

**Supporting information:**

**The use of (cyclopentadienone)iron tricarbonyl complexes for C-N bond-formation reactions between amines and alcohols.**

**Thomas J. Brown,<sup>a</sup> Madeleine Cumbes,<sup>a</sup> Louis J. Diorazio,<sup>b</sup> Guy J. Clarkson<sup>a</sup> and Martin Wills<sup>a\*</sup>**

- a. Department of Chemistry, The University of Warwick, Coventry, CV4 7AL, UK.  
b. Pharmaceutical Development, AstraZeneca, Silk Road Business Park, Macclesfield, Cheshire, SK10 2NA, UK.

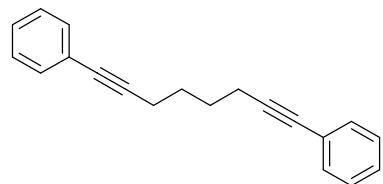
\* Corresponding author; [m.wills@warwick.ac.uk](mailto:m.wills@warwick.ac.uk).

**Contents:**

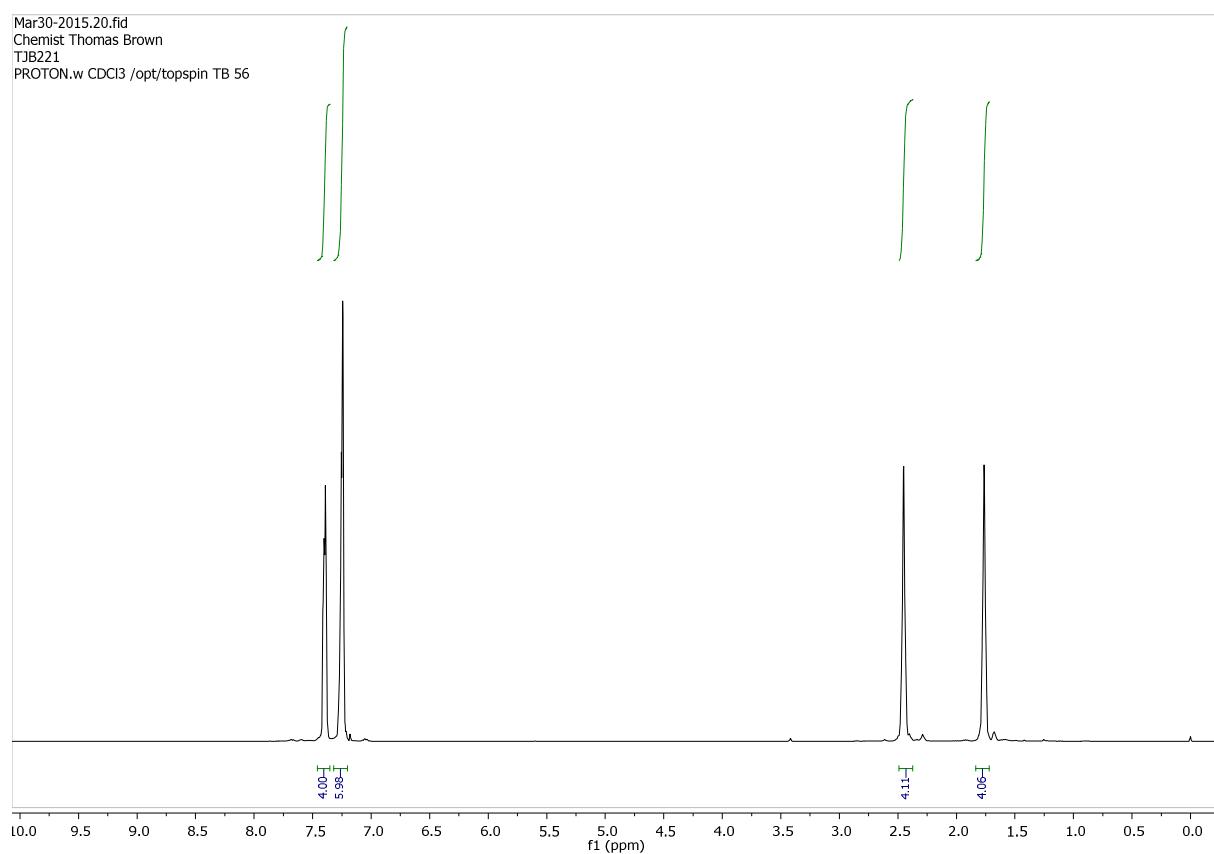
NMR spectra	S2
X-ray Crystallographic structure of <b>6</b>	S117

**1H and 13C NMR Spectra:**

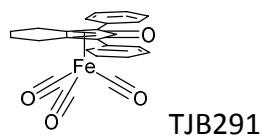
1,8-Diphenylocta-1,7-diyne **10**.



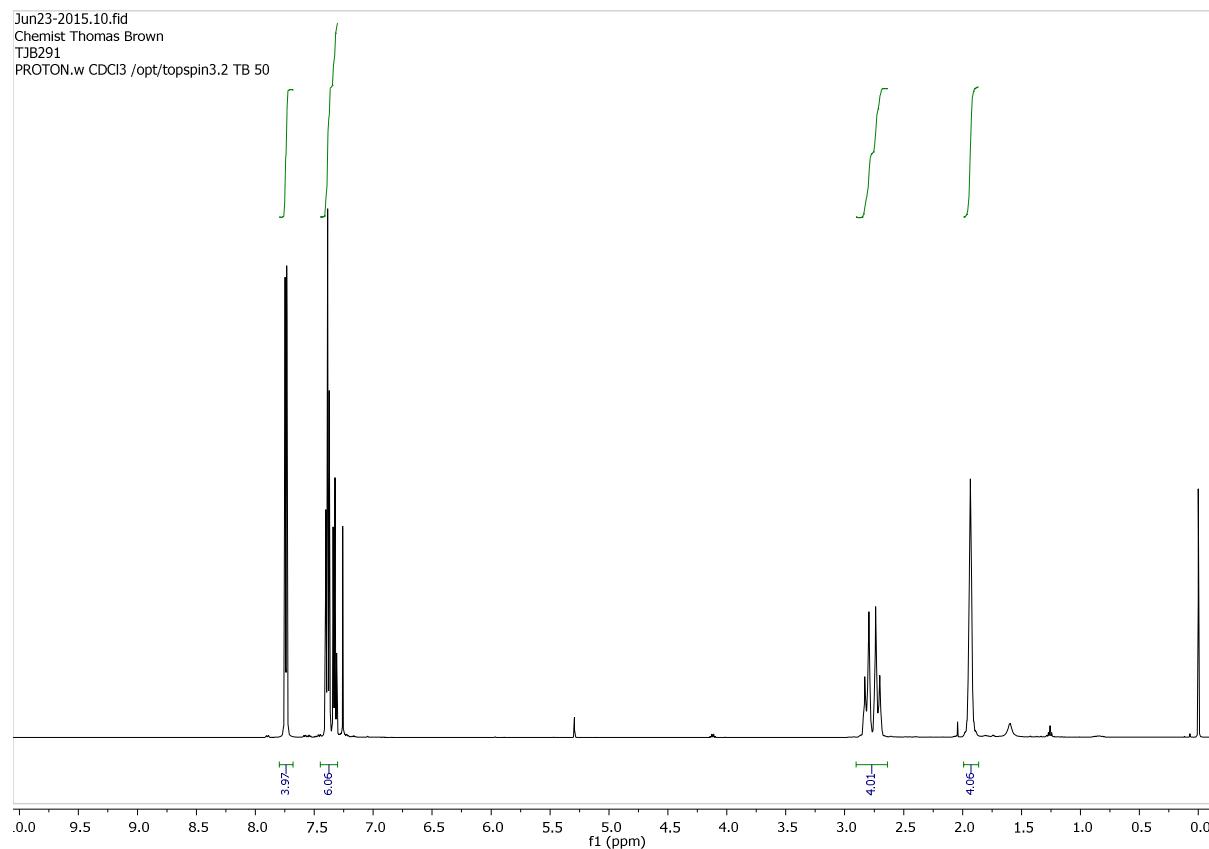
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



Tricarbonyl(1,3-diphenyl-4,5,6,7-tetrahydro-2H-inden-2-one)iron **5**.

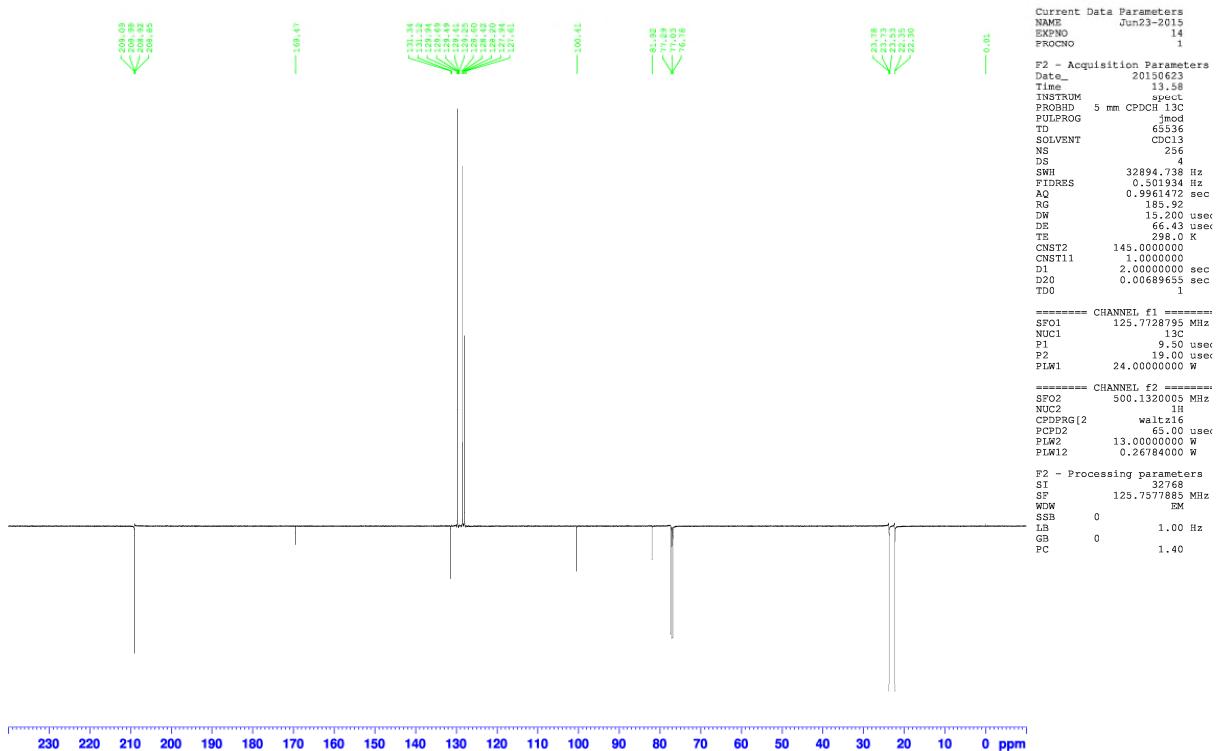


<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)

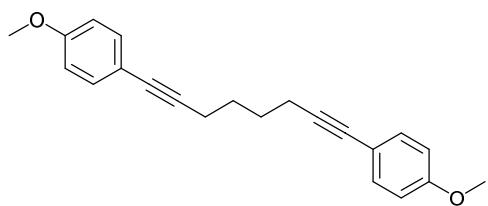


<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)

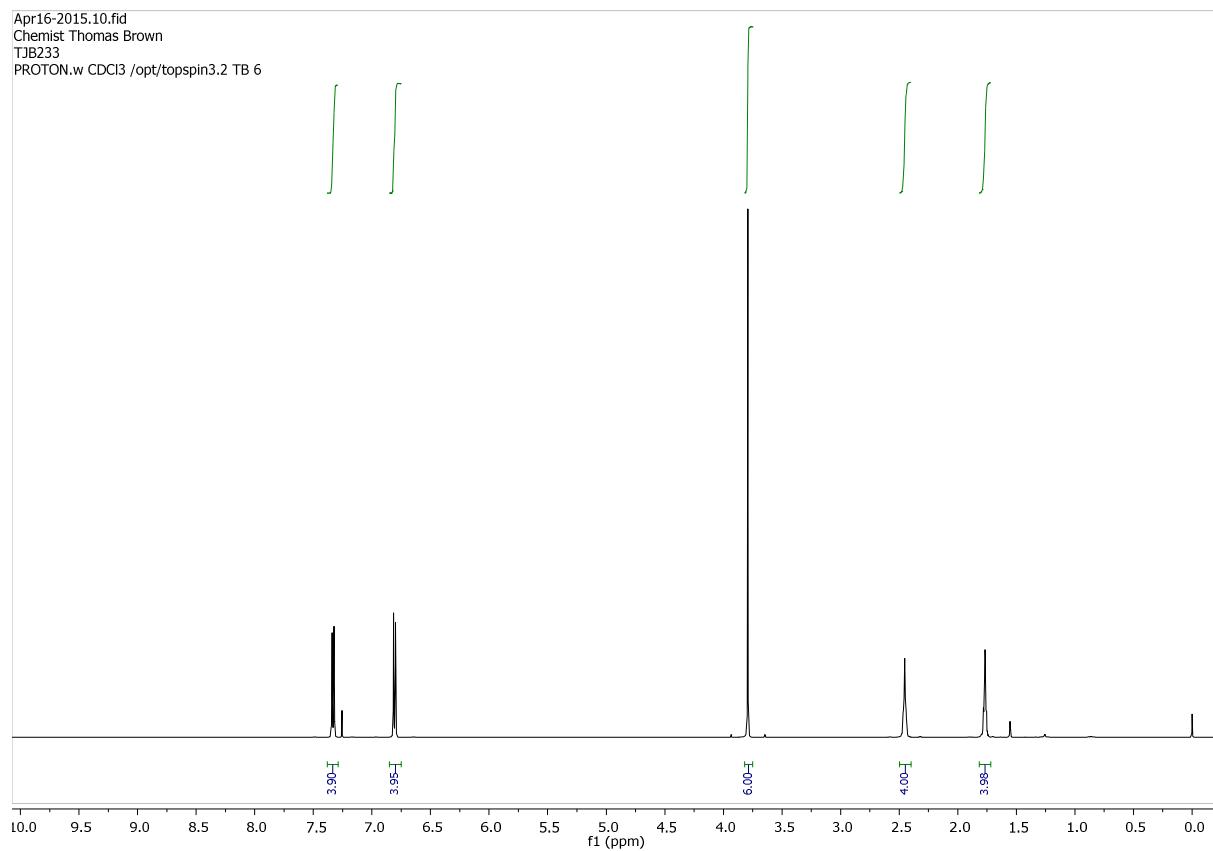
Chemist Thomas Brown  
TJB291  
C13APT.w CDCl3 /opt/topspin3.2 TB 50



1,8-Bis(4-methoxyphenyl)octa-1,7-diyne **11**.



<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>).



<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)

Chemist Thomas Brown  
TJB233  
C13APT.w CDCl<sub>3</sub> /opt/topspin3.2 TB 6

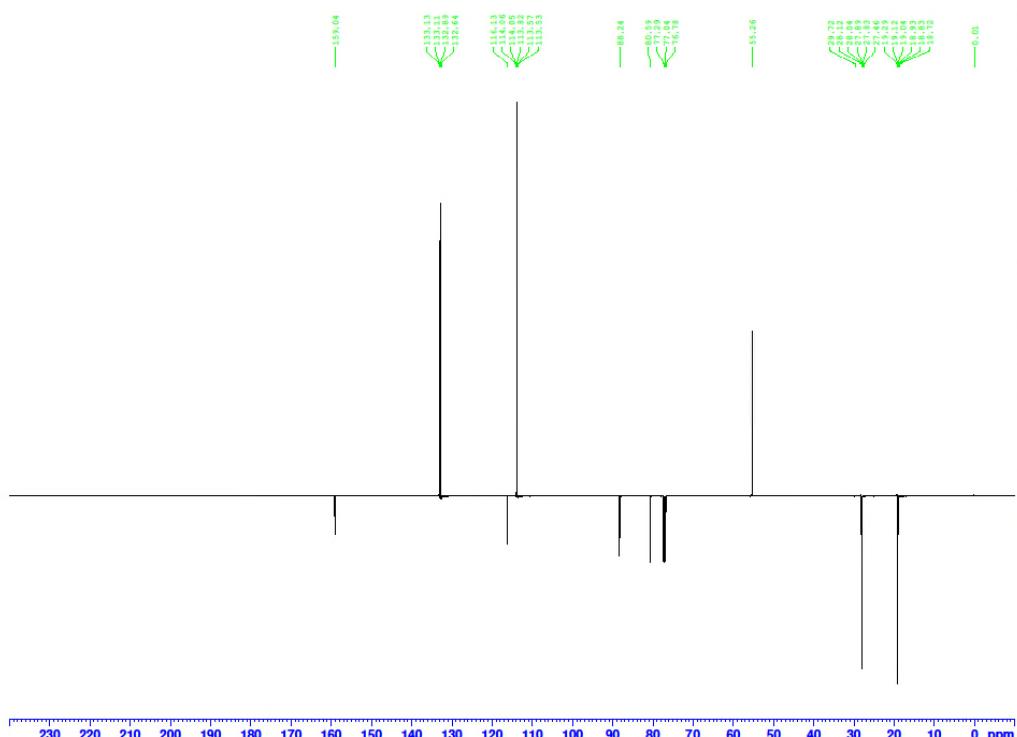


Current Data Parameters  
NAME April6-2015  
EXPNO 13  
PROCNO 1  
  
F2 - Acquisition Parameters  
Date\_ 20150416  
Time\_ 17.09  
INSTRUM spect  
PROBHD 5 mm CPDMC 13C  
PULPROG 300d  
TD 65536  
SOLVENT CDCl<sub>3</sub>  
NS 512  
DS 4  
SWH 32894.738 Hz  
FIDRES 0.501934 Hz  
AQ 0.9961192 sec  
RG 1.0000000  
DW 15.200 usec  
DE 66.43 usec  
TE 298.0 K  
CNSST2 145.000000  
CNSST1 1.0000000  
D1 2.00000000 sec  
D20 0.00689655 sec  
TDO 1

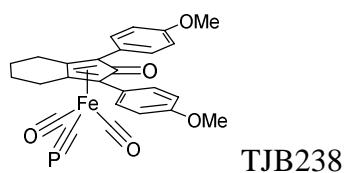
----- CHANNEL f1 -----  
SF01 125.7728795 MHz  
NUC1 1H  
P1 9.50 usec  
P2 19.00 usec  
PLW1 24.0000000 W

----- CHANNEL f2 -----  
SF02 500.1320005 MHz  
NUC2 1H  
CPDPRG[2] Waltz16  
CPDPRG[2] 0.00 usec  
PLW2 13.00000000 W  
PLW12 0.26784000 W

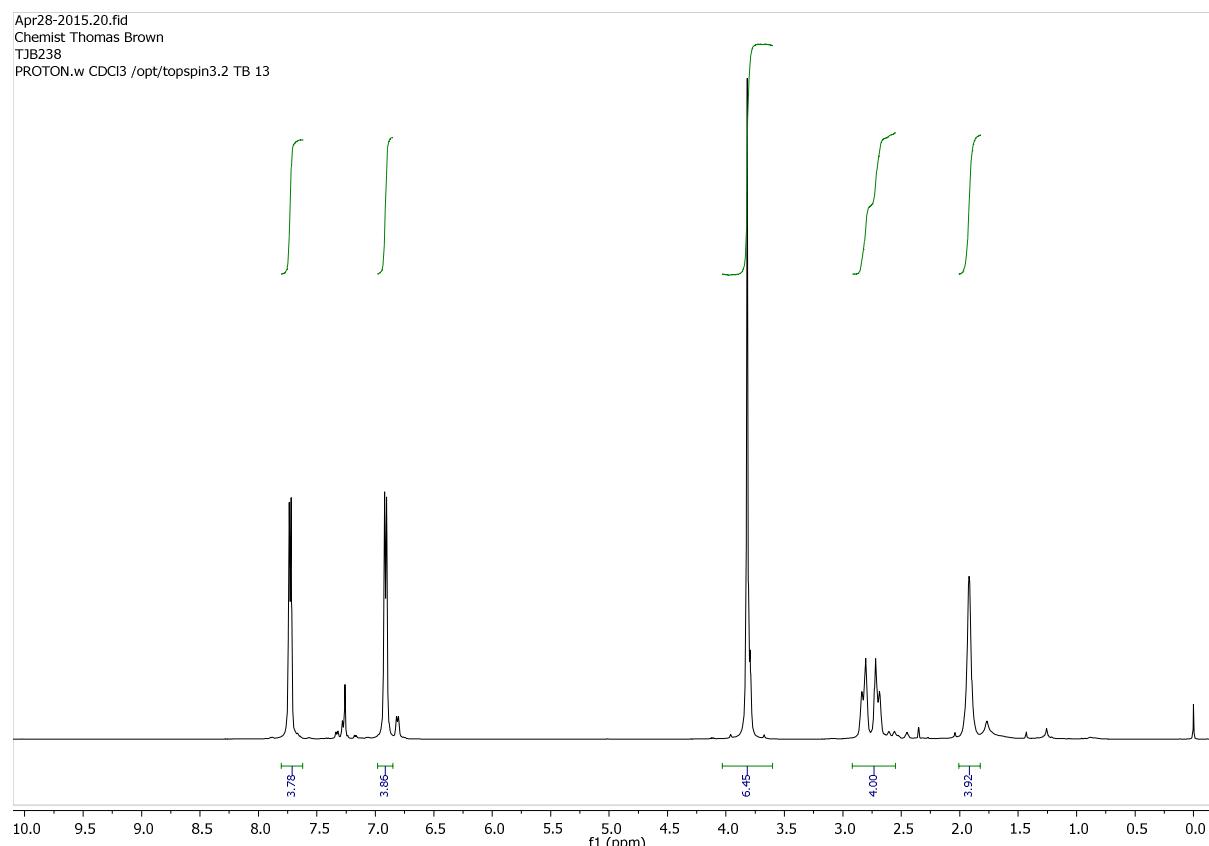
F2 - Processing parameters  
SI 32768  
SF 125.7577885 MHz  
WDW 0  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40



Tricarbonyl(1,3-di(4-methoxyphenyl)-4,5,6,7-tetrahydro-2H-inden-2-one)iron **6**.

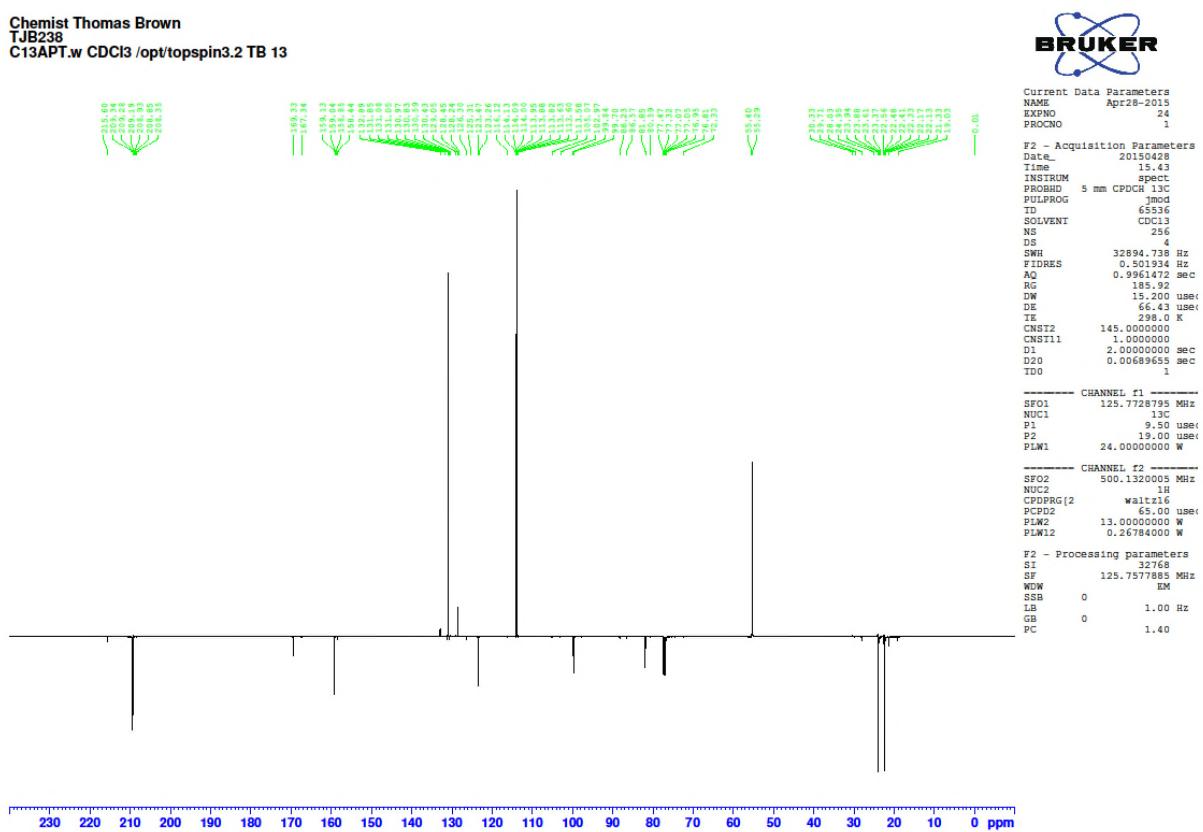


$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ).

