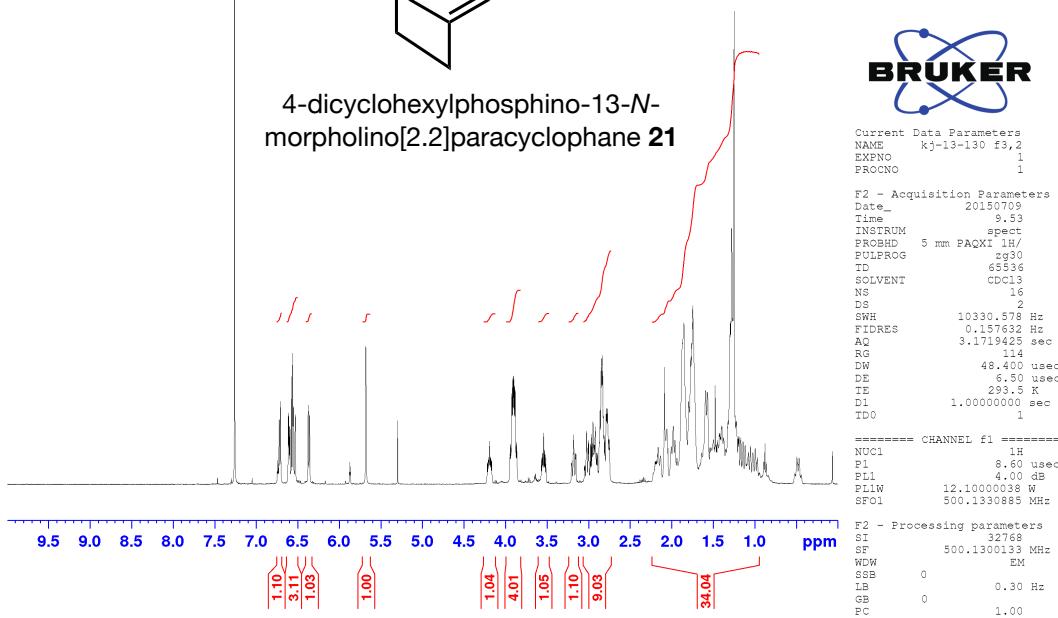
SI 32768
SF 202.4563350 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

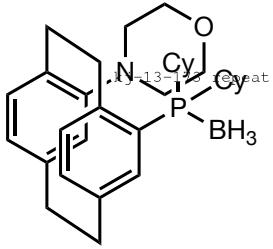


kj-13-130 f3, 2

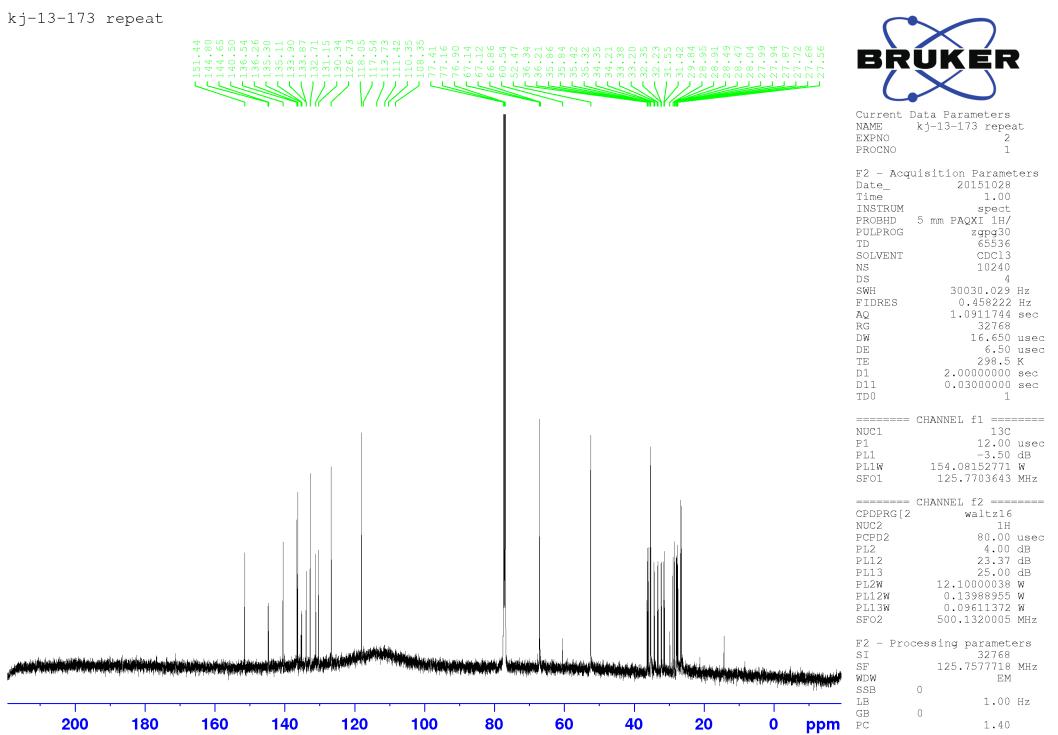
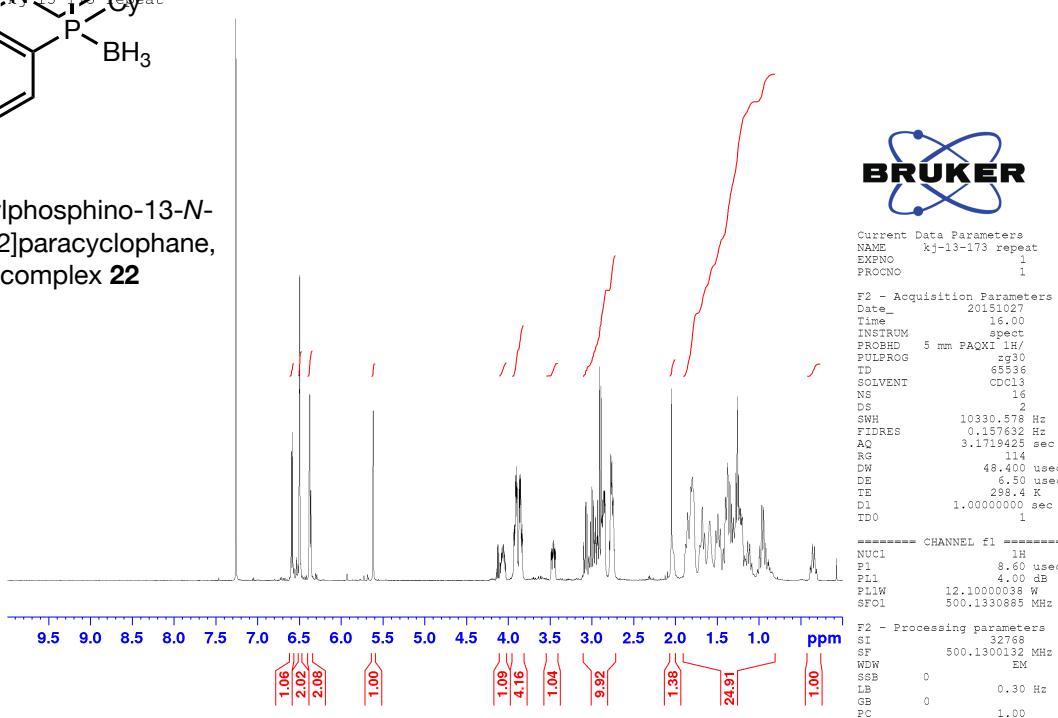


4-dicyclohexylphosphino-13-*N*-morpholino[2.2]paracyclophe **21**





4-dicyclohexylphosphino-13-N-morpholino[2.2]paracyclophane,
borane complex **22**