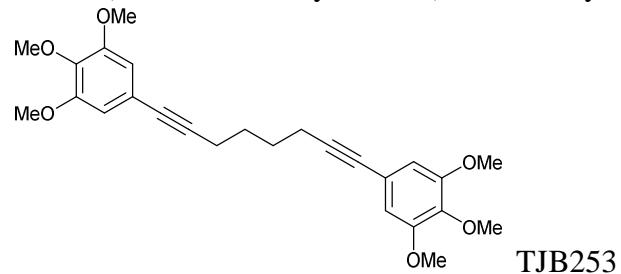


<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)

Chemist Thomas Brown  
TJB238  
C13APT.w CDCl<sub>3</sub> /opt/topspin3.2 TB 13

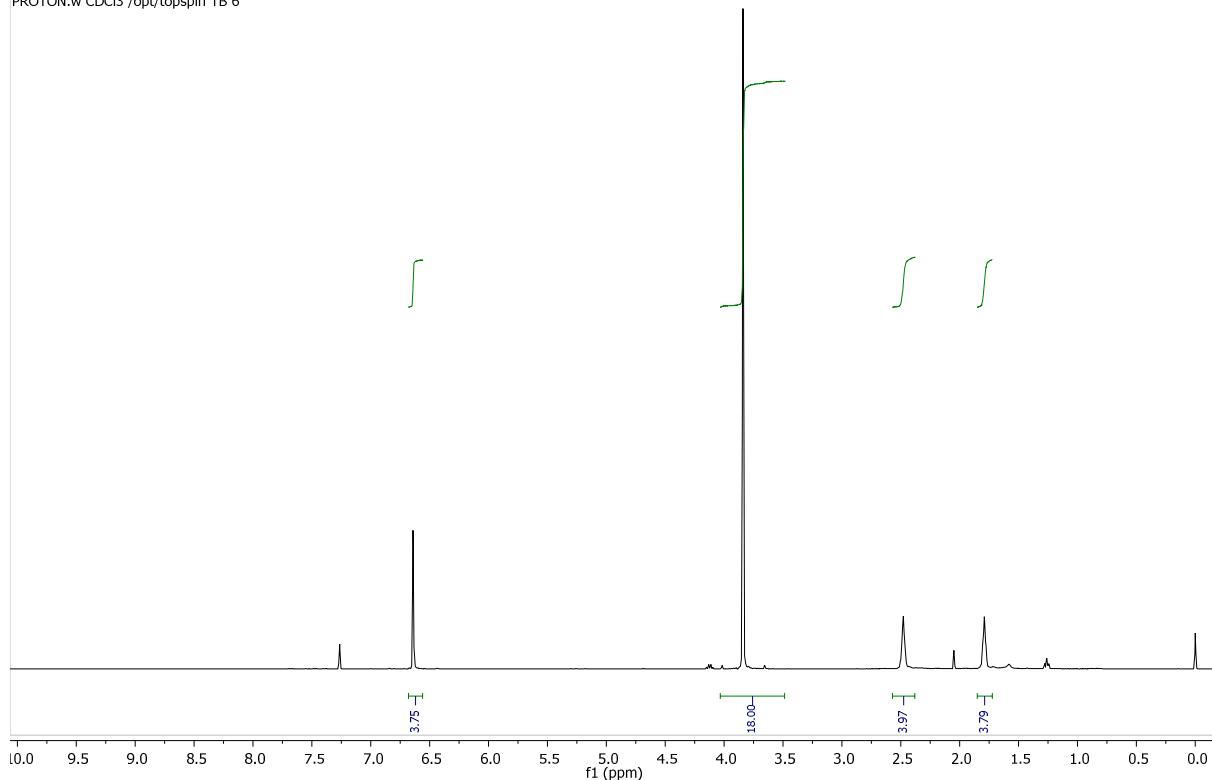


1,8-Bis(3,4,5-trimethoxybenzene)octa-1,7-diyne **12**.



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>).

May11-2015.10.fid  
Chemist Thomas Brown  
TJB253 column fraction  
PROTON.w CDCl<sub>3</sub> /opt/topspin TB 6



<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>).

Chemist Thomas Brown  
TJB trimethoxy precursor  
C13deptql.w CDCl<sub>3</sub> /opt/topspin TB 23

NAME: July29-2015  
EXPTID: 15  
PRFCNO: 1

F2 - Acquisition Parameters

Data\_2D: 20150729

Time: 14:14

INSTRUM: spect

PROBOD: 5 mm Dual ZQ

PROBPG: deptqc\_135

TD: 65536

SWEEP: 1000000

NS: 32

DS: 4

SW0: 23980.814 Hz

TDRES: 0.364795 sec

RG: 16384

DW: 100.00 us

DE: 6.00 us

TE: 6.00 K

CNTS2: 145.0000000

D1: 4.00000000 sec

D2: 0.00000000 sec

D11: 0.03000000 sec

DETA: 0.00001000 sec

SWP01: CHANNEL 13C

P1: 15.05 us

P2: 1.00 us

P1L1: 0.00 dR

SWP01: 100.6014138 MHz

SWP02: CHANNEL 11

CPDPG2: \*1111216

NUC2: 1H

P3: 11.00 us

F4: 22.50 us

CPDPG2: 0.00 us

PL2: 0.00 dB

P12: 1.00 us

SWP02: 400.0000000 MHz

F2 - Processing parameters

S1: 32768

SF: 100.5913138 MHz

WM: 0

SSB: 0

LB: 2.00 Hz

GB: 0

TC: 1.40

SWP01: 100.6014138 MHz

SWP02: 100.5913138 MHz

SWP03: 100.5913138 MHz

SWP04: 100.5913138 MHz

SWP05: 100.5913138 MHz

SWP06: 100.5913138 MHz

SWP07: 100.5913138 MHz

SWP08: 100.5913138 MHz

SWP09: 100.5913138 MHz

SWP10: 100.5913138 MHz

SWP11: 100.5913138 MHz

SWP12: 100.5913138 MHz

SWP13: 100.5913138 MHz

SWP14: 100.5913138 MHz

SWP15: 100.5913138 MHz

SWP16: 100.5913138 MHz

SWP17: 100.5913138 MHz

SWP18: 100.5913138 MHz

SWP19: 100.5913138 MHz

SWP20: 100.5913138 MHz

SWP21: 100.5913138 MHz

SWP22: 100.5913138 MHz

SWP23: 100.5913138 MHz

SWP24: 100.5913138 MHz

SWP25: 100.5913138 MHz

SWP26: 100.5913138 MHz

SWP27: 100.5913138 MHz

SWP28: 100.5913138 MHz

SWP29: 100.5913138 MHz

SWP30: 100.5913138 MHz

SWP31: 100.5913138 MHz

SWP32: 100.5913138 MHz

SWP33: 100.5913138 MHz

SWP34: 100.5913138 MHz

SWP35: 100.5913138 MHz

SWP36: 100.5913138 MHz

SWP37: 100.5913138 MHz

SWP38: 100.5913138 MHz

SWP39: 100.5913138 MHz

SWP40: 100.5913138 MHz

SWP41: 100.5913138 MHz

SWP42: 100.5913138 MHz

SWP43: 100.5913138 MHz

SWP44: 100.5913138 MHz

SWP45: 100.5913138 MHz

SWP46: 100.5913138 MHz

SWP47: 100.5913138 MHz

SWP48: 100.5913138 MHz

SWP49: 100.5913138 MHz

SWP50: 100.5913138 MHz

SWP51: 100.5913138 MHz

SWP52: 100.5913138 MHz

SWP53: 100.5913138 MHz

SWP54: 100.5913138 MHz

SWP55: 100.5913138 MHz

SWP56: 100.5913138 MHz

SWP57: 100.5913138 MHz

SWP58: 100.5913138 MHz

SWP59: 100.5913138 MHz

SWP60: 100.5913138 MHz

SWP61: 100.5913138 MHz

SWP62: 100.5913138 MHz

SWP63: 100.5913138 MHz

SWP64: 100.5913138 MHz

SWP65: 100.5913138 MHz

SWP66: 100.5913138 MHz

SWP67: 100.5913138 MHz

SWP68: 100.5913138 MHz

SWP69: 100.5913138 MHz

SWP70: 100.5913138 MHz

SWP71: 100.5913138 MHz

SWP72: 100.5913138 MHz

SWP73: 100.5913138 MHz

SWP74: 100.5913138 MHz

SWP75: 100.5913138 MHz

SWP76: 100.5913138 MHz

SWP77: 100.5913138 MHz

SWP78: 100.5913138 MHz

SWP79: 100.5913138 MHz

SWP80: 100.5913138 MHz

SWP81: 100.5913138 MHz

SWP82: 100.5913138 MHz

SWP83: 100.5913138 MHz

SWP84: 100.5913138 MHz

SWP85: 100.5913138 MHz

SWP86: 100.5913138 MHz

SWP87: 100.5913138 MHz

SWP88: 100.5913138 MHz

SWP89: 100.5913138 MHz

SWP90: 100.5913138 MHz

SWP91: 100.5913138 MHz

SWP92: 100.5913138 MHz

SWP93: 100.5913138 MHz

SWP94: 100.5913138 MHz

SWP95: 100.5913138 MHz

SWP96: 100.5913138 MHz

SWP97: 100.5913138 MHz

SWP98: 100.5913138 MHz

SWP99: 100.5913138 MHz

SWP100: 100.5913138 MHz

SWP101: 100.5913138 MHz

SWP102: 100.5913138 MHz

SWP103: 100.5913138 MHz

SWP104: 100.5913138 MHz

SWP105: 100.5913138 MHz

SWP106: 100.5913138 MHz

SWP107: 100.5913138 MHz

SWP108: 100.5913138 MHz

SWP109: 100.5913138 MHz

SWP110: 100.5913138 MHz

SWP111: 100.5913138 MHz

SWP112: 100.5913138 MHz

SWP113: 100.5913138 MHz

SWP114: 100.5913138 MHz

SWP115: 100.5913138 MHz

SWP116: 100.5913138 MHz

SWP117: 100.5913138 MHz

SWP118: 100.5913138 MHz

SWP119: 100.5913138 MHz

SWP120: 100.5913138 MHz

SWP121: 100.5913138 MHz

SWP122: 100.5913138 MHz

SWP123: 100.5913138 MHz

SWP124: 100.5913138 MHz

SWP125: 100.5913138 MHz

SWP126: 100.5913138 MHz

SWP127: 100.5913138 MHz

SWP128: 100.5913138 MHz

SWP129: 100.5913138 MHz

SWP130: 100.5913138 MHz

SWP131: 100.5913138 MHz

SWP132: 100.5913138 MHz

SWP133: 100.5913138 MHz

SWP134: 100.5913138 MHz

SWP135: 100.5913138 MHz

SWP136: 100.5913138 MHz

SWP137: 100.5913138 MHz

SWP138: 100.5913138 MHz

SWP139: 100.5913138 MHz

SWP140: 100.5913138 MHz

SWP141: 100.5913138 MHz

SWP142: 100.5913138 MHz

SWP143: 100.5913138 MHz

SWP144: 100.5913138 MHz

SWP145: 100.5913138 MHz

SWP146: 100.5913138 MHz

SWP147: 100.5913138 MHz

SWP148: 100.5913138 MHz

SWP149: 100.5913138 MHz

SWP150: 100.5913138 MHz

SWP151: 100.5913138 MHz

SWP152: 100.5913138 MHz

SWP153: 100.5913138 MHz

SWP154: 100.5913138 MHz

SWP155: 100.5913138 MHz

SWP156: 100.5913138 MHz

SWP157: 100.5913138 MHz

SWP158: 100.5913138 MHz

SWP159: 100.5913138 MHz

SWP160: 100.5913138 MHz

SWP161: 100.5913138 MHz

SWP162: 100.5913138 MHz

SWP163: 100.5913138 MHz

SWP164: 100.5913138 MHz

SWP165: 100.5913138 MHz

SWP166: 100.5913138 MHz

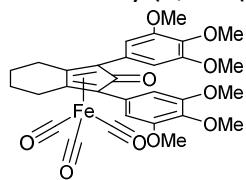
SWP167: 100.5913138 MHz

SWP168: 100.5913138 MHz

SWP169: 100.5913138 MHz

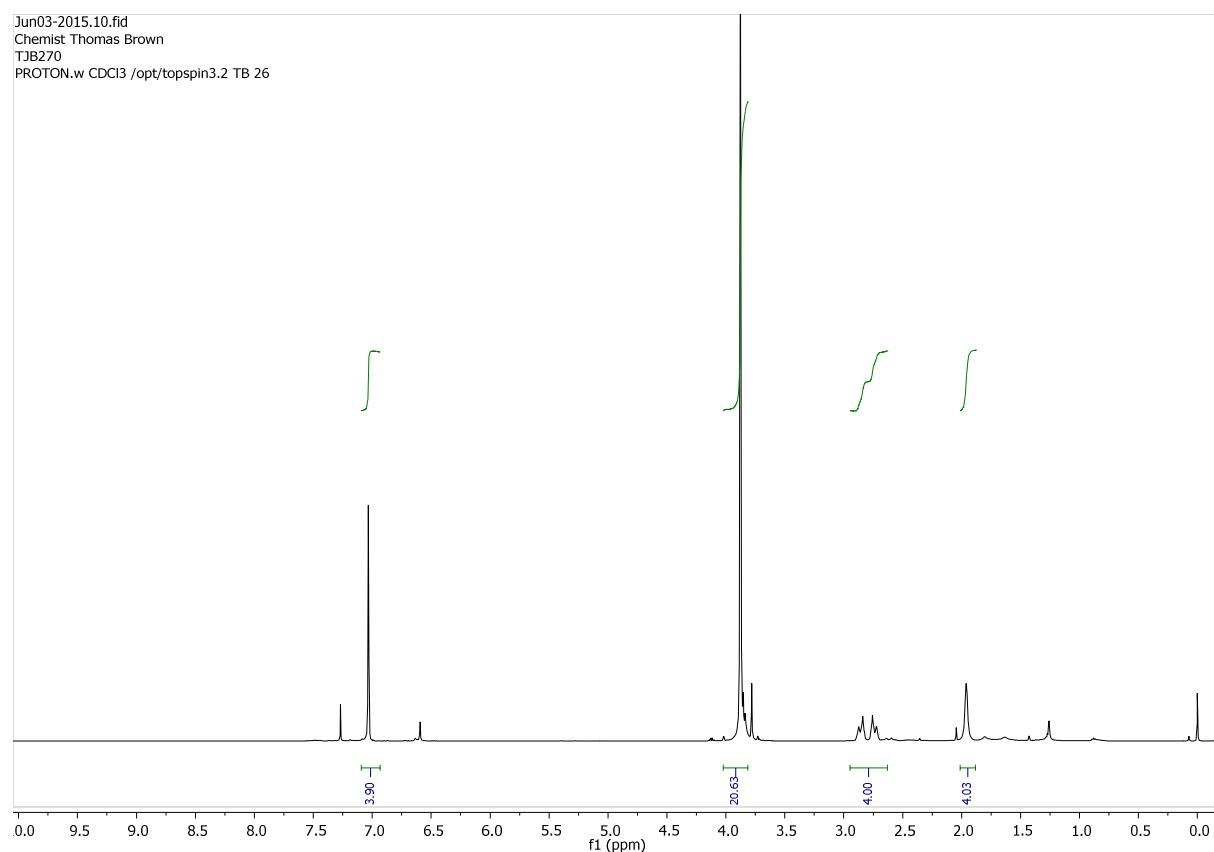
SWP170: 100.5913138 MHz

Tricarbonyl(1,3-di(3,4,5-trimethoxyphenyl)-4,5,6,7-tetrahydro-2H-inden-2-one)iron **7**.



TJB270.

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ).



<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>).

Chemist Thomas Brown  
TJB270  
C13APT.w CDCl<sub>3</sub> /opt/topspin3.2 TB 26

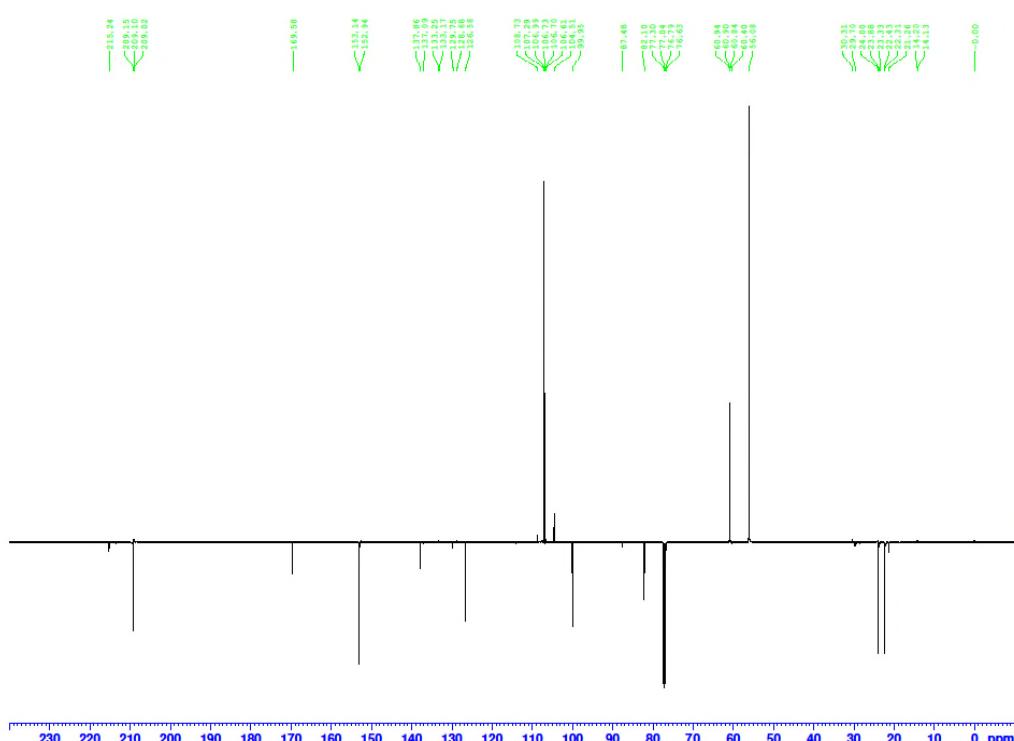


Current Data Parameters  
NAME Jun03-2015  
EXPNO 14  
PROCNO 1  
  
F2 - Acquisition Parameters  
Date 20150603  
Time 16.48  
INSTRUM spect  
PROBHD 5 mm CPDMC 13C  
PULPROG 300d  
TD 65536  
SOLVENT CDCl<sub>3</sub>  
NS 256  
DS 4  
SWH 32894.738 Hz  
FIDRES 0.501934 Hz  
AQ 0.9961192 sec  
RG 1.0000000  
DW 15.200 usec  
DE 66.43 usec  
TE 298.0 K  
CNS1ST2 145.000000  
CNS1T1 1.0000000  
D1 2.00000000 sec  
D20 0.00689655 sec  
TDO 1

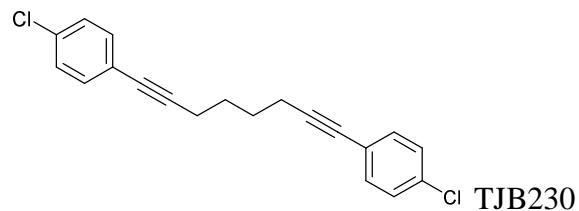
----- CHANNEL f1 -----  
SF01 125.7728795 MHz  
NUC1 13C  
P1 9.00 usec  
P2 19.00 usec  
PLW1 24.0000000 W

----- CHANNEL f2 -----  
SF02 500.1320005 MHz  
NUC2 1H  
CPDPGR2 Waltz16  
PCP02 0.0000000 usec  
PLW2 13.000000000 W  
PLW12 0.267840000 W

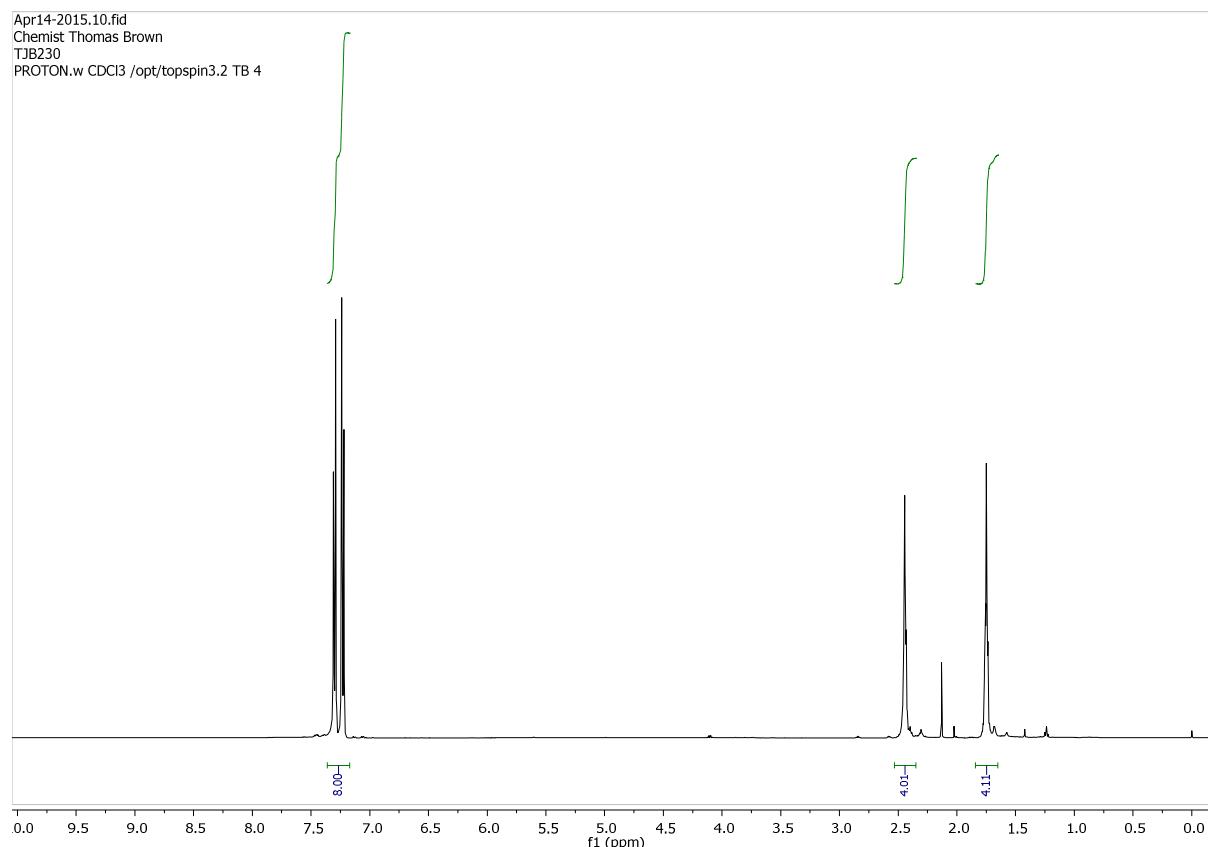
F2 - Processing parameters  
SI 32768  
SF 125.7577885 MHz  
WDW 0  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40



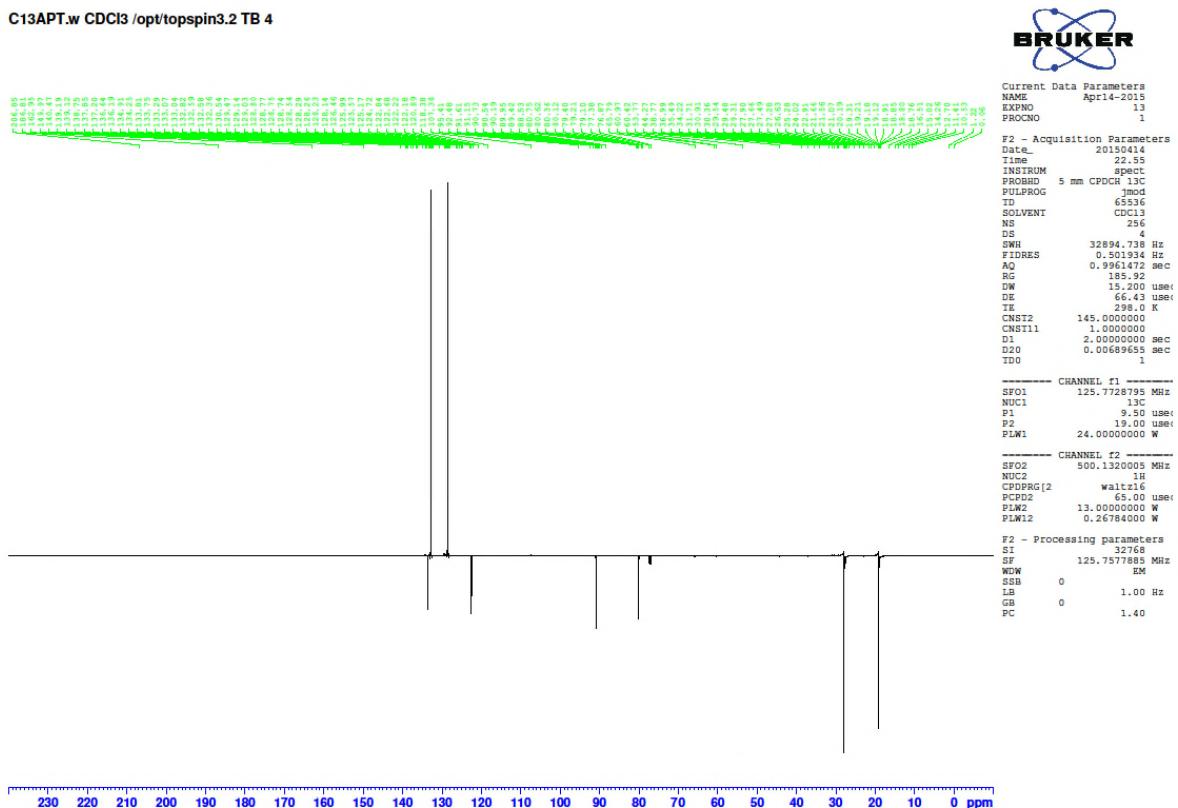
1,8-(4-Chlorophenyl)octa-1,7-diyne **13**.



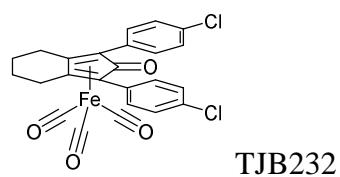
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>).



<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)

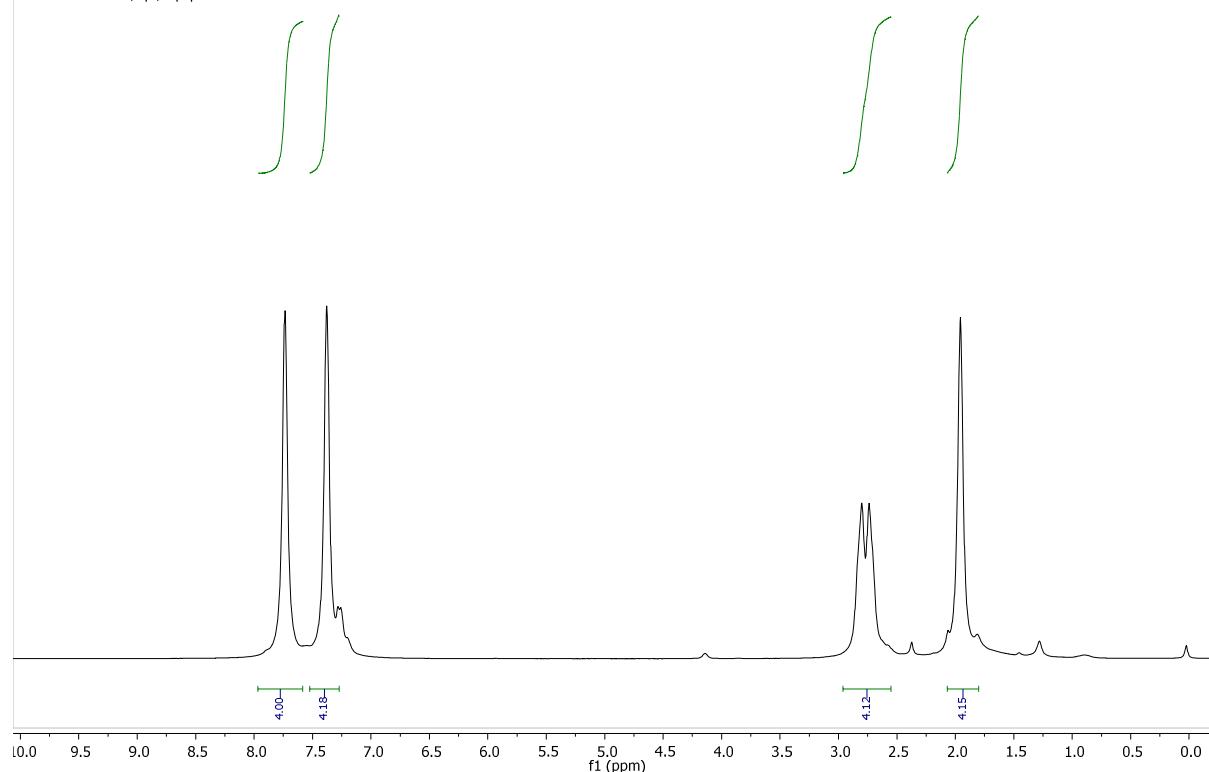


Tricarbonyl(1,3-di(4-chloro)phenyl-4,5,6,7-tetrahydro-2H-inden-2-one)iron **8**.