kj-13-173 repeat



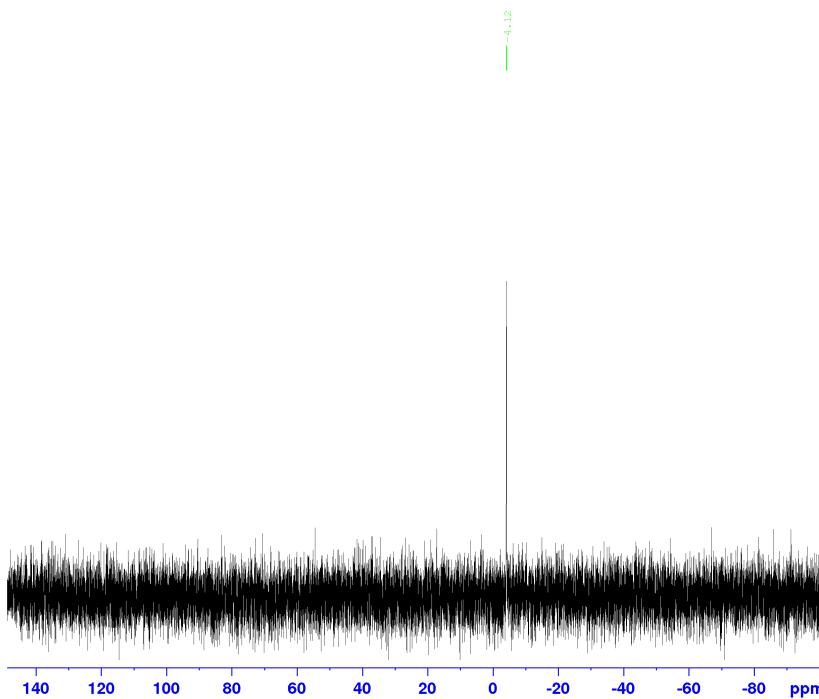
Current Data Parameters
NAME kj-13-173 repeat
EXPNO 3
PRGCNO 1

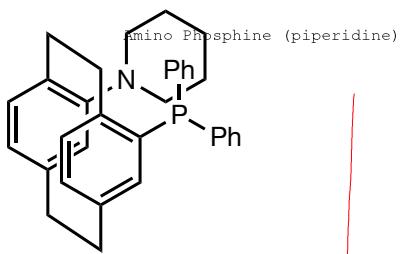
F2 - Acquisition Parameters
Date_ 20151008
Time 13.49
INSTRUM spect
PROBHD 5 mm PABGX1 1H/
PULPROG zgpg30
TD 65536
SW1 16384
DW 6536
SOLVENT CDCl3
NS 16
DS 4
SWH 80645.164 Hz
FIDRES 1.230548 Hz
AQ 0.4063232 sec
RG 20645
TDW 6.200 usec
DE 6.50 usec
TE 297.9 K
D1 2.0000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 31P
P1 20.00 usec
PL1 -2.40 dB
PL1W 131.39126587 W
SF01 202.4462122 MHz

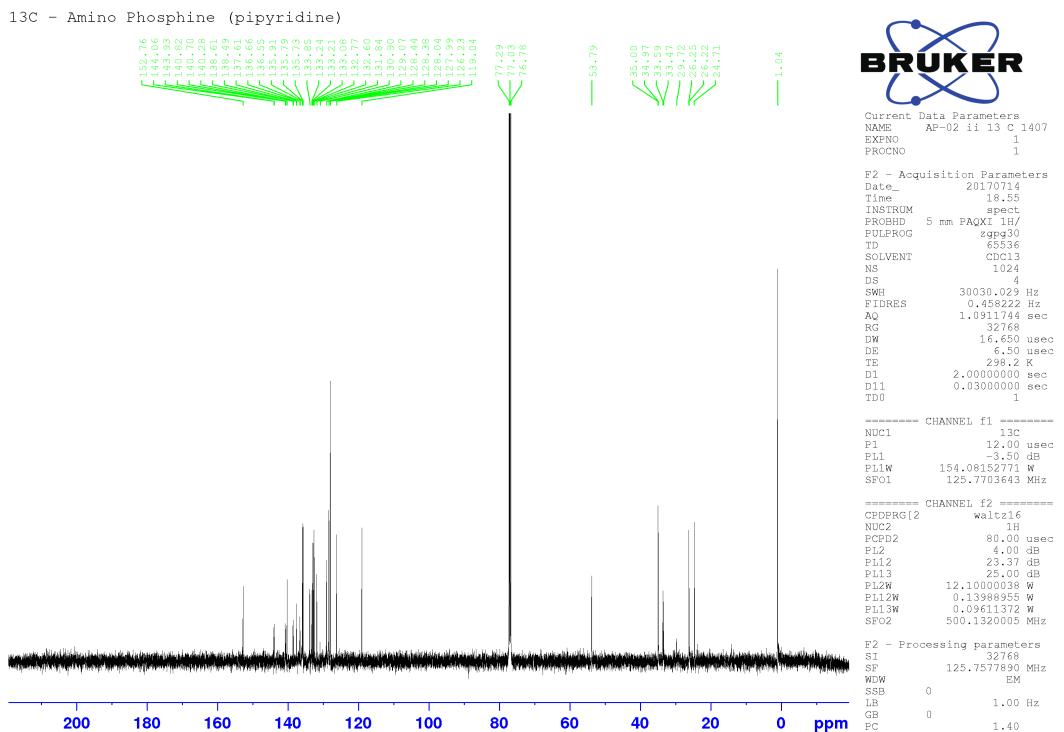
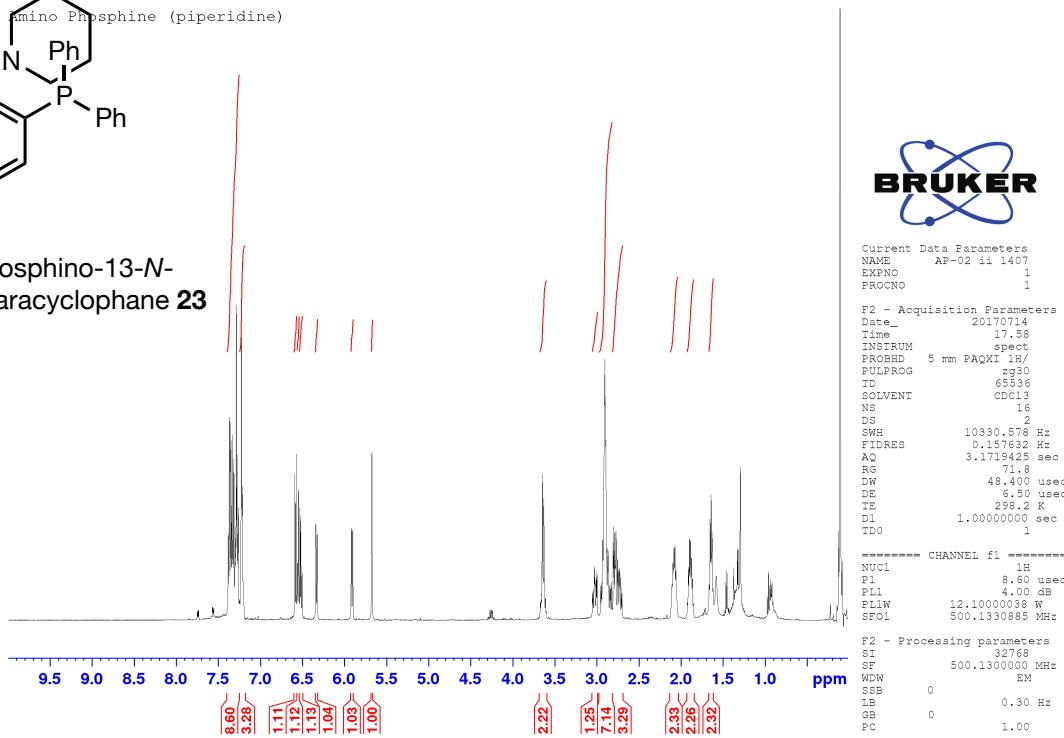
===== CHANNEL f2 =====
CPDPRG[2 walc16
NUC2 1H
P1C2 80.00 usec
PL2 4.00 dB
PL12 23.37 dB
PL13 25.00 dB
PL2W 12.10000038 W
PL12W 0.13988955 W
PL13W 0.19811372 W
SF02 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 202.4563350 MHz
NDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





4-diphenylphosphino-13-N-piperidino[2.2]paracyclophane **23**



31P - Phosphine formation



Current Data Parameters
NAME AP-02 ii 31P 1407
EXPTNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170614
Time 19.04
INSTRUM spect
PROBHD 5 mm PABQ1H
PULPROG zgpp30
TD 65536
SOLVENT CDC13
NS 64
DS 4
SWH 80645.164 Hz
FIDRES 1.230548 Hz
AQ 0.4063232 sec
RG 20642.5
DW 6.200 usec
DE 6.000 usec
TE 293.2 K
D1 2.0000000 sec
D11 0.03000000 sec
TDO 1

NUC1 31P
P1 20.00 usec
PL1 -2.40 dB
PL1W 131.39126587 W
SF01 202.4462122 MHz

NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL1Z 23.37 dB
PL13 20.00 dB
PL2W 12.10000038 W
PL12W 0.13988955 W
PL13W 0.09611372 W
SF02 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 202.4563350 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