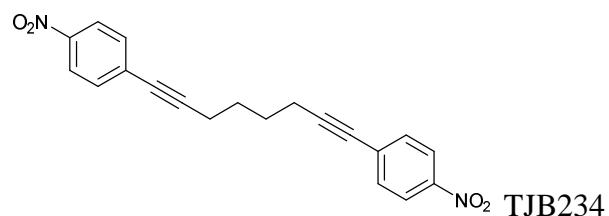
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>).

Apr28-2015.10.fid  
Chemist Thomas Brown  
TJB232  
PROTON.w CDCl<sub>3</sub> /opt/topspin3.2 TB 12

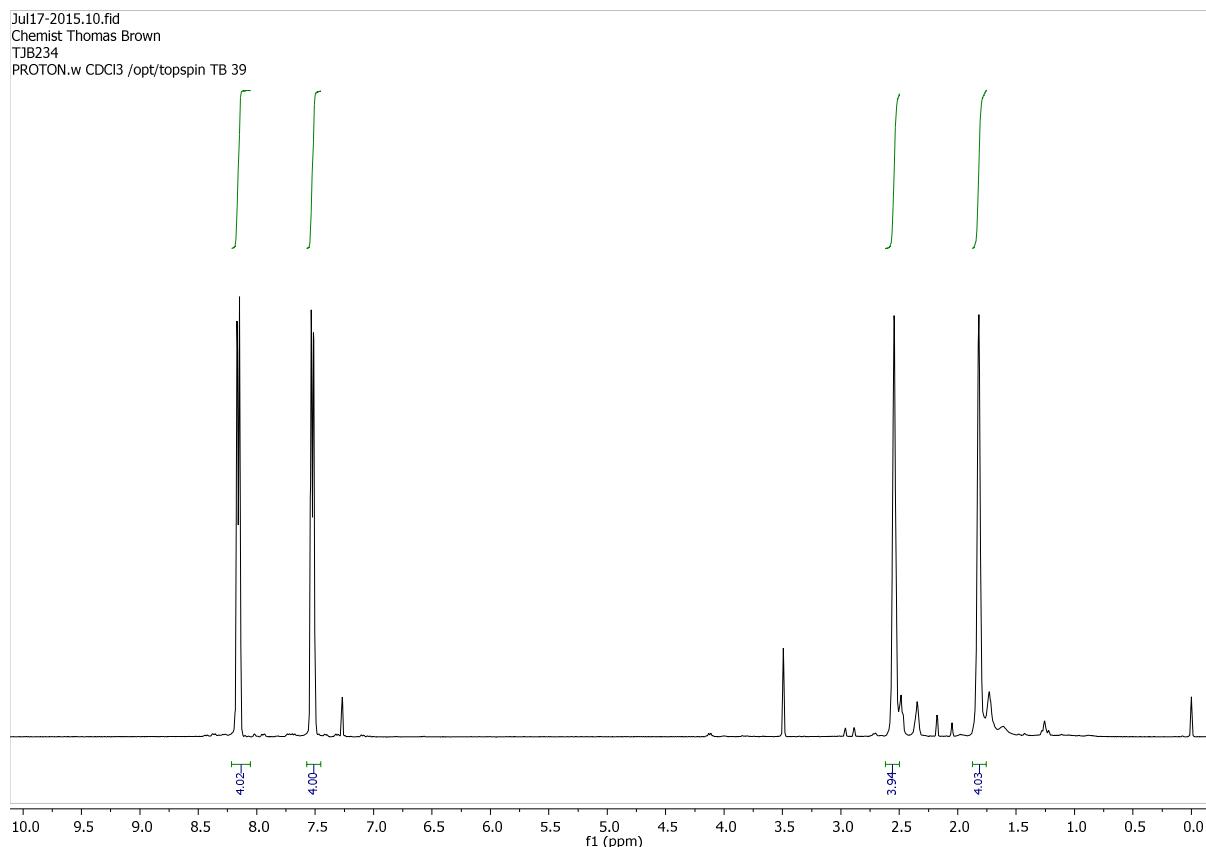




1,8-Bis(4-nitrophenyl)octa-1,7-diyne **14**.



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)

Chemist Thomas Brown

TJB234

C13deptq.w CDCl<sub>3</sub> /opt/topspin TB 39

Current Data Parameters

Name: C13deptq.w Date: July-17-2015

EXPPRO: 12

PRNCNO: 1

F2 - Acquisition Parameters

Date: 2017-07-17

Time: 12.17

INSTRUM: spect

PROBOD: 5 mm Dual T2

PULPROG: deptq\_135

TD: 65536

SOLVENT: CDCl<sub>3</sub>

NS: 128

DS: 4

SWH: 23898.814 Hz

TDZ: 63840000

AQ: 1.3644716 sec

RG: 1.6384

DE: 6.00 usec

TE: 294.00

CNTZ: 145.0000000

D1: 2.00000000 sec

D12: 0.00000000 sec

D11: 0.00000000 sec

DELT1: 0.00001916 sec

NUC1: 13C

P1: 15.05 usec

P2: 30.10 usec

PL1: 90.00 dB

SW01: 100.6014538 MHz

NUC2: 13C

CPDPG2: waltz16

NUC3: 1H

P3: 11.25 usec

PCPD2: 90.00 usec

PL2: 90.00 dB

PSI2: 110.00

SW02: 400.0466002 MHz

F2 - Processing parameters

SI: 32768

SF: 100.3913900 MHz

WID: 1K

SSB: 0

LB: 2.00 Hz

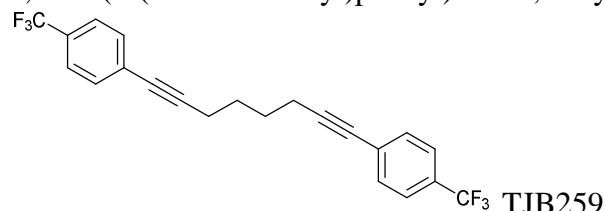
GB: 0

PC: 1.40

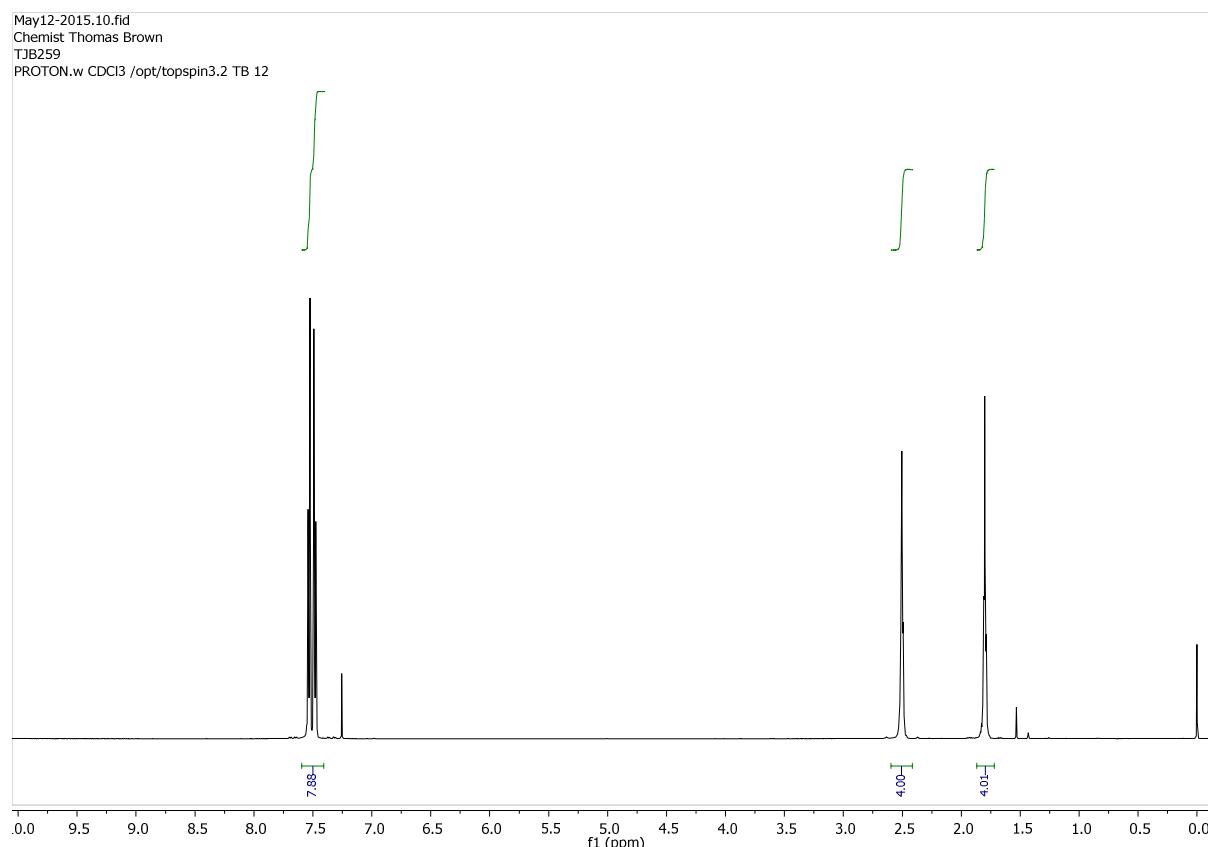


210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

1,8-Bis(4-(trifluoromethyl)phenyl)octa-1,7-diyne **15**.



$^1H$  NMR (500 MHz,  $CDCl_3$ ).



<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)

C13APT.w CDCl<sub>3</sub> /opt/topspin3.2 TB 12

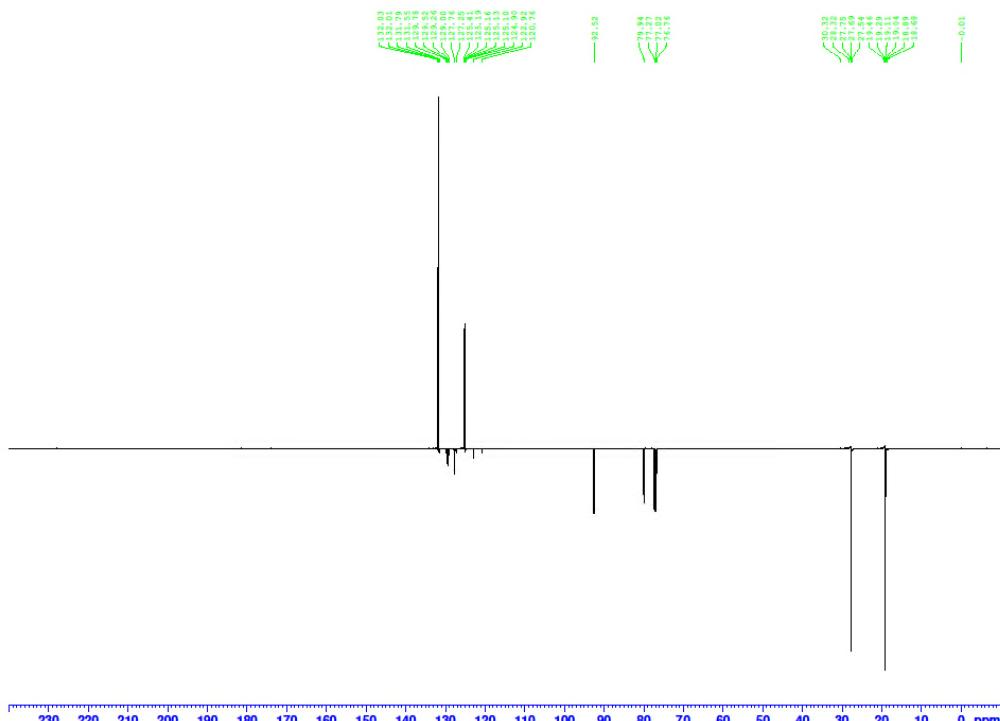


Current Data Parameters  
 NAME May12-2015  
 EXPNO 14  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date 20150512  
 Time 13:00  
 INSTRUM spect  
 PROBHD 5 mm CPDPCH 13C  
 PULPROG jmod  
 TD 6536  
 SWLVENT CDCl<sub>3</sub>  
 NS 256  
 DS 4  
 SW0 32894.738 Hz  
 FIDRES 0.500000 Hz  
 AQ 0.061472 sec  
 RG 185.92  
 DW 15.200 usec  
 DE 66.43 usec  
 TDE 20.0 K  
 CNST2 145.000000  
 CNST11 1.000000  
 D1 2.0000000 sec  
 D9 0.06689655 sec  
 TDD 1

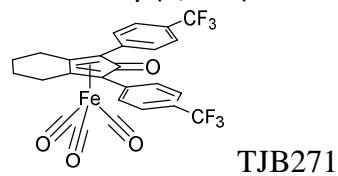
CHANNEL f1  
 SF01 125.7728795 MHz  
 NUC1 <sup>13</sup>C  
 P1 9.50 usec  
 P2 19.00 usec  
 PLW1 24.0000000 W

CHANNEL f2  
 SF02 500.1320000 MHz  
 NUC2 <sup>1</sup>H  
 CPDPG2 waltz16  
 PCPD2 65.00 usec  
 PLW2 13.0000000 W  
 PLW12 0.267840000 W

F2 - Processing parameters  
 SI 32768  
 SF 125.7577885 MHz  
 WM 0 EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

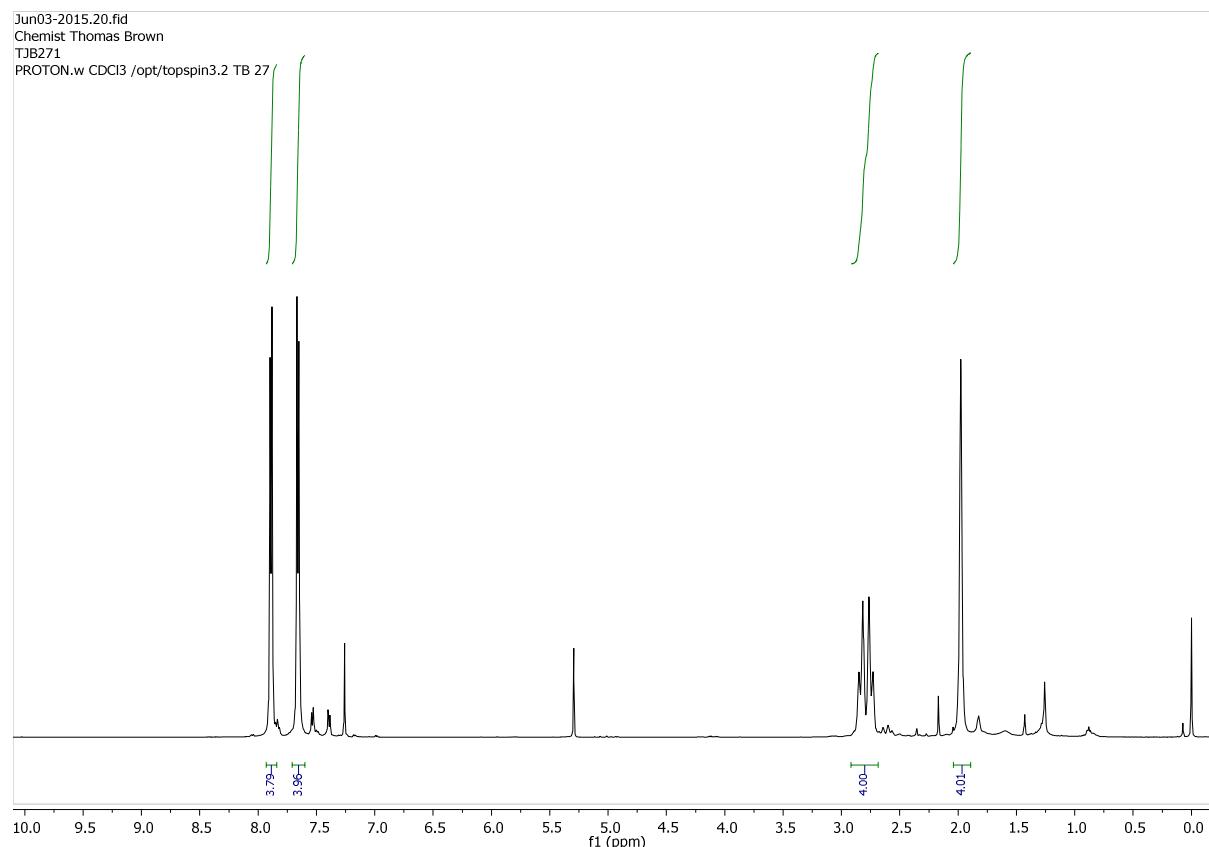


Tricarbonyl(1,3-di(4-trifluoromethylphenyl)-4,5,6,7-tetrahydro-2H-inden-2-one)iron **16**.



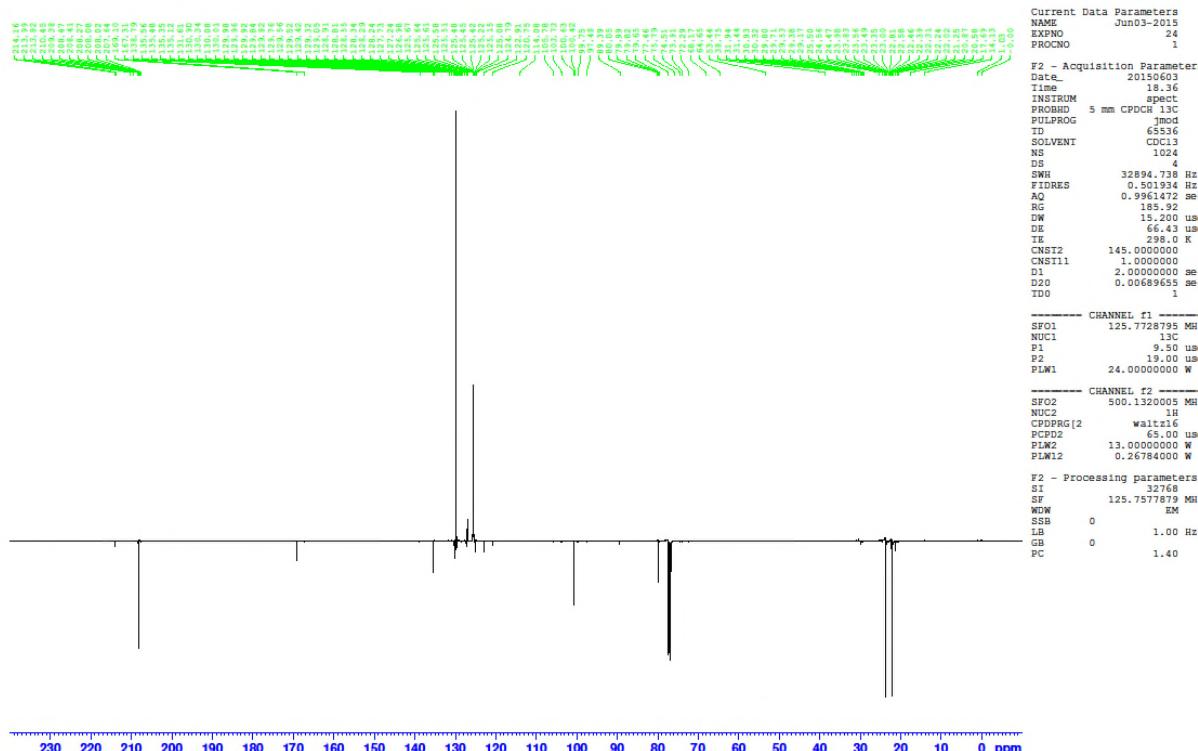
TJB271

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ).

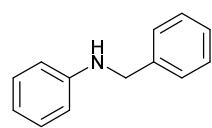


<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)

Chemist Thomas Brown  
TJB271  
C13APT.w CDCl<sub>3</sub> /opt/topspin3.2 TB 27



*N*-Benzylaniline **19.**

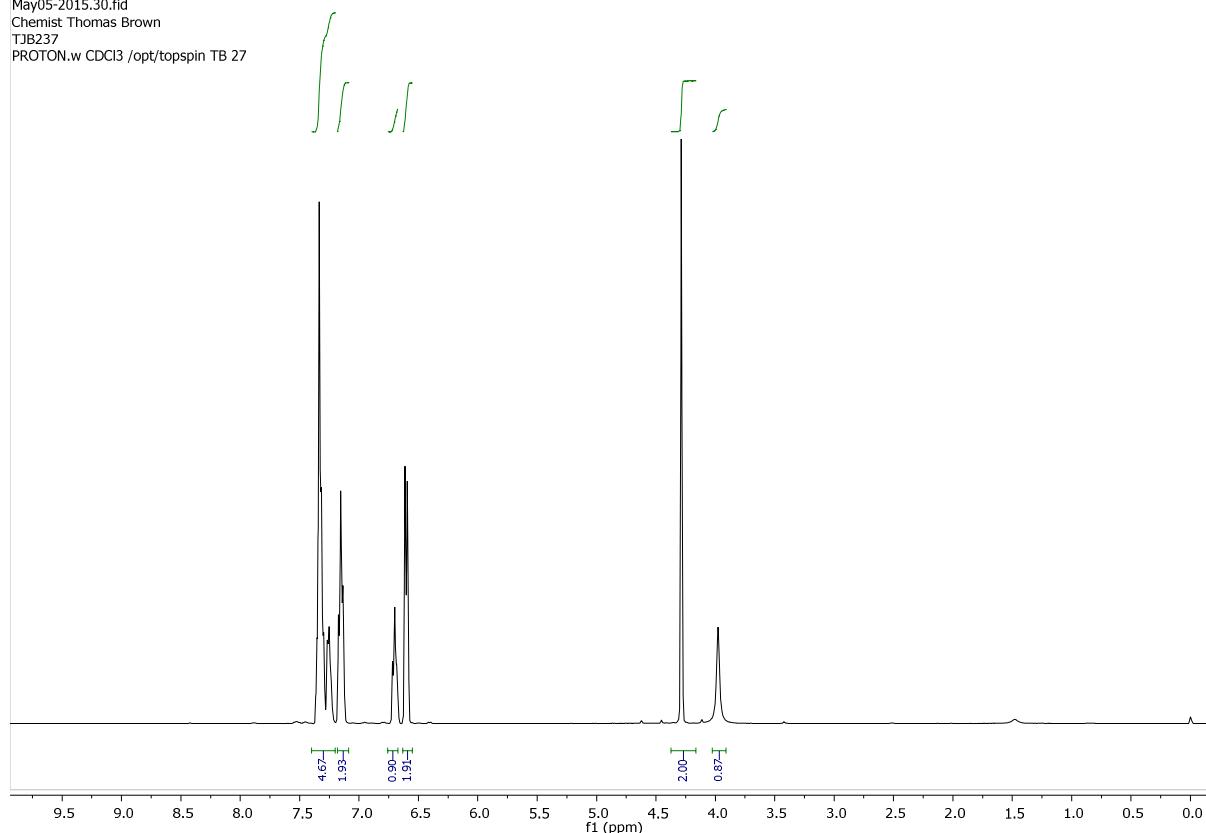


*N*-benzylaniline

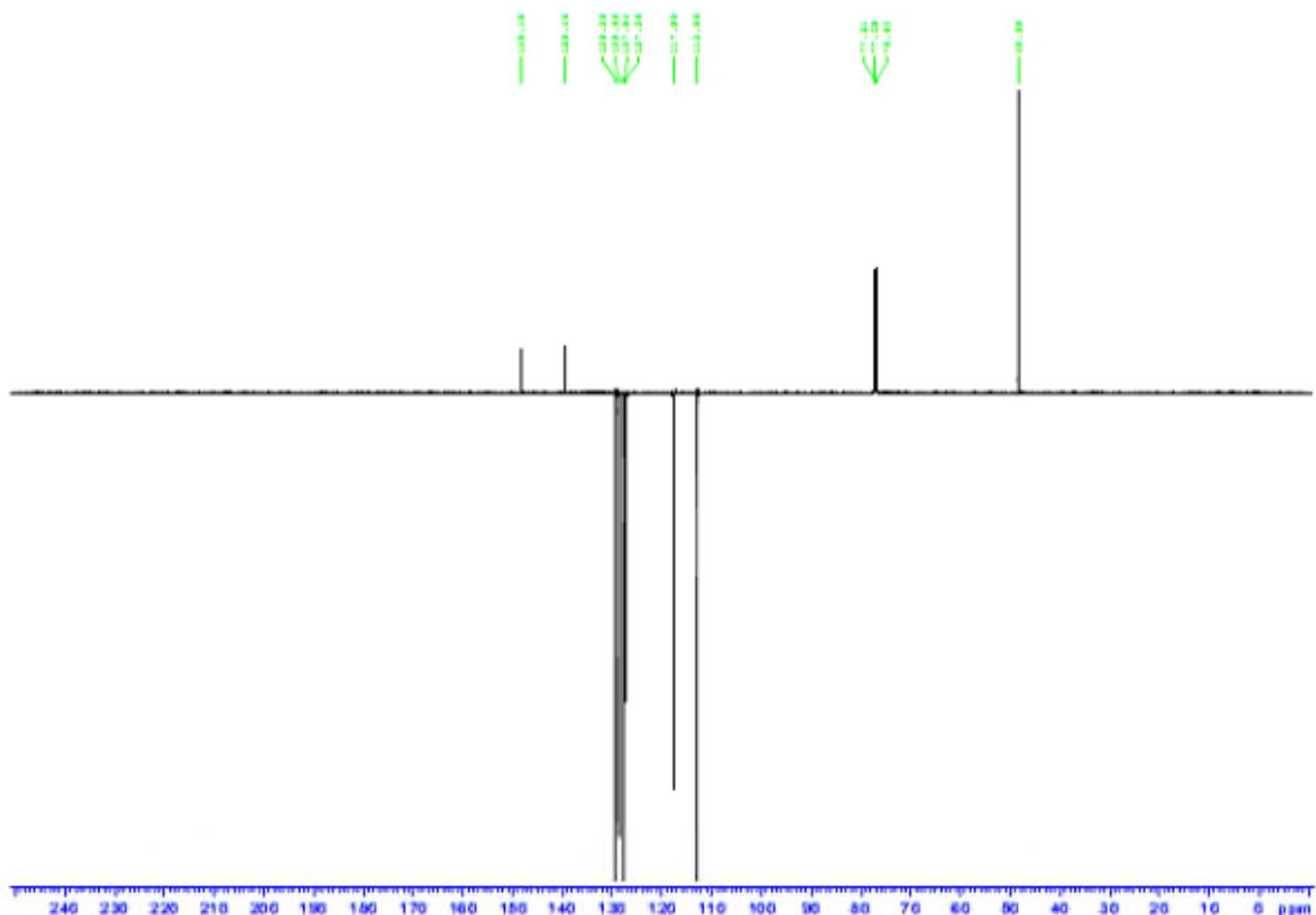
TJB237

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ).

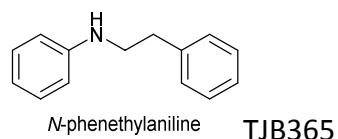
May05-2015.30.fid  
Chemist Thomas Brown  
TJB237  
PROTON.w  $\text{CDCl}_3$  /opt/topspin TB 27



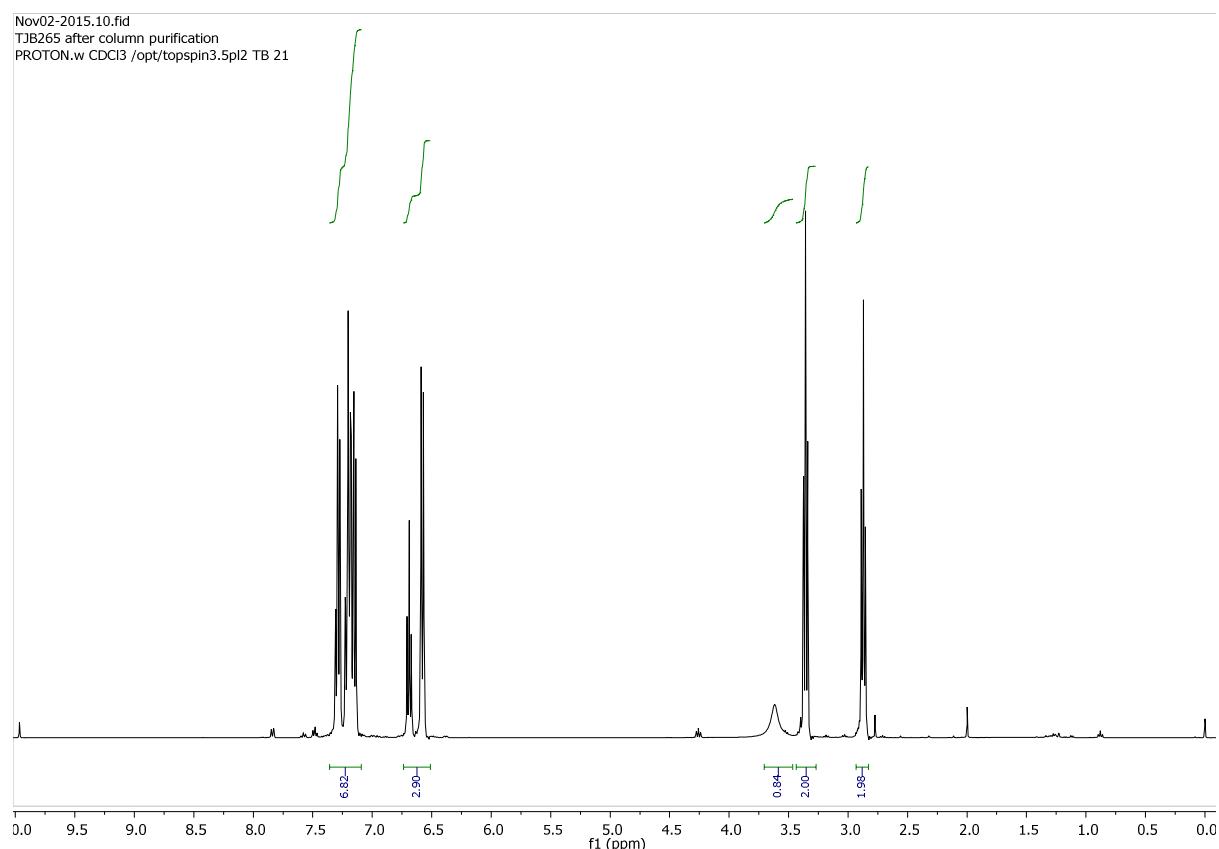
<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>).



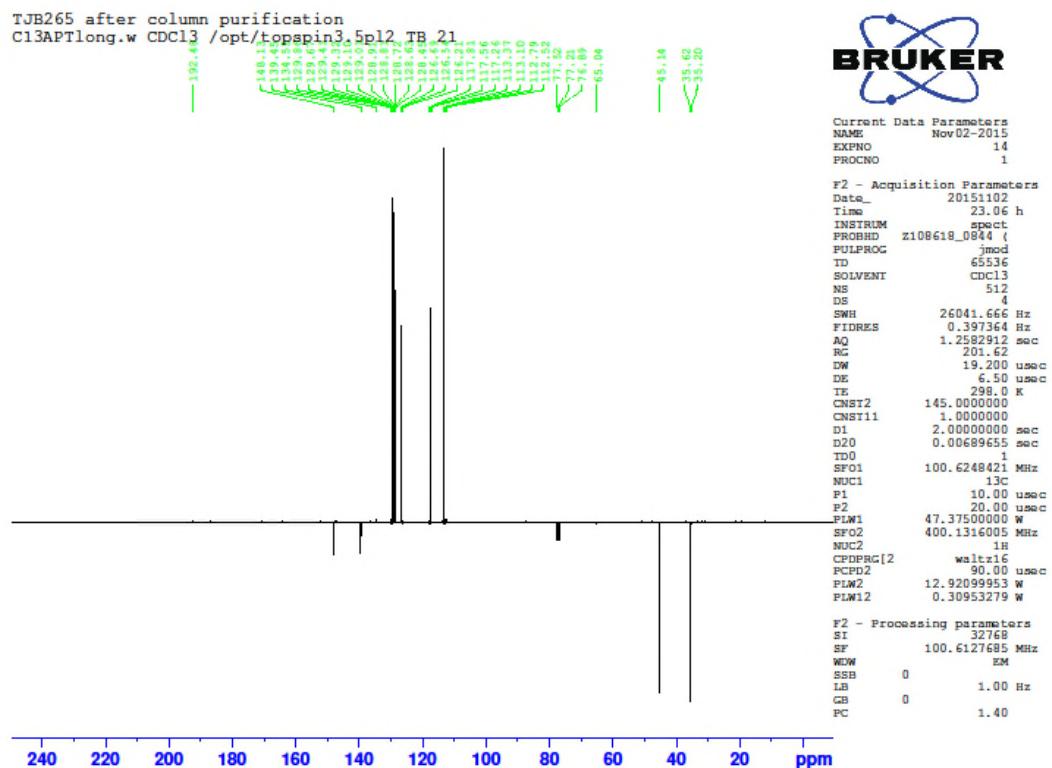
*N*-Phenethylaniline **20**.



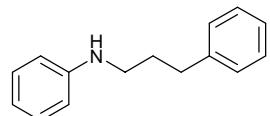
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ).



<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>).

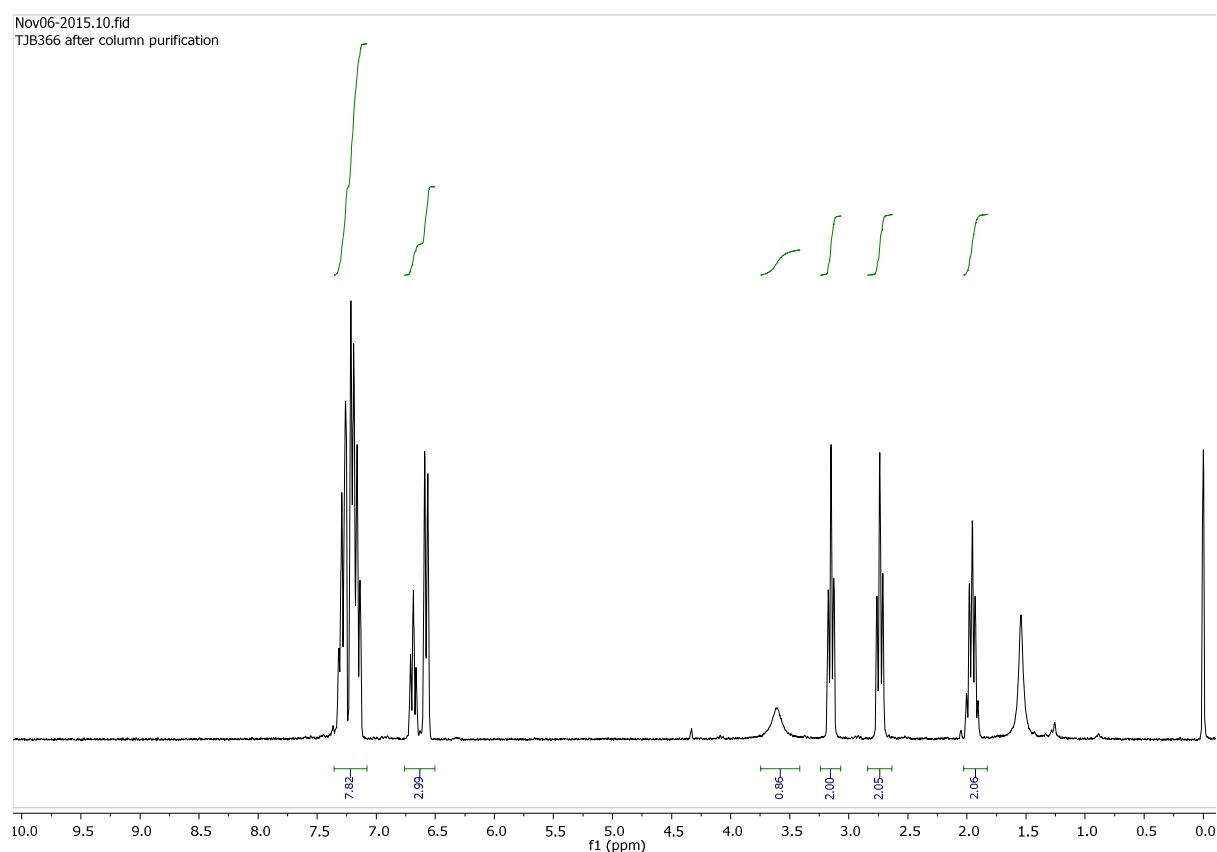


*N*-(3-Phenylpropyl)aniline **21**.

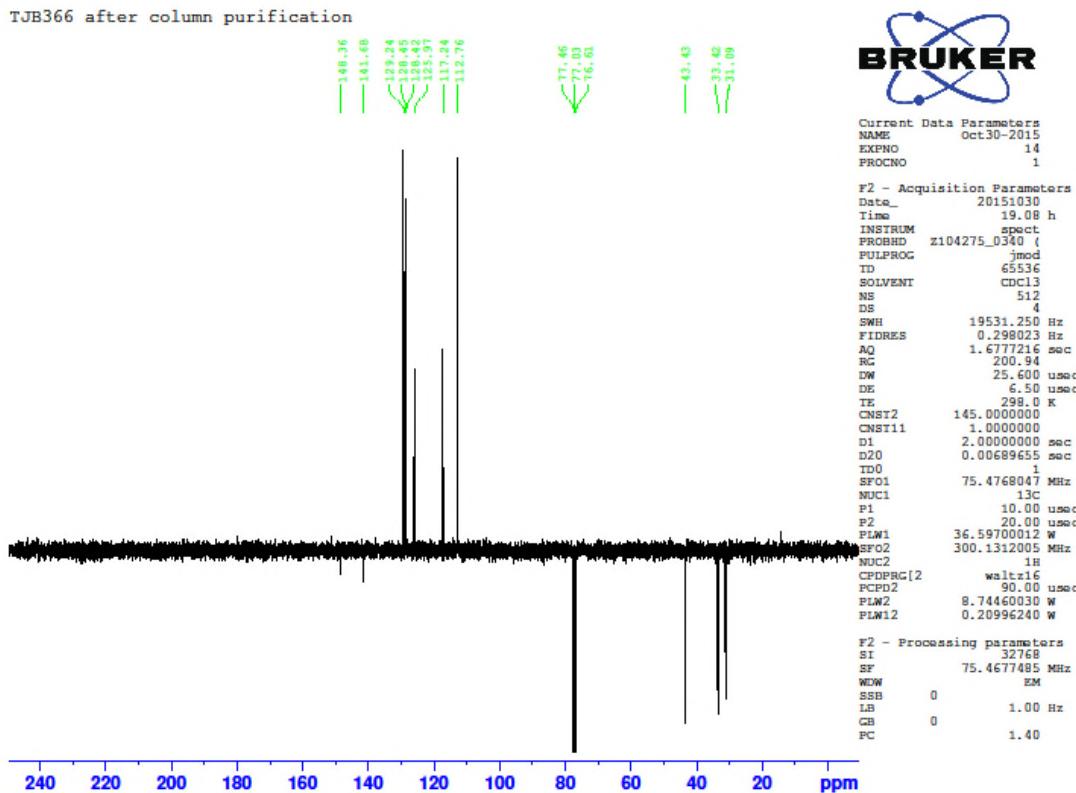


*N*-(3-phenylpropyl)aniline    TJB366.

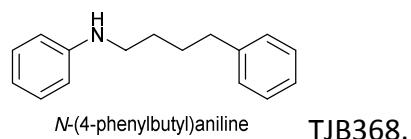
$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ).



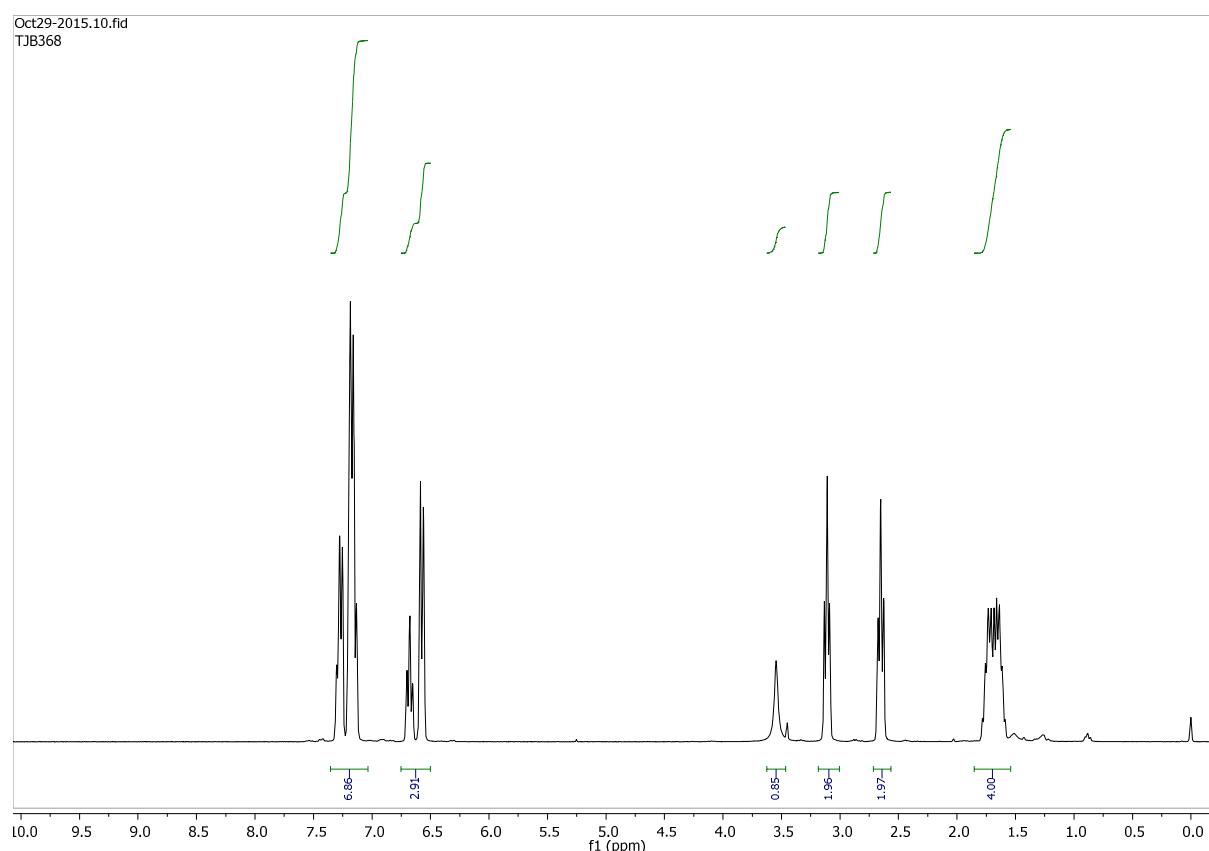
<sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>).



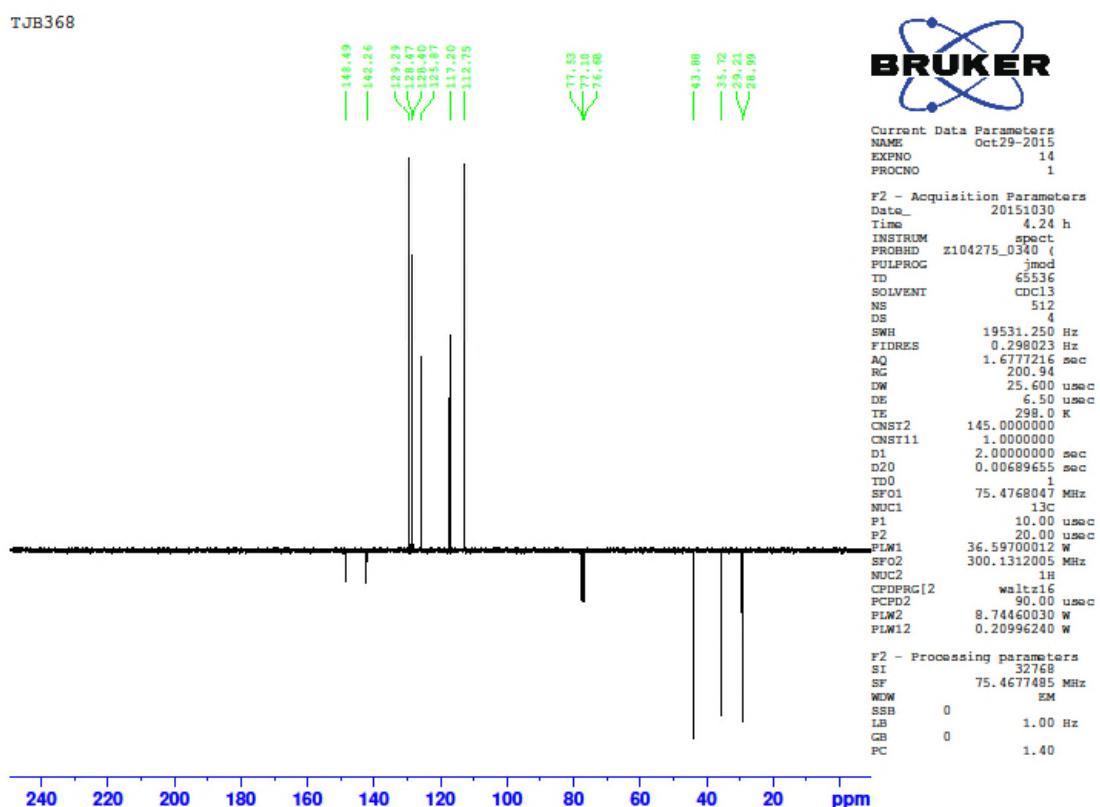
*N*-(4-Phenylbutyl)aniline **22**.



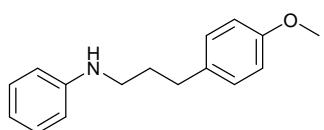
$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ).



<sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>).

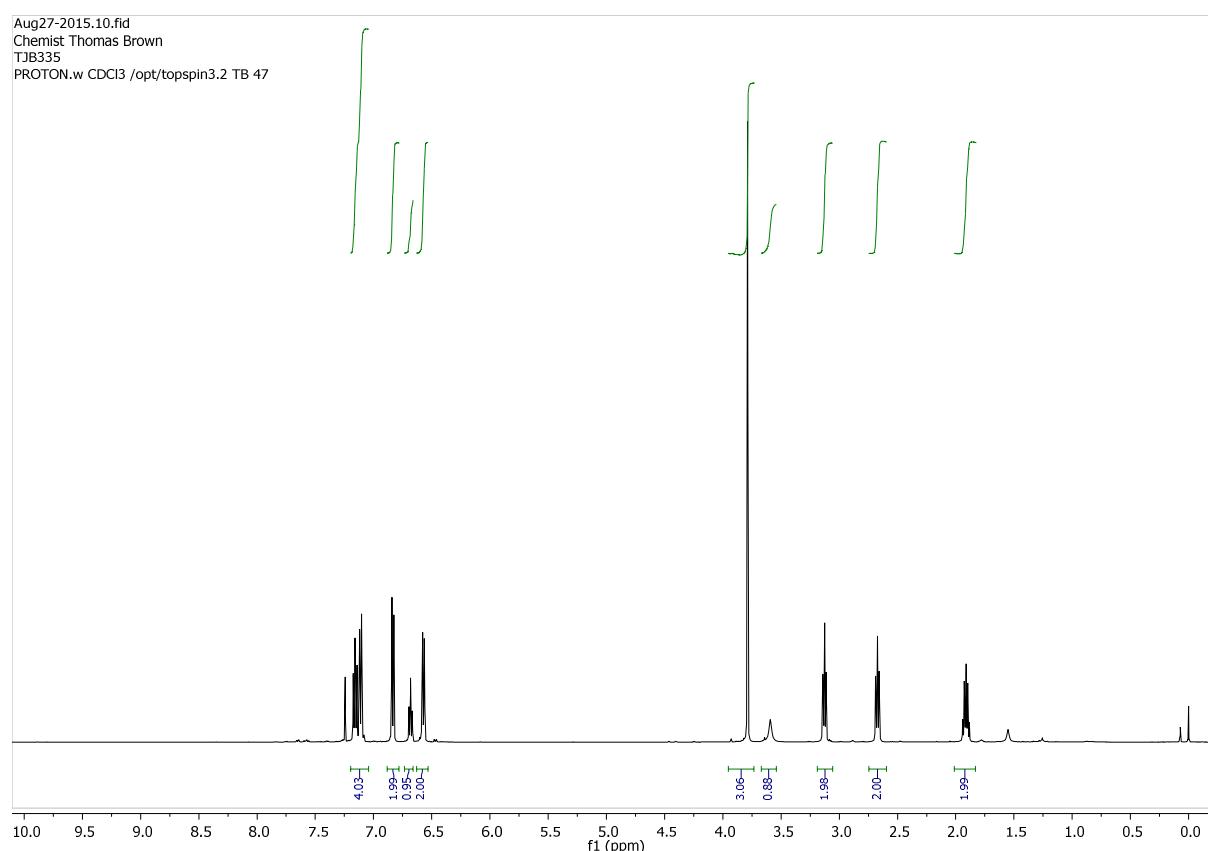


*N*-(3-(4-Methoxyphenyl)propyl)aniline **23**.



*N*-(3-(4-methoxyphenyl)propyl)aniline **TJB335**

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ).

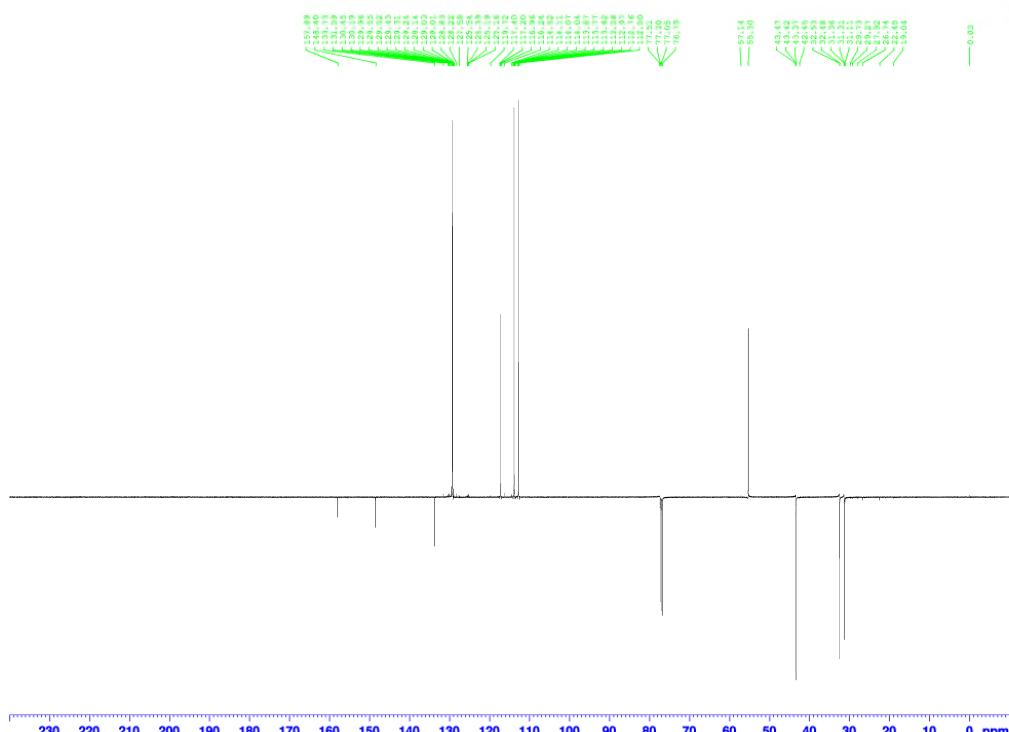


<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>).

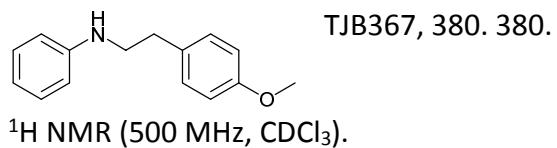
Chemist Thomas Brown  
TJB335  
C13APT.w CDCl<sub>3</sub> /opt/topspin3.2 TB 47



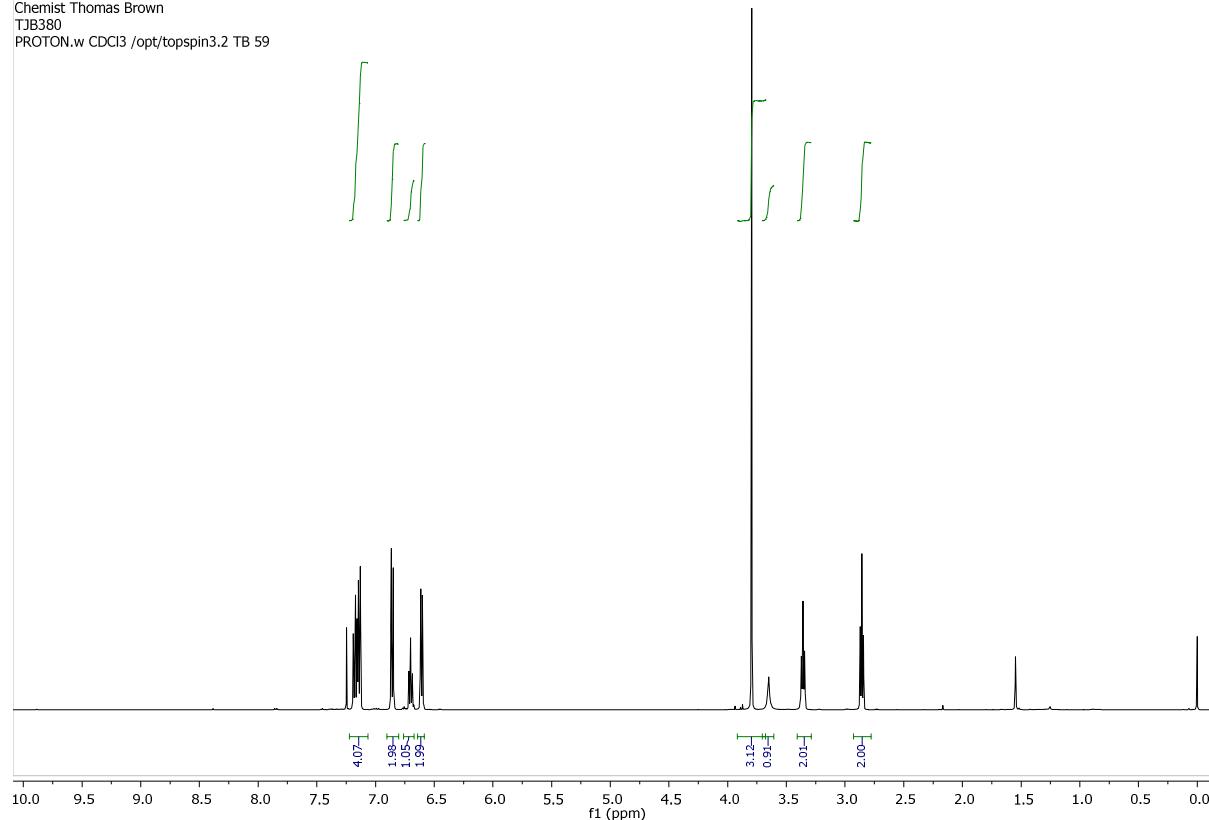
Current Data Parameters  
NAME Aug27-2015  
EXPNO 14  
PROCNO 1  
  
F2 - Acquisition Parameters  
Date 20150827  
Time 12.38  
INSTRUM spect  
PROBODIM 5 mm CPDCH DQ  
PULPROG jmod  
TD 65536  
SOLVENT CDCl<sub>3</sub>  
NS 256  
DS 4  
SWH 32894.738 Hz  
FIDRES 0.501934 Hz  
AQ 0.501934 sec  
RG 18.92  
DW 15.200 used  
DE 66.43 used  
TE 29.0 K  
CST2 145.000000  
CNST11 1.0000000  
D1 2.0000000 sec  
D20 0.00689655 sec  
TDO 1  
  
===== CHANNEL f1 =====  
SFO1 125.7728795 MHz  
NUC1 1H  
CPDPRG12 waltz16  
P1 9.50 used  
P2 19.00 used  
PLW1 24.0000000 W  
  
===== CHANNEL f2 =====  
SFO2 500.1320005 MHz  
NUC2 1H  
CPDPRG12 waltz16  
P1 0.00 used  
P2 13.0000000 W  
PLW2 0.26784000 W  
  
F2 - Processing parameters  
SI 32768  
SF 125.7577885 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40



*N*-(4-Methoxyphenethyl)aniline **24**.

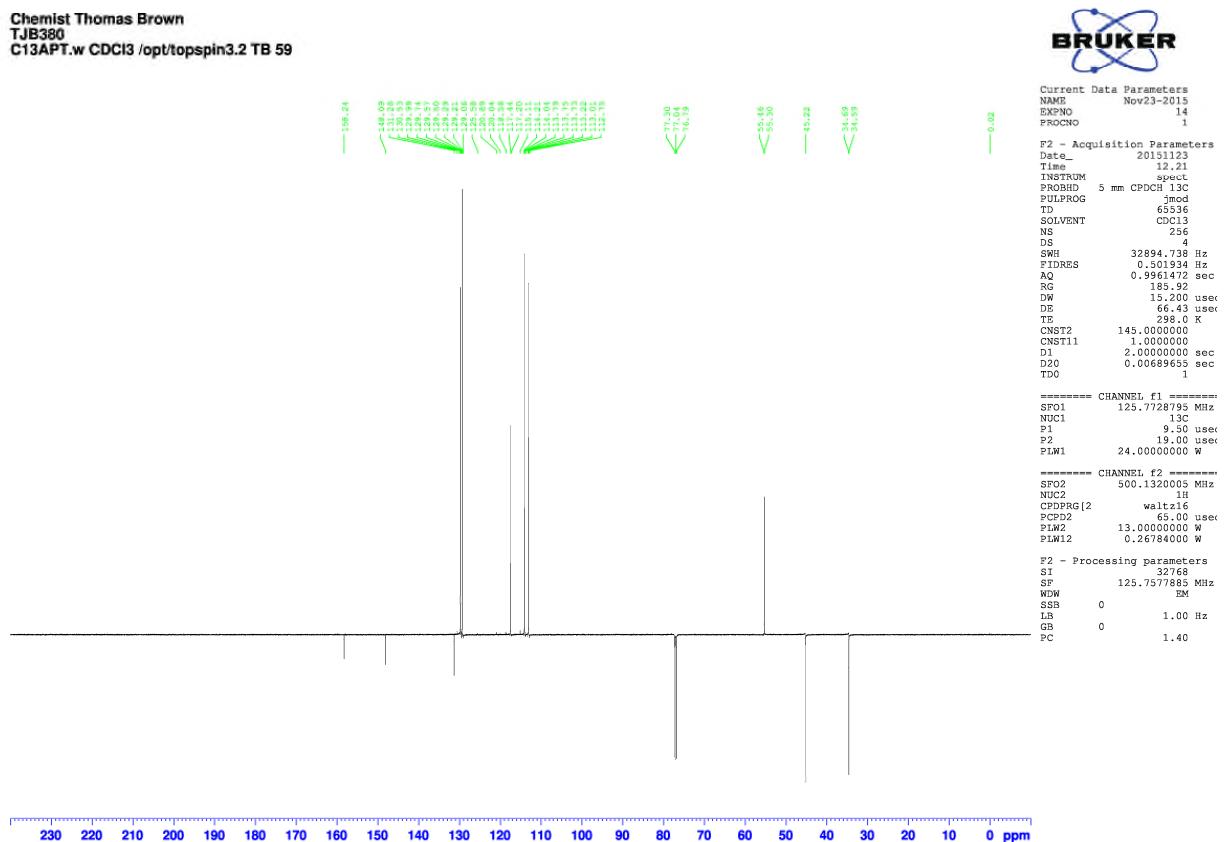


Nov23-2015.10.fid  
Chemist Thomas Brown  
TJB380  
PROTON.w CDCl<sub>3</sub> /opt/topspin3.2 TB 59

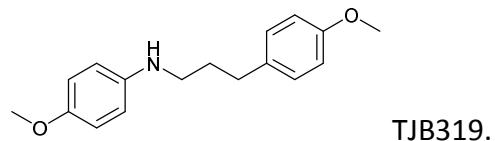


<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>).

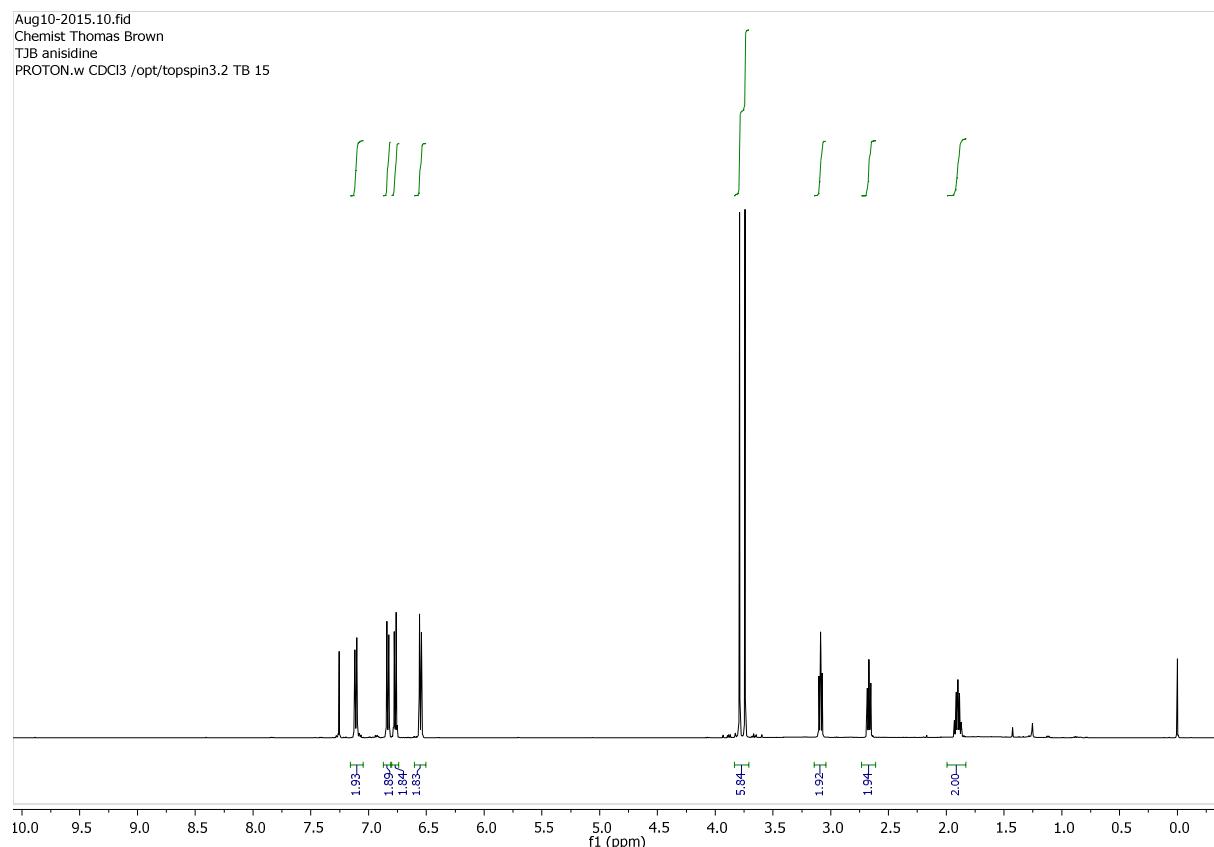
Chemist Thomas Brown  
TJB380  
C13APT.w CDCl<sub>3</sub> /opt/topspin3.2 TB 59



**4-Methoxy-N-(3-(4-methoxyphenyl)propyl)aniline **25**.**



$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ).

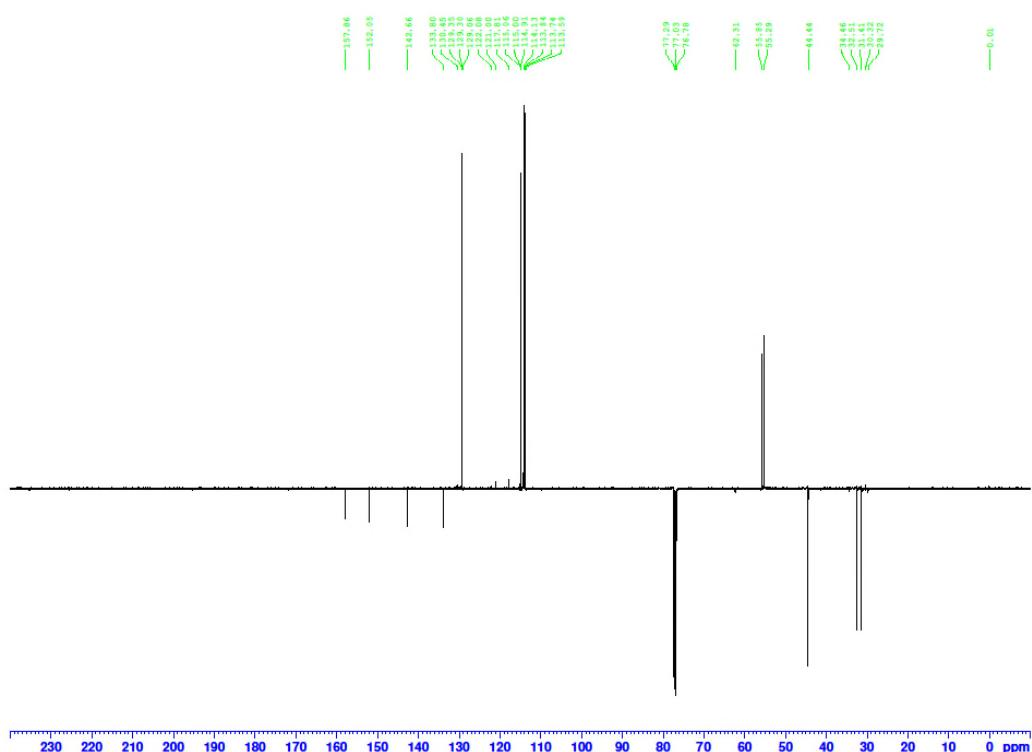


<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>).

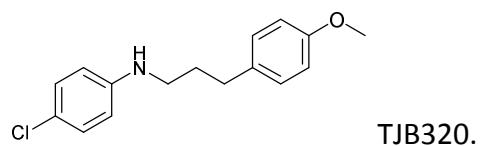
Chemist Thomas Brown  
TJB anisidine  
C13APT.w CDCl<sub>3</sub> /opt/topspin3.2 TB 15



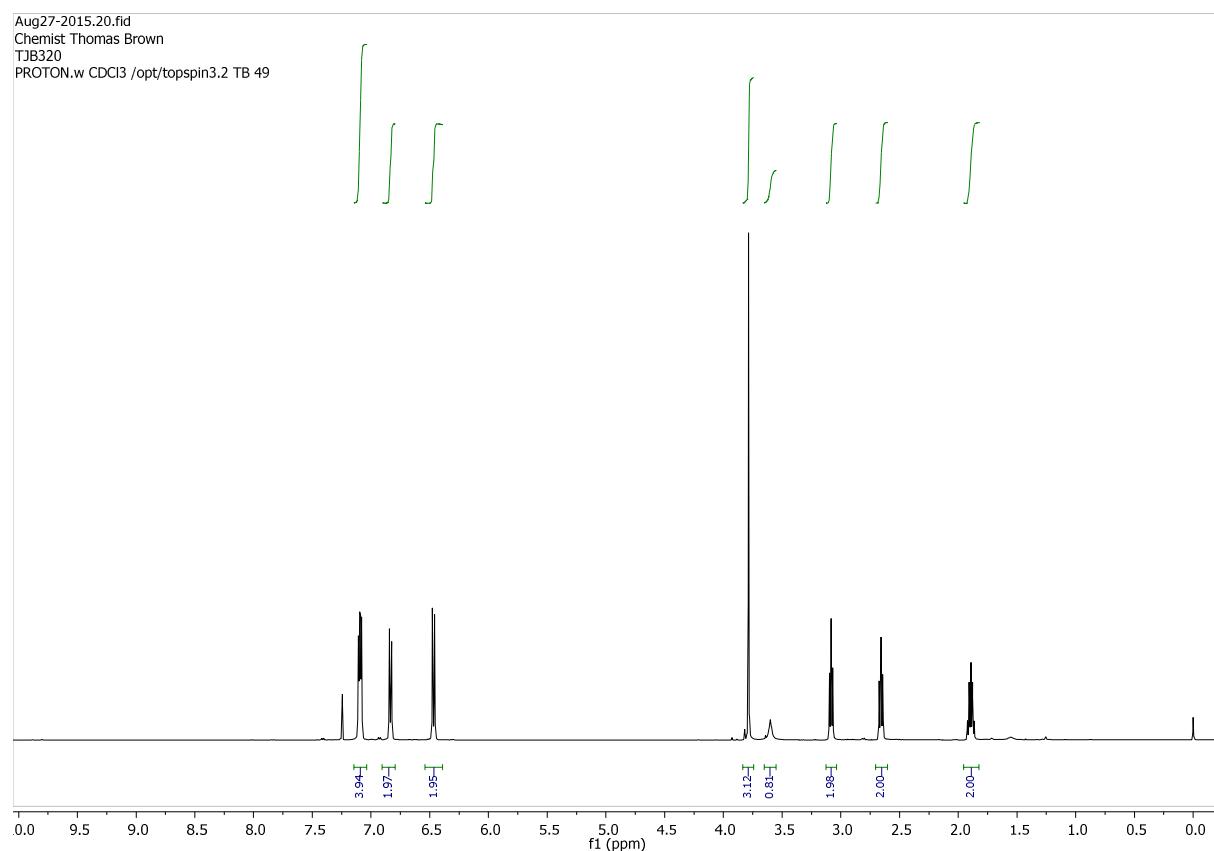
Current Data Parameters  
NAME Aug10-2015  
EXPNO 14  
PROCNO 1  
  
F2 - Acquisition Parameters  
DATE\_ 20150810  
TIME 14.12  
INSTRUM spect  
PROBHD 5 mm CPDCH 13C  
PULPROG fmod  
ID 6316  
SOLVENT CDCl<sub>3</sub>  
NS 256  
DS 4  
SWH 32894.734 Hz  
FIDRES 0.501934 Hz  
AQ 0.9961472 sec  
RG 185.92  
DW 1.200 usec  
DE 64.43 usec  
TE 298.0 K  
CNS12 145.000000  
CNS11 1.000000  
D1 2.0000000 sec  
D20 0.00689655 sec  
TDO 1  
  
----- CHANNEL F1 -----  
SF01 125.7728795 MHz  
NUC1 13C  
P1 9.50 usec  
P2 19.00 usec  
PLW1 24.0000000 W  
  
----- CHANNEL F2 -----  
SF02 500.1320005 MHz  
NUC2 1H  
CPDPG[2] Waltz16  
PCPD2 65.00 usec  
PLW2 13.00000000 W  
PLW12 0.26784000 W  
  
F2 - Processing parameters  
SI 32768  
SF 125.7577885 MHz  
WM 1000000 RM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40



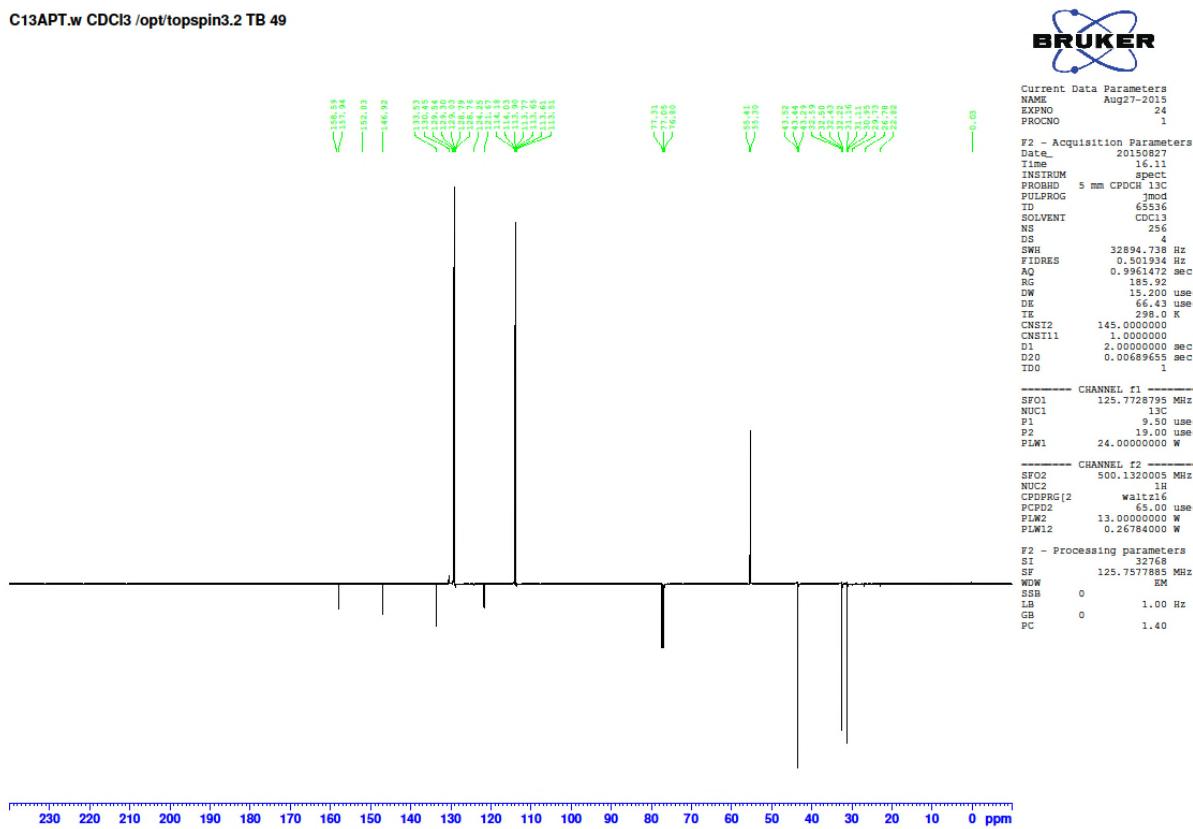
4-Chloro-N-(3-(4-methoxyphenyl)propyl)aniline **26**.



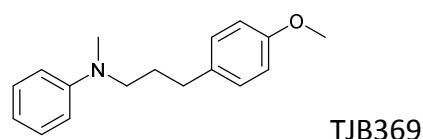
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ).



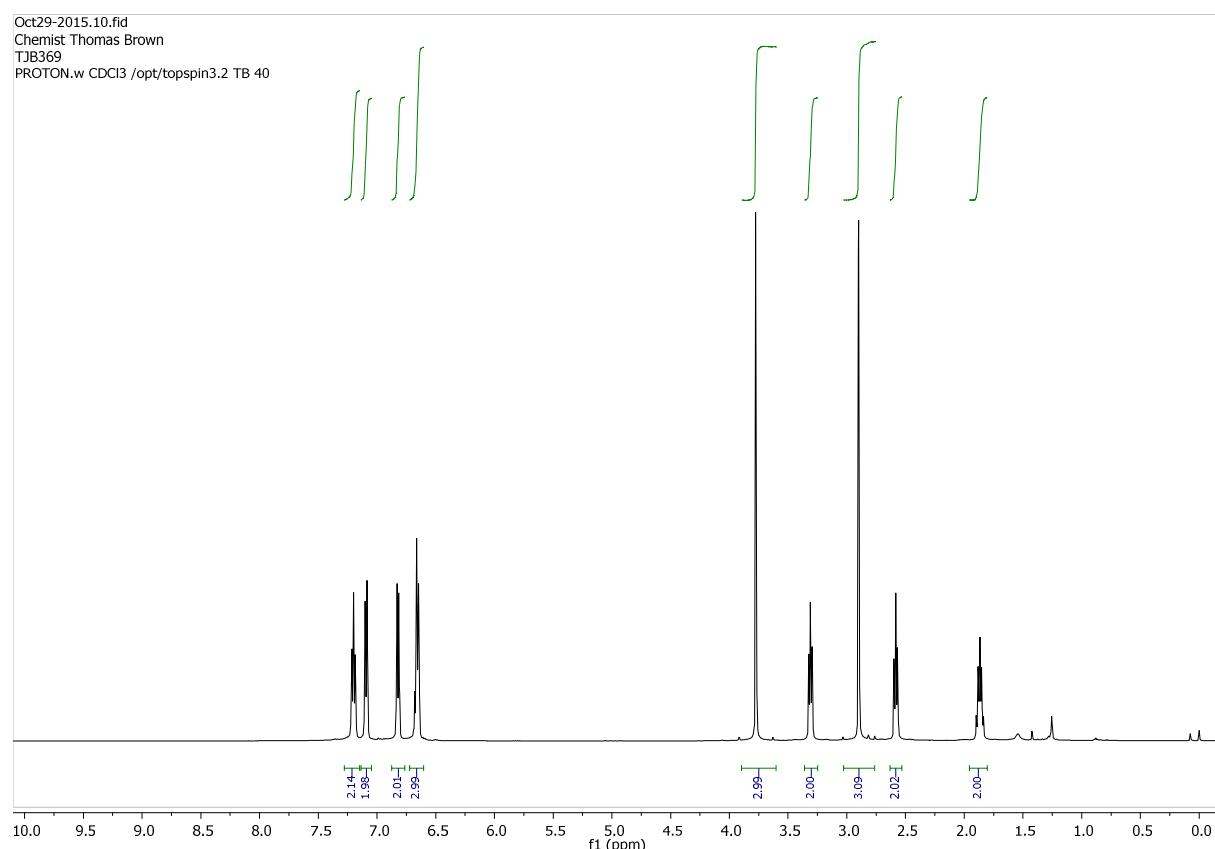
<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>).



*N*-(3-(4-Methoxyphenyl)propyl)-*N*-methylaniline **27**.



$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ).

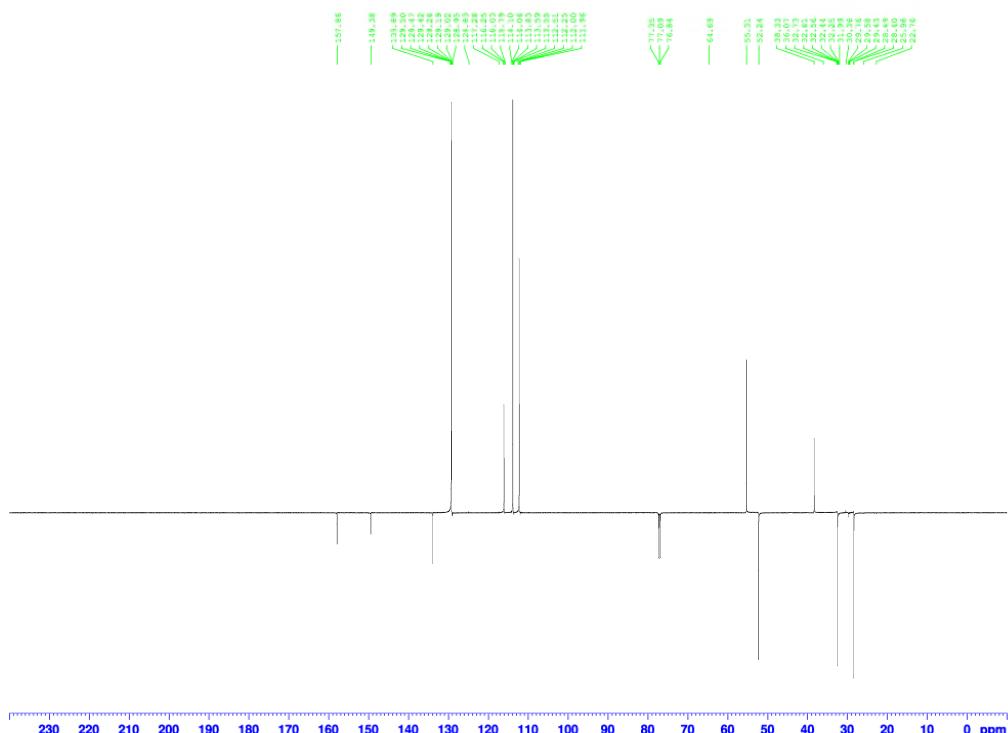


<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>).

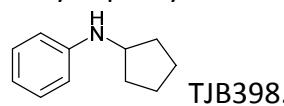
C13APT.w CDCl<sub>3</sub> /opt/topspin3.2 TB 40



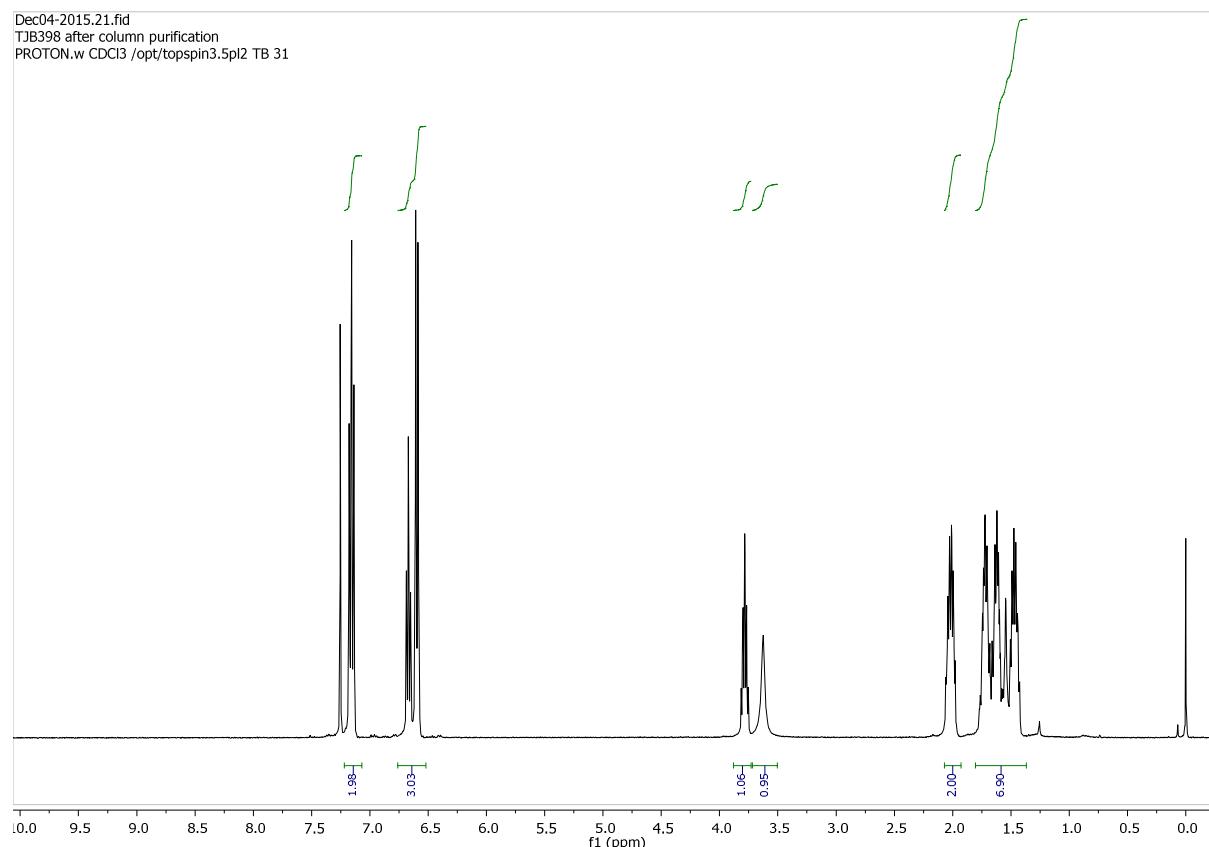
Current Data Parameters  
 NAME Oct29-2015  
 EXPNO 14  
 PROCNO 1  
  
 F2 - Acquisition Parameters  
 Date 20151030  
 Time 10.28  
 INSTRUM spect  
 PROBHD 5 mm CPDCH DC  
 PROBPRG jmod  
 TD 65536  
 SOLVENT CDCl<sub>3</sub>  
 NS 256  
 DS 4  
 SWH 32894.738 Hz  
 FIDRES 0.501934 Hz  
 AG 0.900000 sec  
 RG 185.82  
 DW 15.200 usec  
 DE 66.43 usec  
 T6 298.0 K  
 CNST2 145.000000  
 CNST11 1.0000000  
 D1 2.0000000 sec  
 D20 0.00689655 sec  
 T50 1  
  
 ===== CHANNEL f1 =====  
 SFO1 125.7728795 MHz  
 NUC1 1H  
 P1 9.30 usec  
 P2 19.00 usec  
 PLW1 24.0000000 W  
  
 ===== CHANNEL f2 =====  
 SFO2 500.1320005 MHz  
 NUC2 1H  
 CPDPRG[2 w1,tz16  
 PDPG2 60.00 usec  
 PLW2 13.0000000 W  
 PLW12 0.26784000 W  
  
 F2 - Processing parameters  
 SI 32768  
 SF 125.7577885 MHz  
 WDW 0  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40



*N*-Cyclopentylaniline **28**.



$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ).

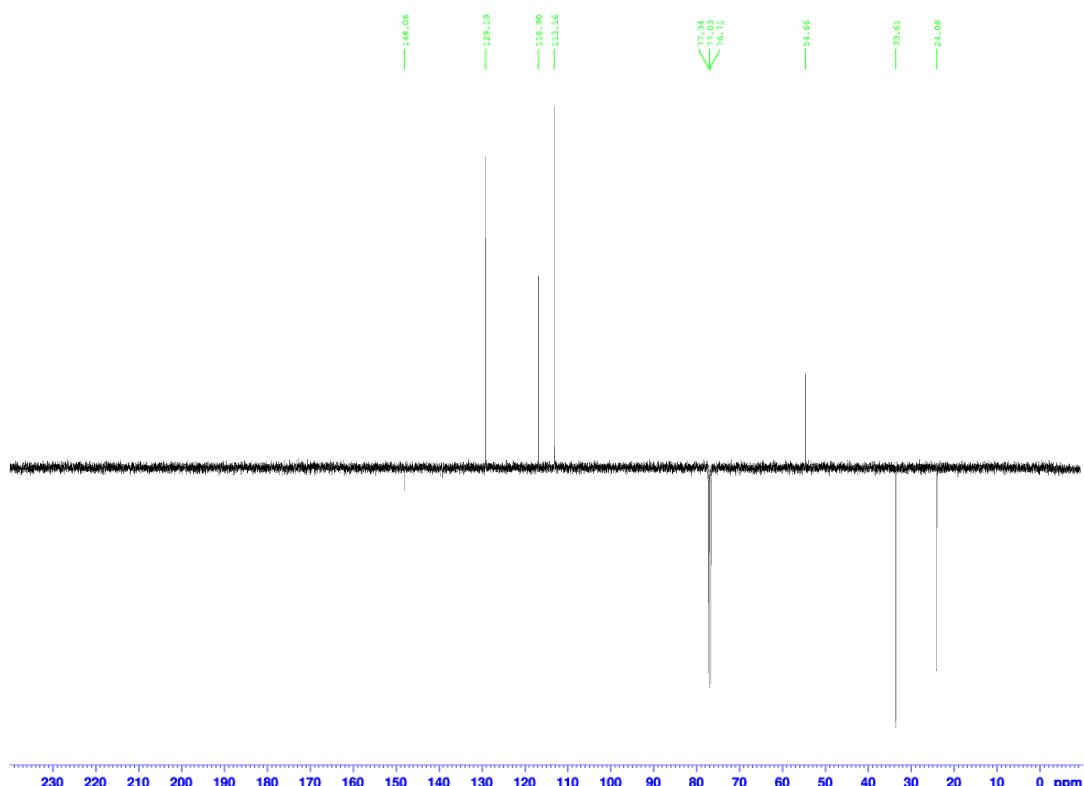


<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>).

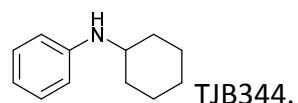
TJB398 after column purification  
C13APTlong.w CDCl<sub>3</sub> /opt/loppspin3.5pl2 TB 31



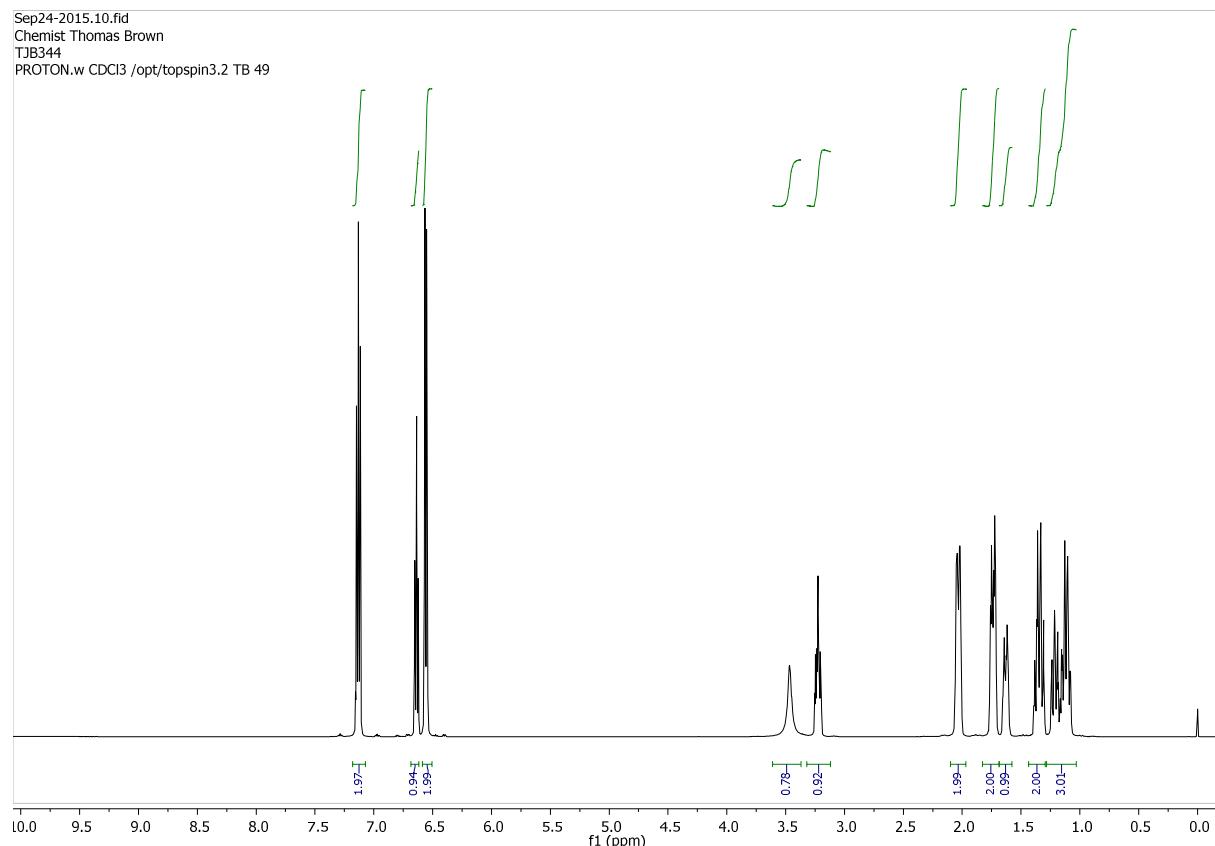
Current Data Parameters  
NAME 20151204  
EXPNO 24  
PROCNO 1  
  
P2 - Acquisition Parameters  
Date\_ 20151204  
Time 21:30 h  
INSTRUM spect  
PROBHD Z108618\_0844\_1  
DULPROG 65536  
TD 65536  
SOLVENT CDCl<sub>3</sub>  
NS 512  
DS 4  
SWH 26041.664 Hz  
ETRIM 0.397564 Hz  
AQ 1.2582912 sec  
RG 201.62  
DW 19.200 usec  
DE 6.50 usec  
TEC 300.0 K  
CNUST2 145.0000000  
CNUST11 1,0000000  
D1 2.0000000 sec  
D20 0.00689655 sec  
TD00 100.6248421 MHz  
SP01 100.6248421 MHz  
NUC1 <sup>13</sup>C  
P1 10.00 usec  
P2 20.00 usec  
P1M1 47.37500000 W  
SP02 400.13160000 MHz  
NUC2 <sup>1</sup>H  
CPDPRG[2] waltz16  
PCPD2 90.00 usec  
P1W2 12.92099953 W  
P1W12 0.30953279 W  
  
P2 - Processing parameters  
SI 32768  
SF 100.6127685 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40



*N*-Cyclohexylaniline **29**.



$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ).



<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>).

C13APT.w CDCl<sub>3</sub> /opt/topspin3.2 TB 49

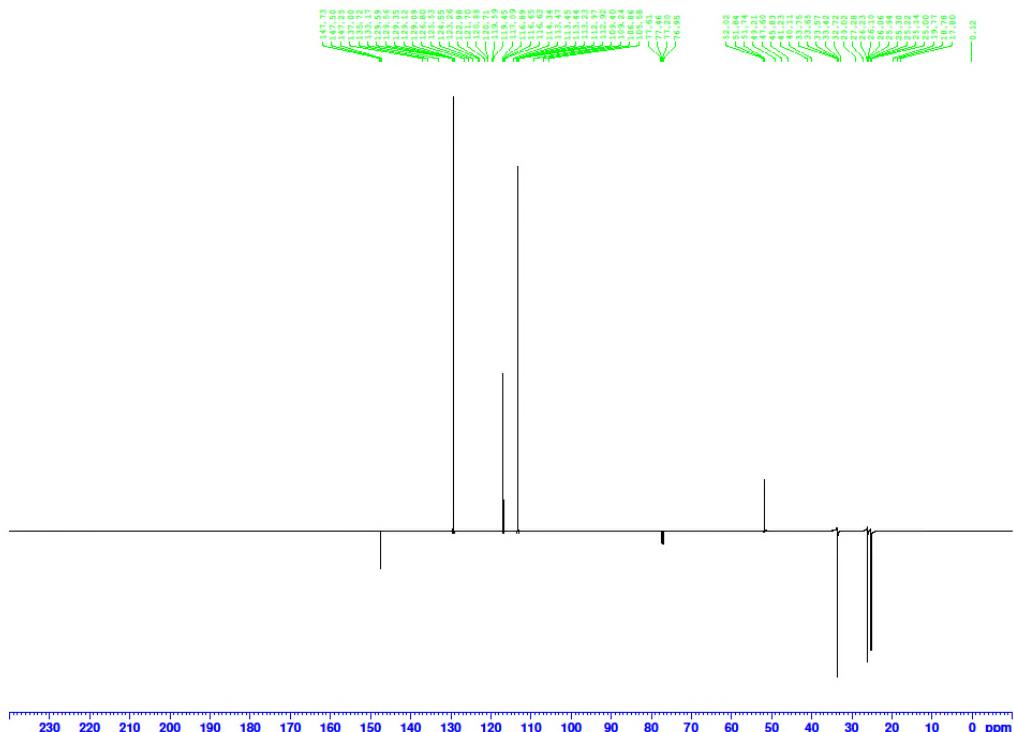


Current Data Parameters  
 NAME Sep24-2015  
 EXPNO 14  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date 20150924  
 Time 22.52  
 INSTRUM spect  
 PROBHD 5 mm CPDCH<sup>13</sup>C  
 PULPROG 300d  
 TD 65536  
 SOLVENT CDCl<sub>3</sub>  
 NS 256  
 D1 4  
 SWH 32894.738 Hz  
 FIDRES 0.501934 Hz  
 AQ 0.999999 sec  
 RG 185.32  
 DW 15.200 usec  
 DE 66.43 usec  
 TE 298.0 K  
 CNTZ2 145.000000  
 CNTZ11 1.0000000  
 D1 2.0000000 sec  
 D20 0.00689655 sec  
 TDO 1

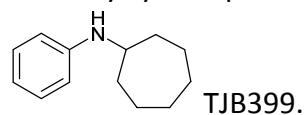
CHANNEL f1  
 SF01 125.7728795 MHz  
 NUC1 1H  
 P1 9.50 usec  
 P2 19.00 usec  
 PLW1 24.0000000 W

CHANNEL f2  
 SF02 500.1320005 MHz  
 NUC2 1H  
 CPDPRG[2] waltz16  
 P02 1.0000000 usec  
 PLW2 13.0000000 W  
 PLW12 0.267840000 W

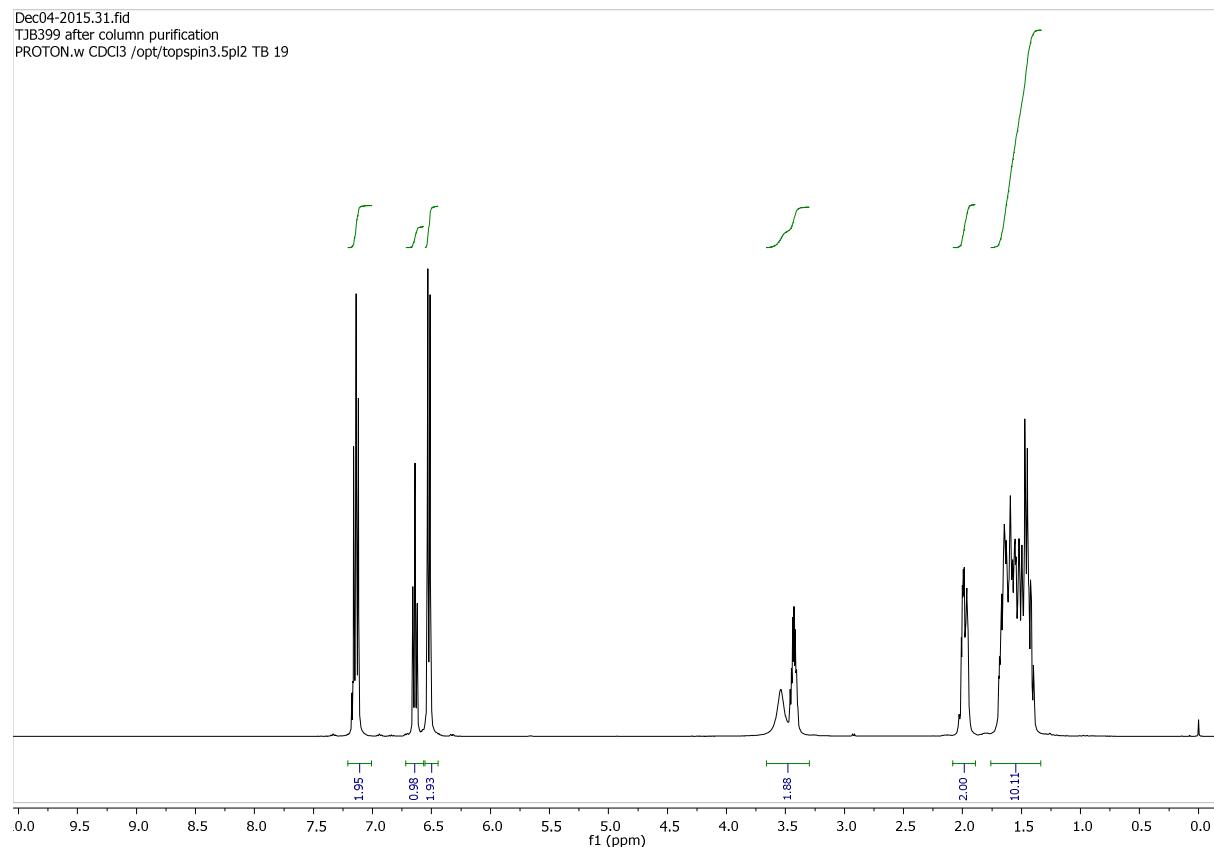
F2 - Processing parameters  
 SI 32768  
 SF 125.7577885 MHz  
 WDW EN  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40



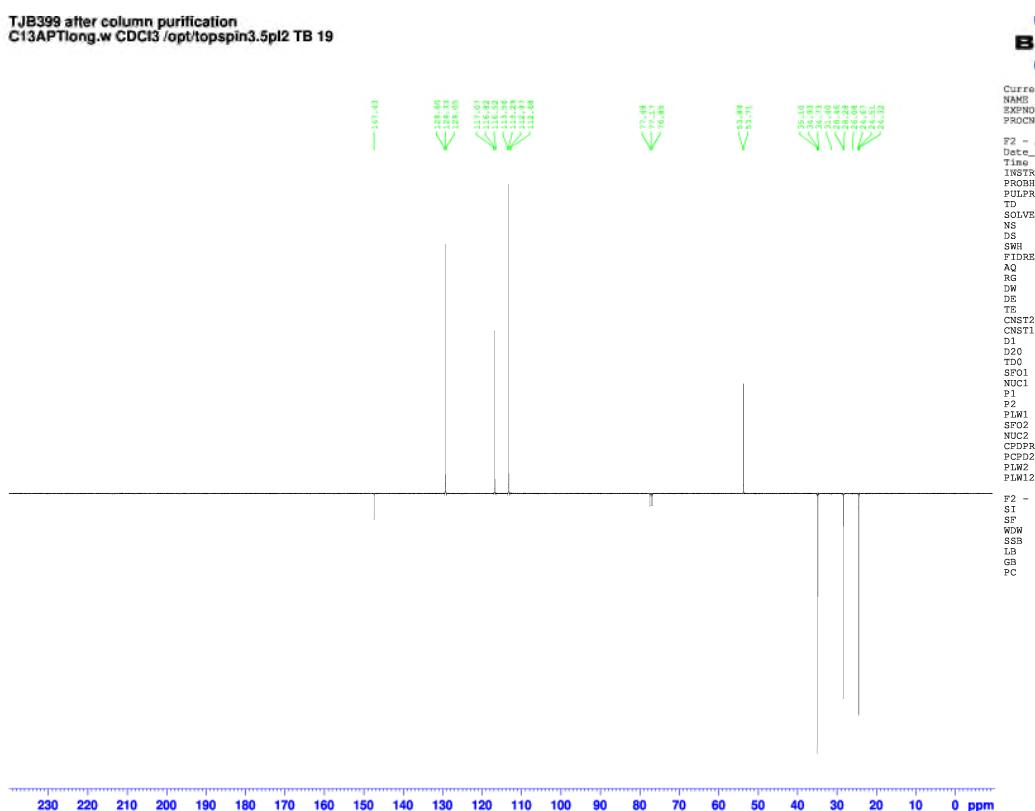
*N*-Phenylcycloheptanamine **30**.



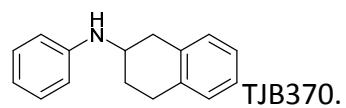
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ).



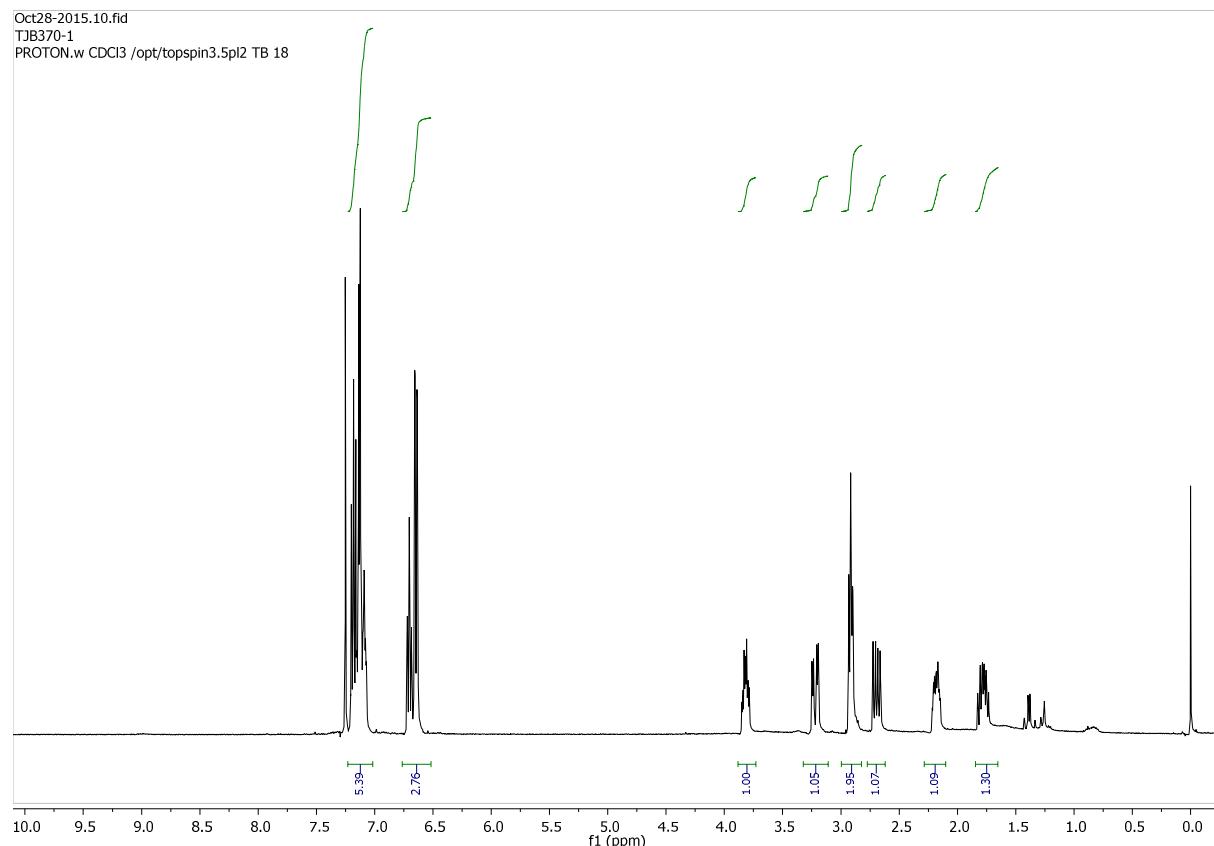
<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>).



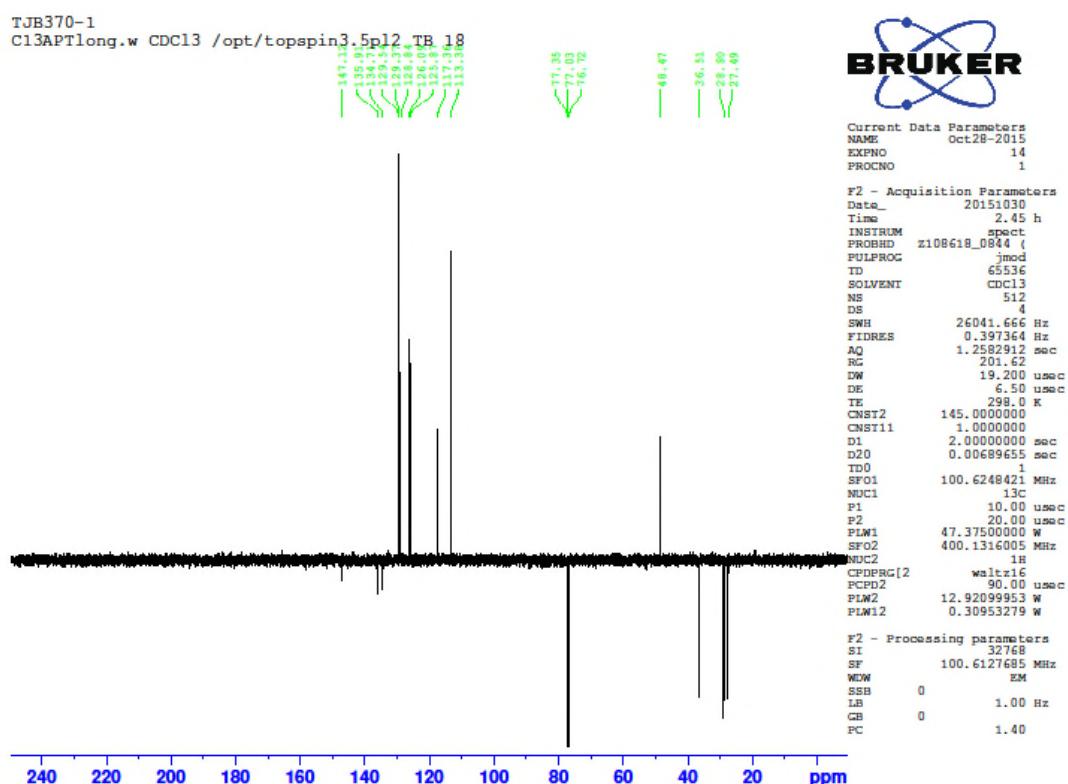
*N*-Phenyl-1,2,3,4-tetrahydronaphthalen-2-amine **31**.



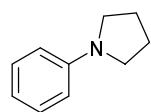
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ).



<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>).

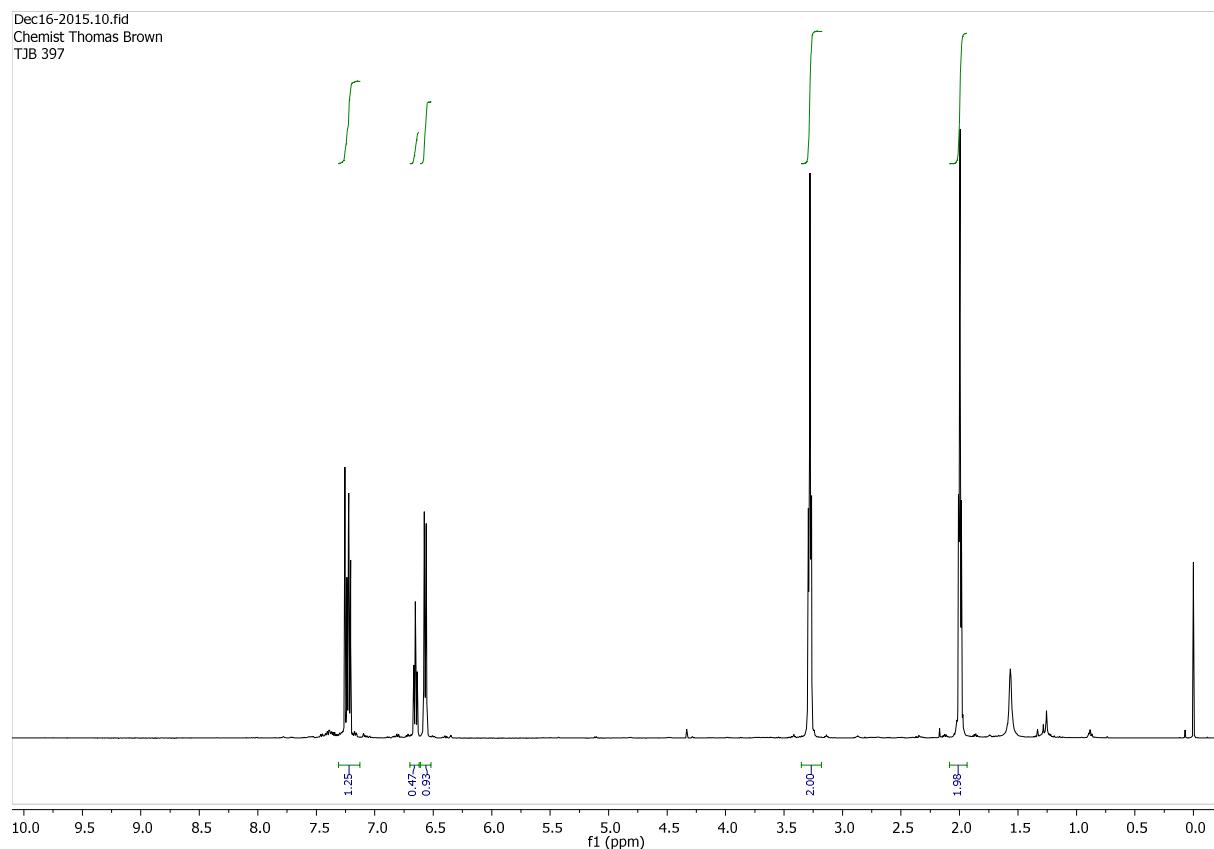


**1-Phenylpyrrolidine 32.**



TJB395 original pdf spectra below –filename says 397.

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ).

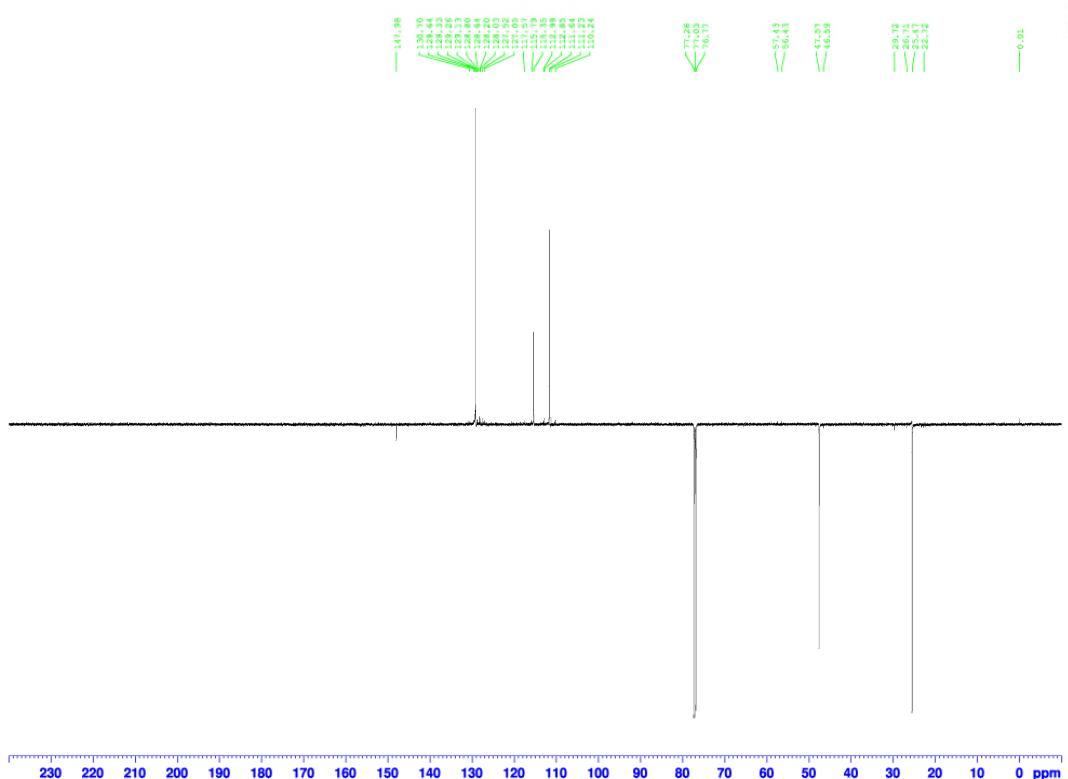


<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>).

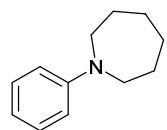
Chemist Thomas Brown  
TJB 397  
C13APT.w CDCl<sub>3</sub> /opt/topspin3.2 TB 32



Current Data Parameters  
NAME Dec16-2015  
EXPNO 11  
PROCNO 1  
  
F2 - Acquisition Parameters  
Date\_ 20151217  
Time 1.03  
INSTRUM spect  
PROBID 5 mm CPDCH 13C  
PULPROG jmod  
TD 65536  
SOLVENT CDCl<sub>3</sub>  
NS 256  
DS 4  
SWH 32894.738 Hz  
FIDRES 0.501934 Hz  
AQ 0.9961472 sec  
RG 185.92  
DW 15.60 usec  
DE 66.43 usec  
TE 298.0 K  
CNUST1 145.000000  
CNUST11 1.000000  
D1 2.0000000 sec  
D2 0.00689655 sec  
TD0 1  
  
===== CHANNEL, f1 =====  
SF01 125.7728795 MHz  
NUC1 13C  
P1 9.50 usec  
P2 19.00 usec  
PLW1 24.00000000 W  
  
===== CHANNEL, f2 =====  
SF02 500.1320005 MHz  
NUC2 1H  
CPDPRG[2 waltz16  
PCP 65.00 usec  
PLW2 13.00000000 W  
PLW12 0.26784000 W  
  
F2 - Processing parameters  
SI 32768  
SF 125.7577885 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40



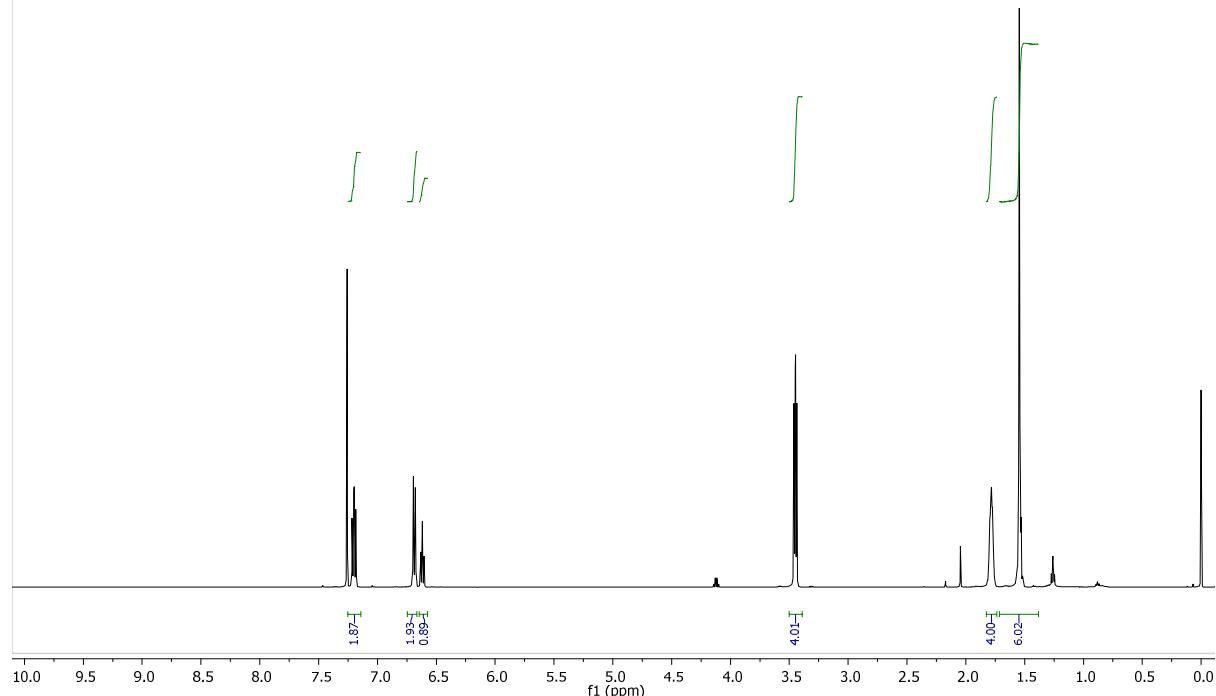
**1-Phenylazapine 33.**



TJB402.

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ).

Dec09-2015.10.fid  
Chemist Thomas Brown  
TJB402  
PROTON.w  $\text{CDCl}_3$  /opt/topspin3.2 TB 43

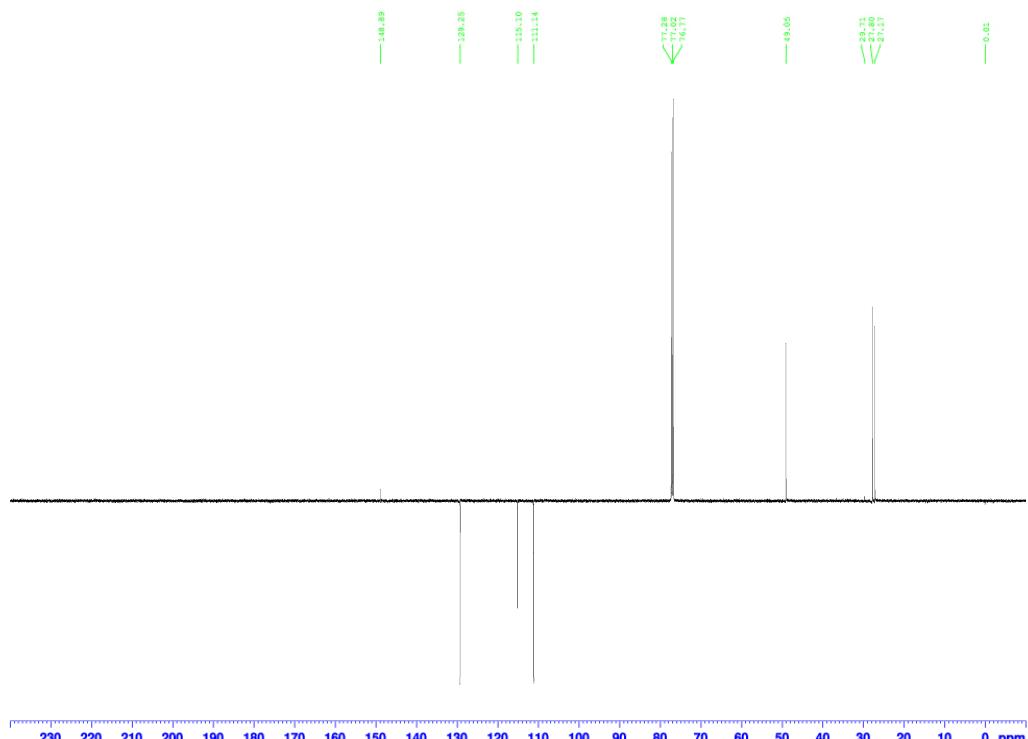


<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>).

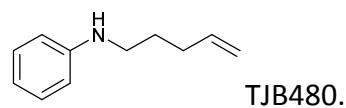
Chemist Thomas Brown  
TJB402  
C13APT.w CDCl<sub>3</sub> /opt/topspin3.2 TB 43



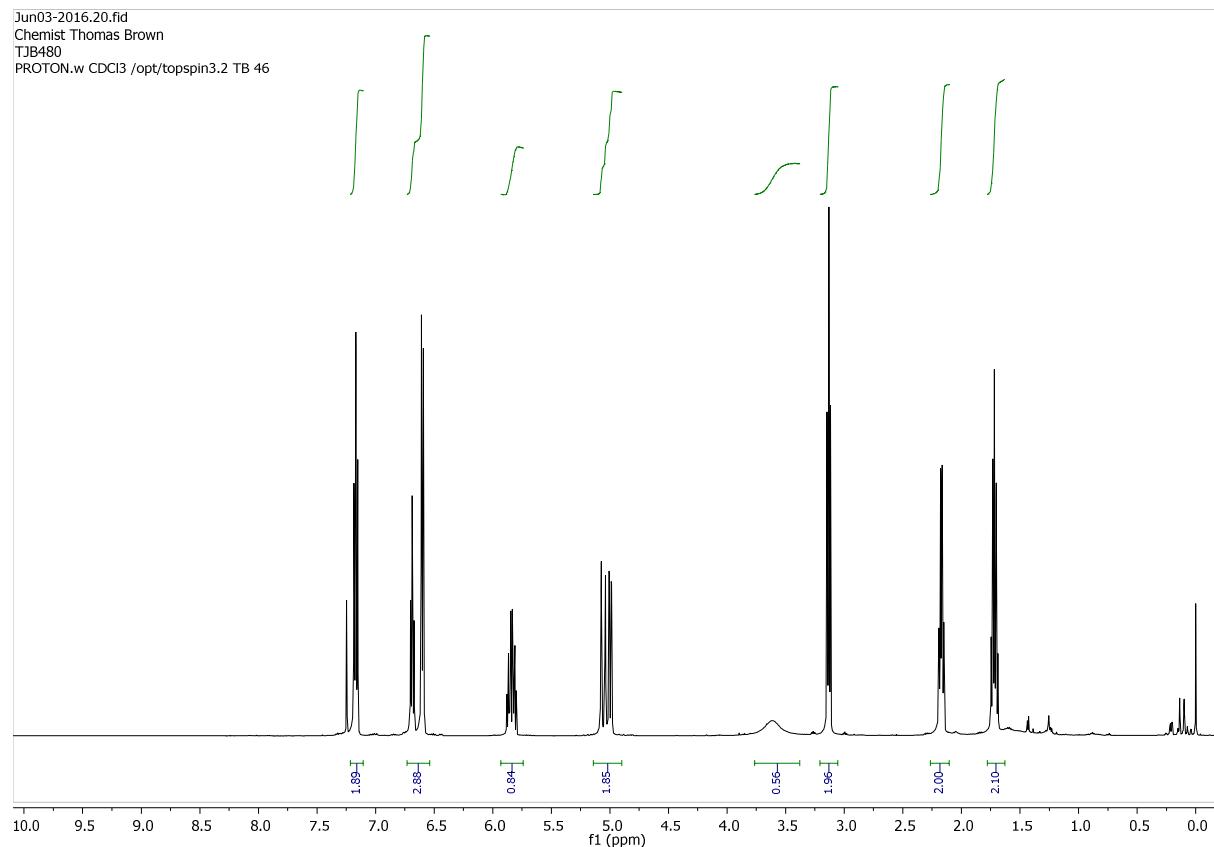
Current Data Parameters  
NAME Dec09-2015  
EXPNO 11  
PROCNO 1  
  
P2 - Acquisition Parameters  
Date\_ 20151209  
Time\_ 23.40  
INSTRUM spect  
PROBHD 5 mm CPDCT-13C  
PROBPRG :  
TD 65536  
SOLVENT CDCl<sub>3</sub>  
NS 256  
DS 4  
SWH 32894.738 Hz  
FIDRES 0.501934 Hz  
AQ 0.9961472 sec  
RG 185.35  
DW 15.00 usec  
DE 66.43 usec  
TE 298.0 K  
CNS2T2 145.000000  
CNS2T11 1.000000  
D1 2.0000000 sec  
D20 0.00689655 sec  
TD0 1  
  
===== CHANNEL f1 =====  
SFO1 125.7728795 MHz  
NUC1 <sup>13</sup>C  
P1 9.50 usec  
P2 19.00 usec  
PLW1 24.0000000 W  
  
===== CHANNEL f2 =====  
SFO2 500.1320005 MHz  
NUC2 <sup>1</sup>H  
CPDPRG[2] waltz16  
PCPD2 65.00 usec  
PLW2 13.0000000 W  
PLW2 0.26784000 W  
  
P2 - Processing parameters  
SI 32768  
SF 125.7577885 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40



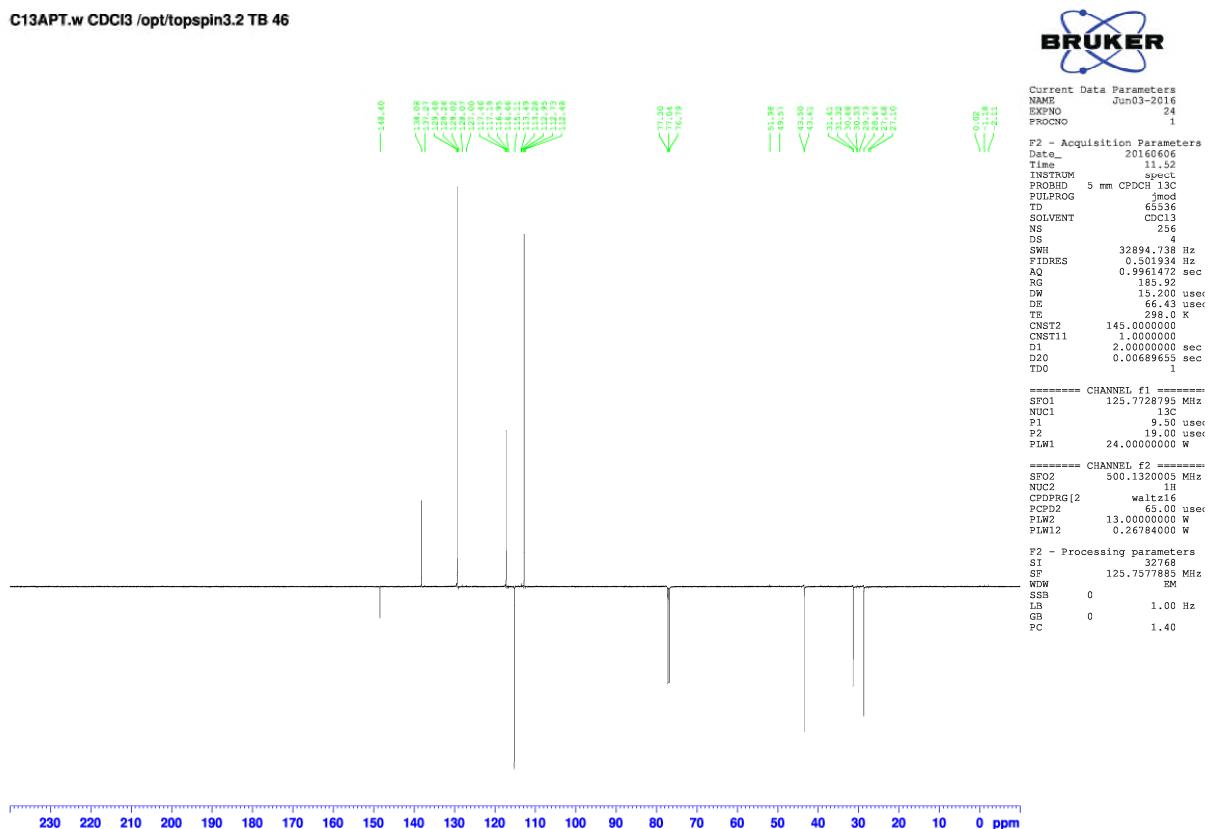
*N*-(Pent-4-en-1-yl)aniline **37**.



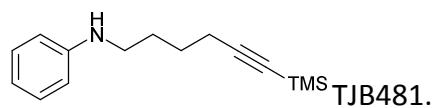
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ).



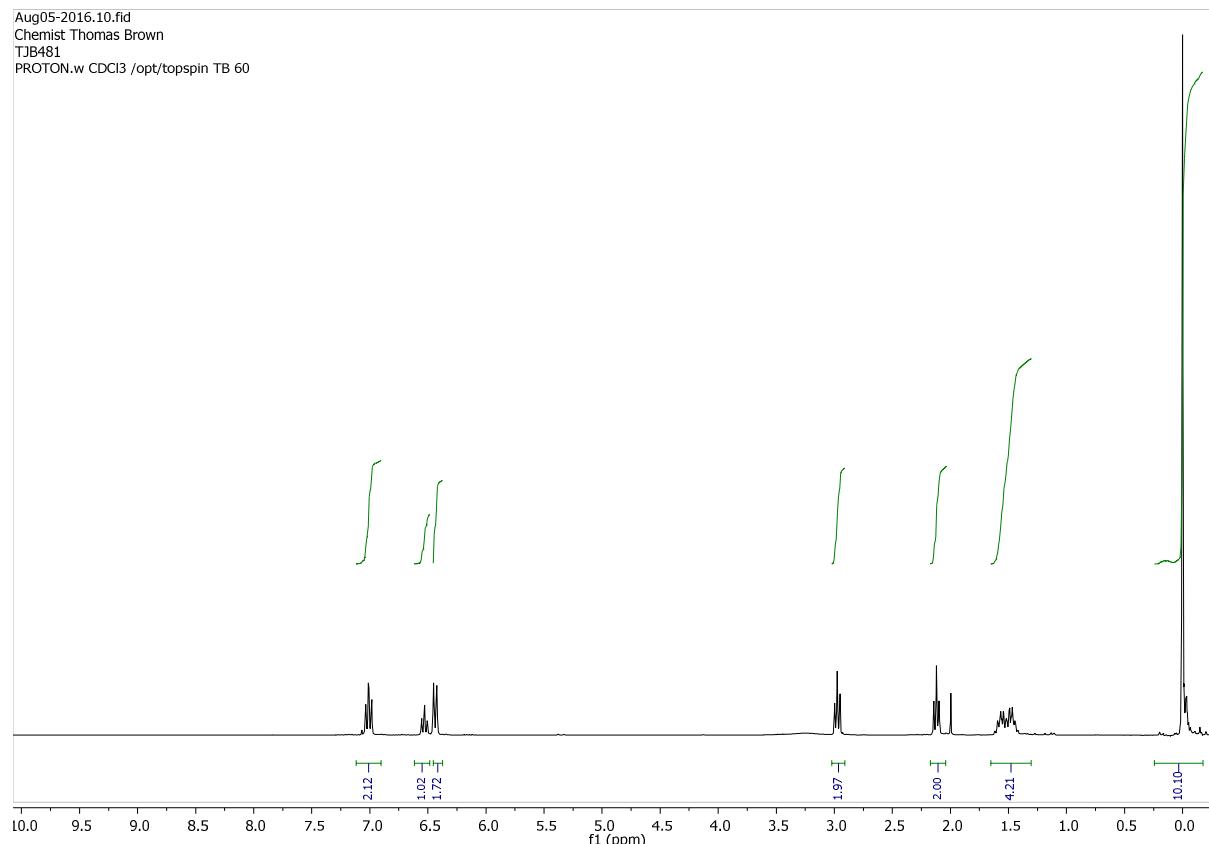
<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>).



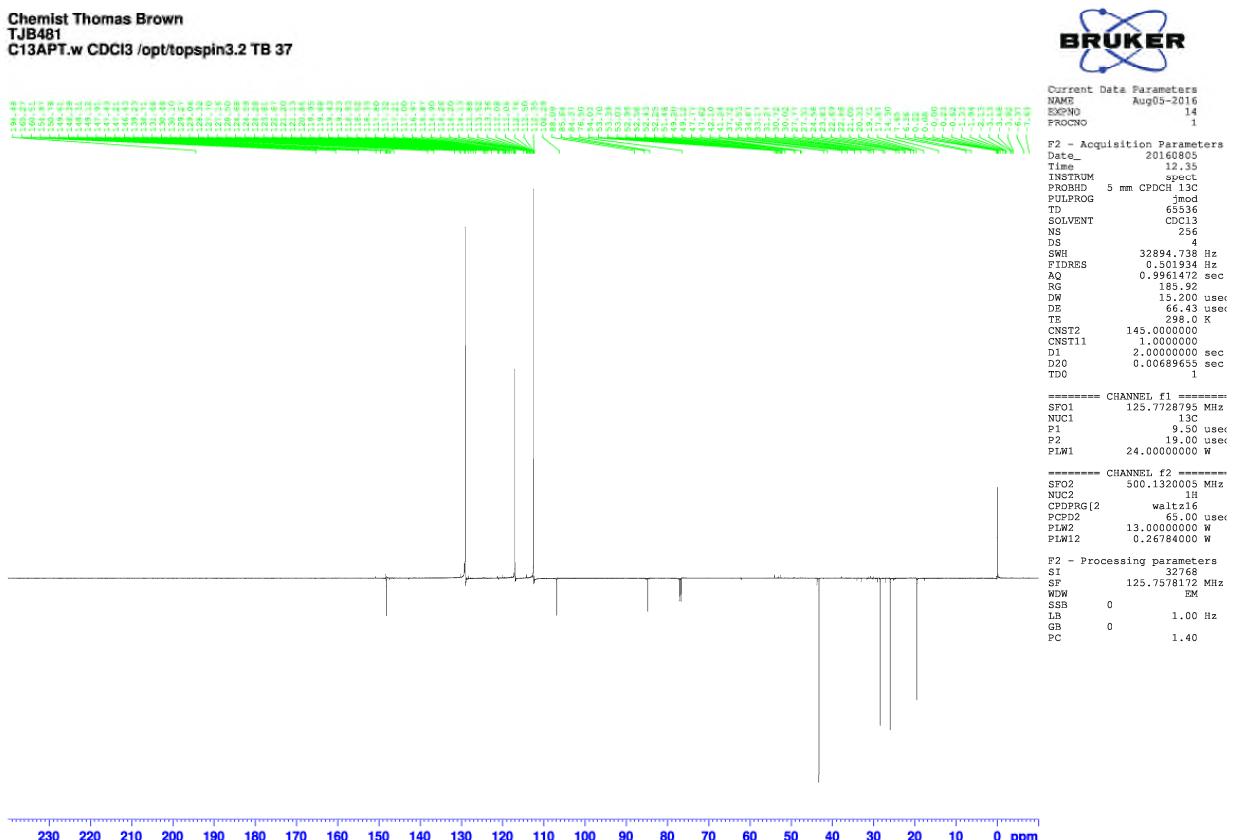
*N*-(6-(trimethylsilyl)hex-5-yn-1-yl)aniline **38**.



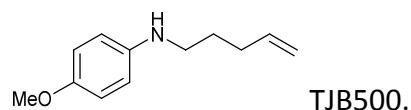
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ).



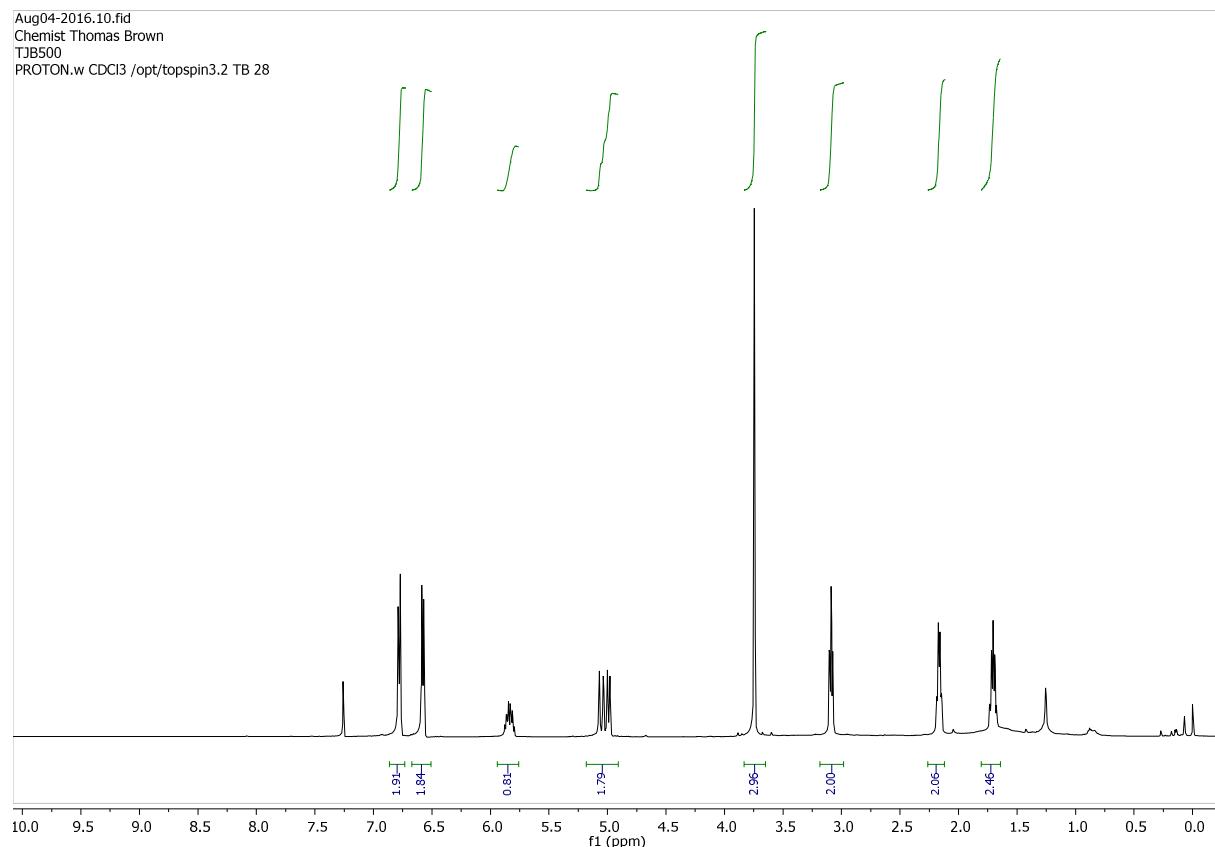
<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>).



4-Methoxy-N-(pent-4-en-1-yl)aniline **39**.



$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ).

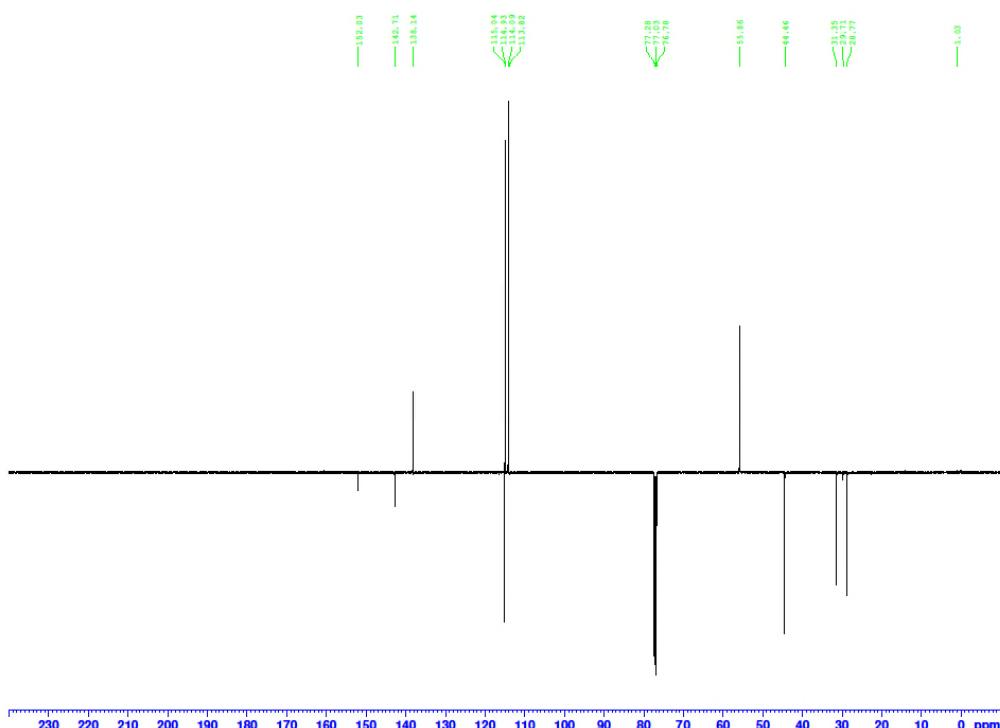


<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>).

Chemist Thomas Brown

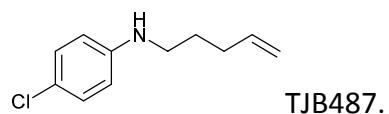
TJB500

C13APT.w CDCl<sub>3</sub> /opt/topspin3.2 TB 28

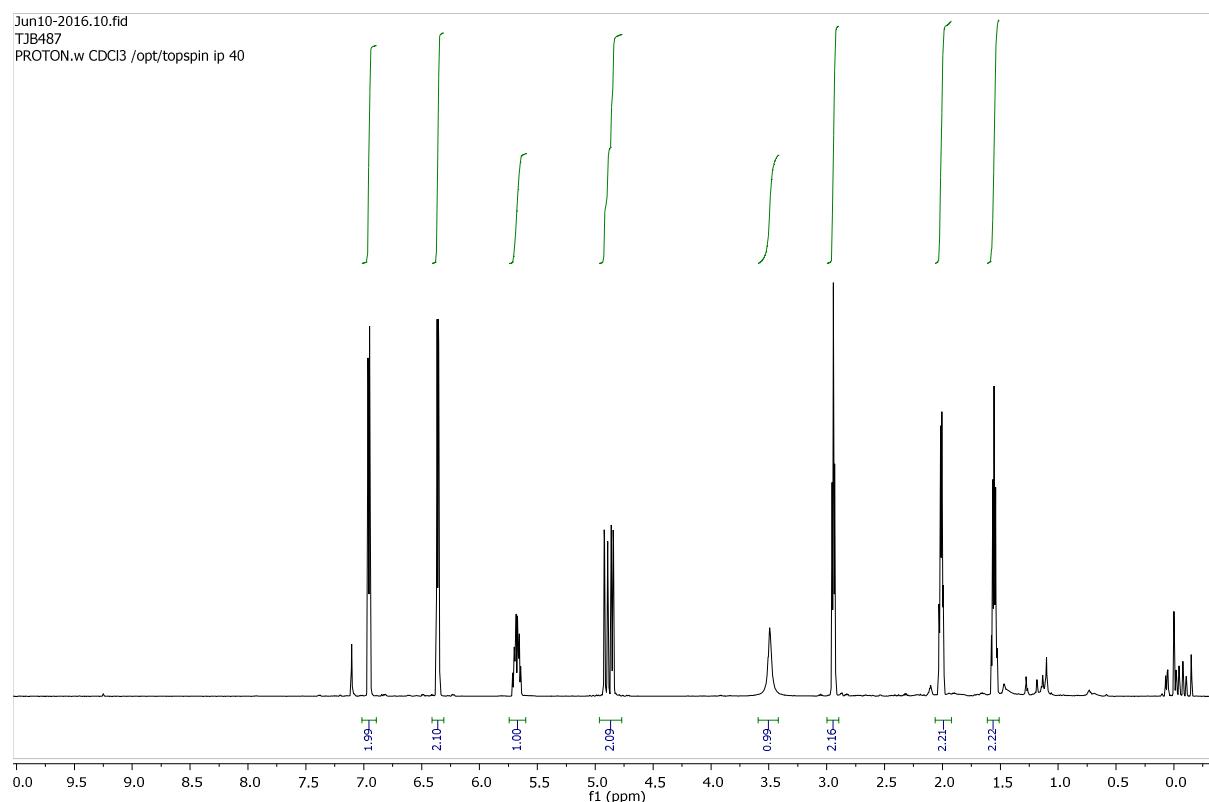


230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

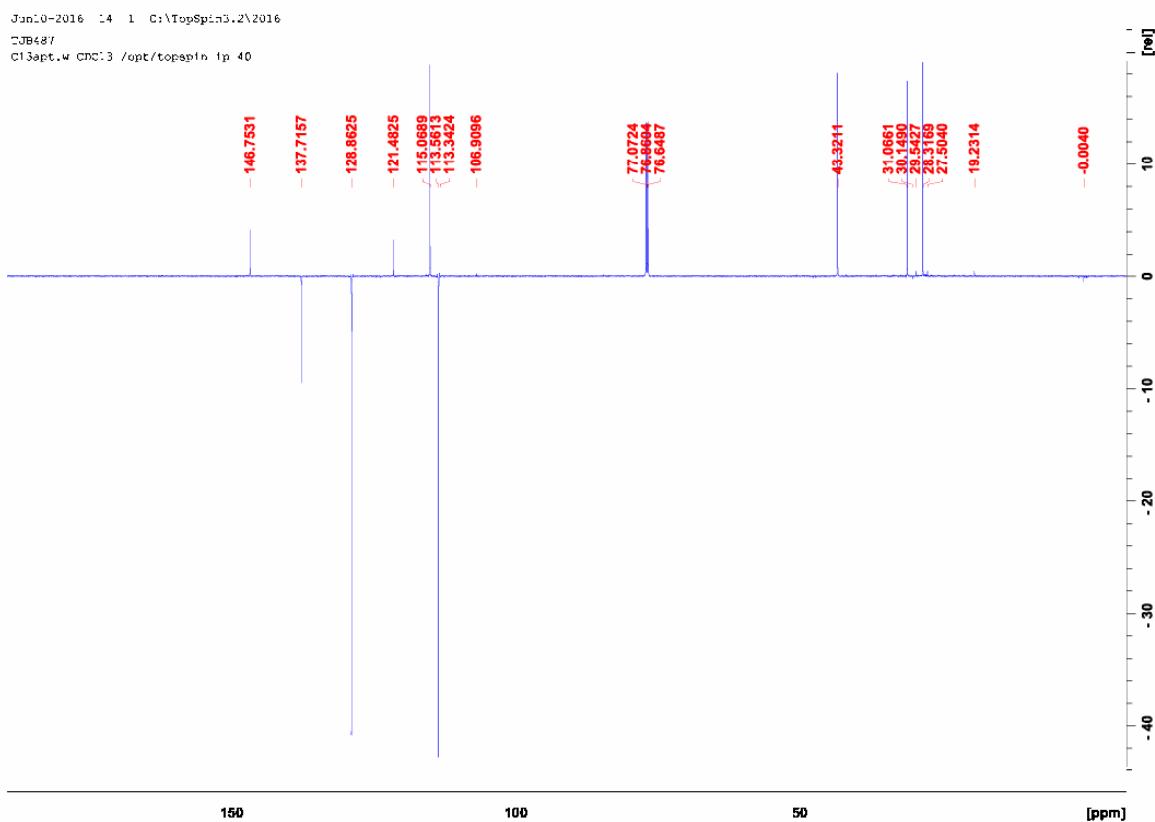
4-Chloro-N-(pent-4-en-1-yl)aniline **40**.



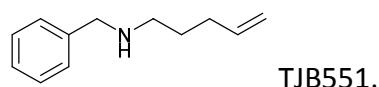
$^1\text{H}$  NMR (600 MHz,  $\text{CDCl}_3$ ).



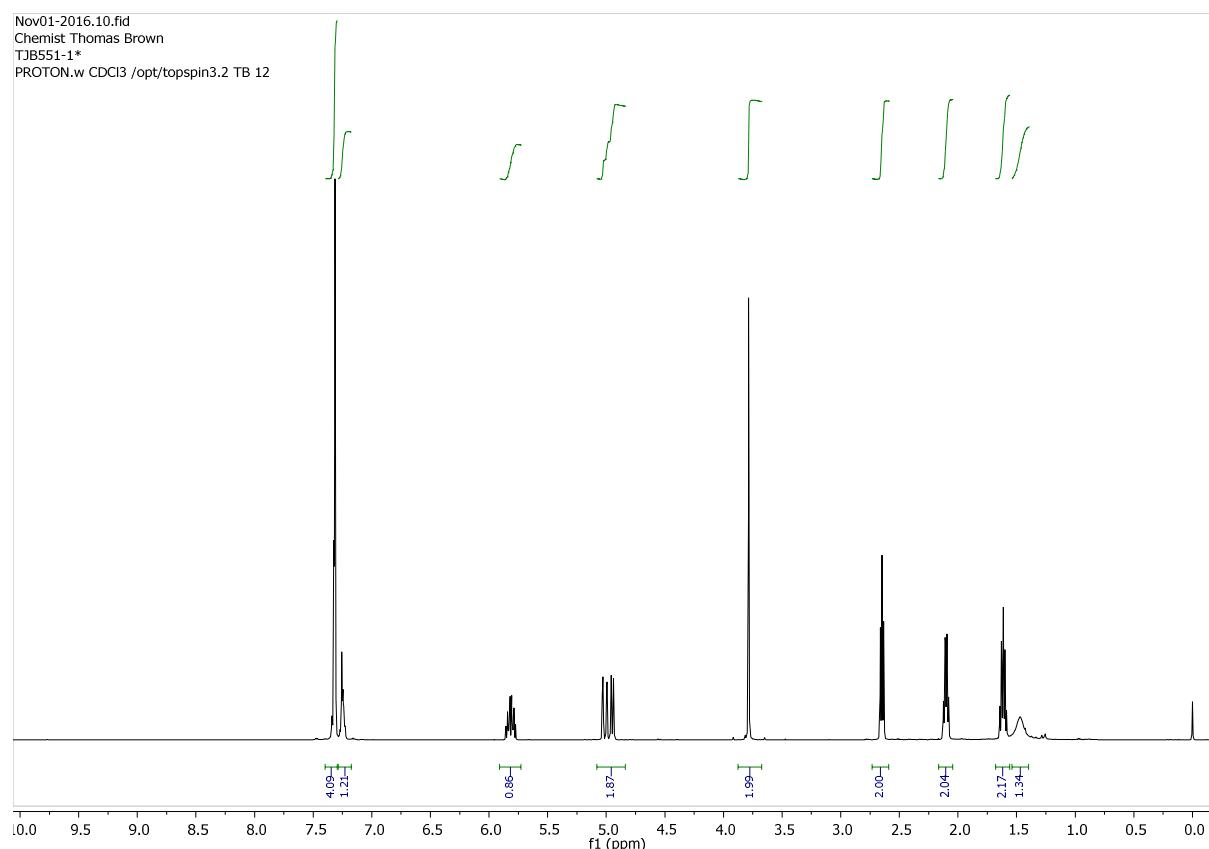
<sup>13</sup>C NMR (150 MHz, CDCl<sub>3</sub>).



*N*-Benzylpent-4-en-1-amine **41**.

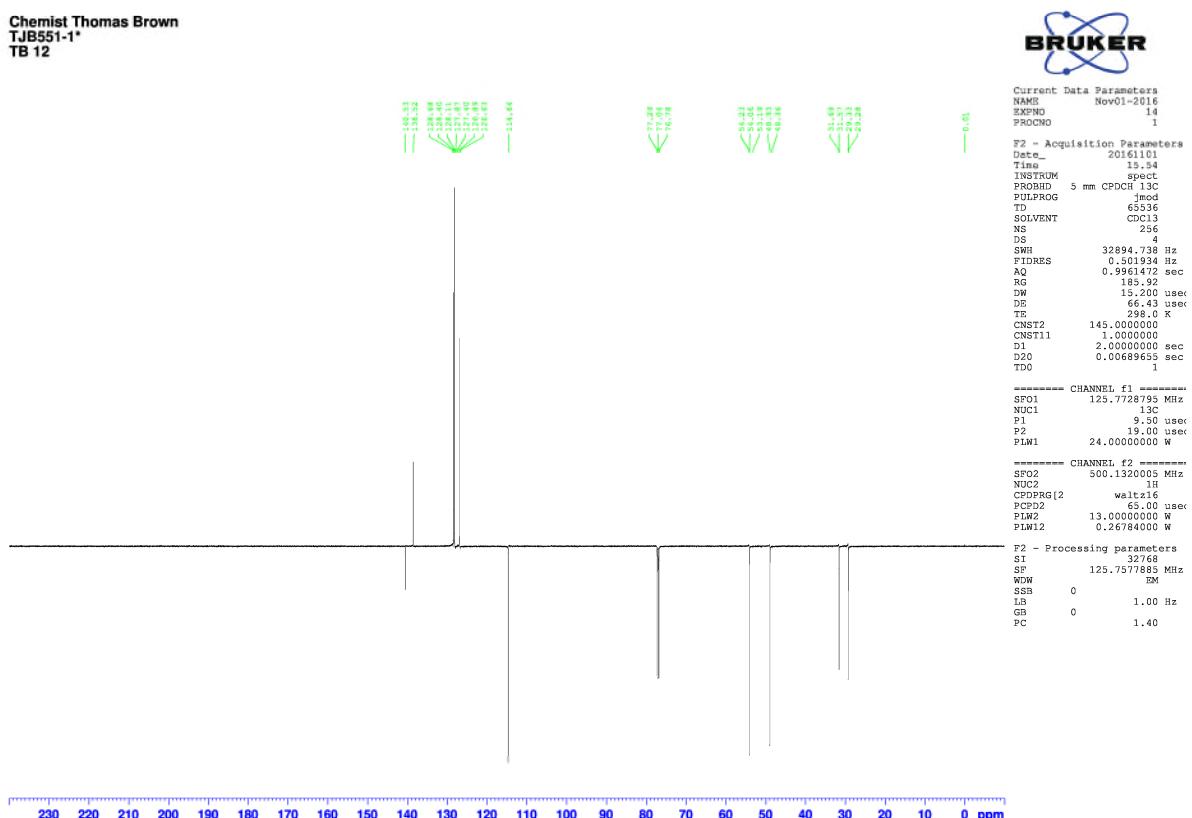


$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ).

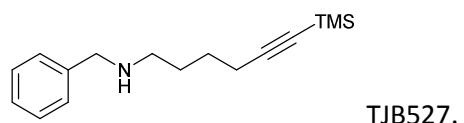


<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>).

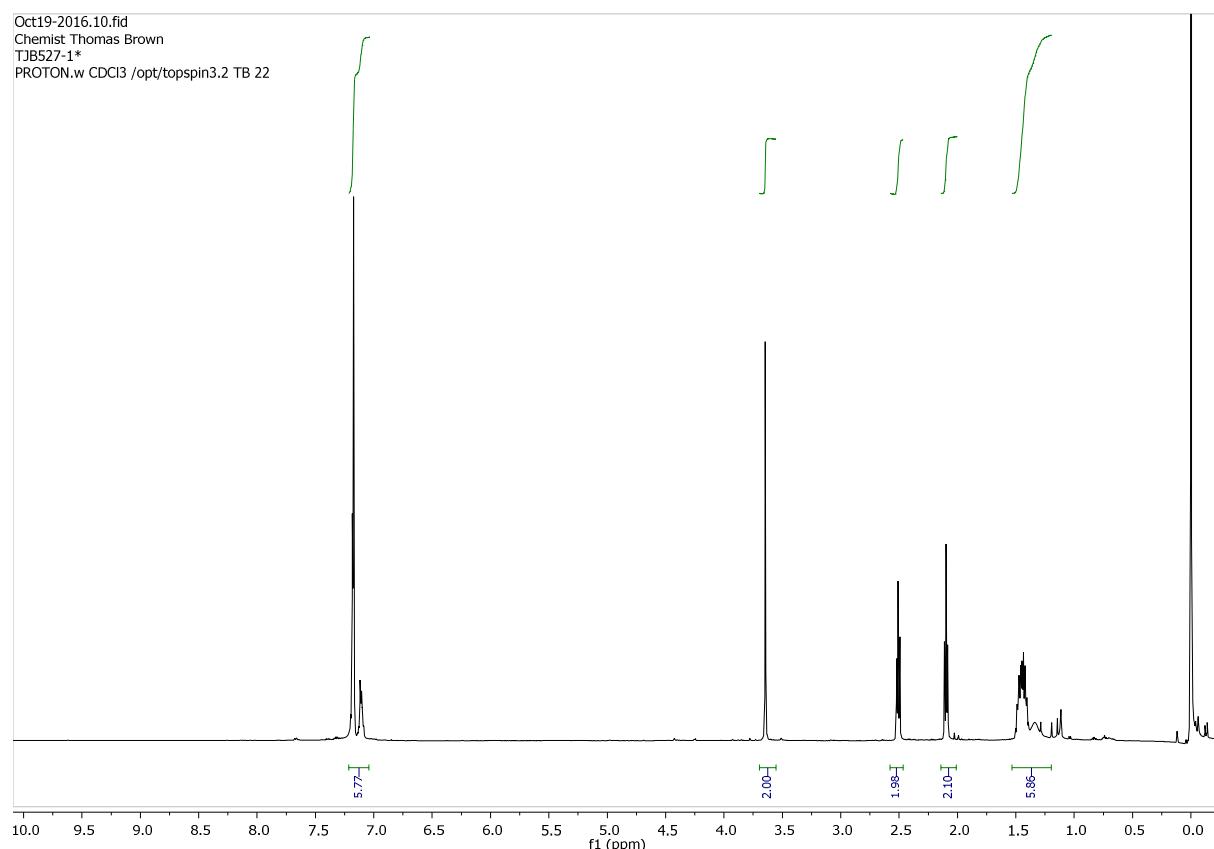
**Chemist Thomas Brown  
TJB551-1\*  
TB 12**



*N*-Benzyl-6-(trimethylsilyl)hex-5-yn-1-amine **42**.

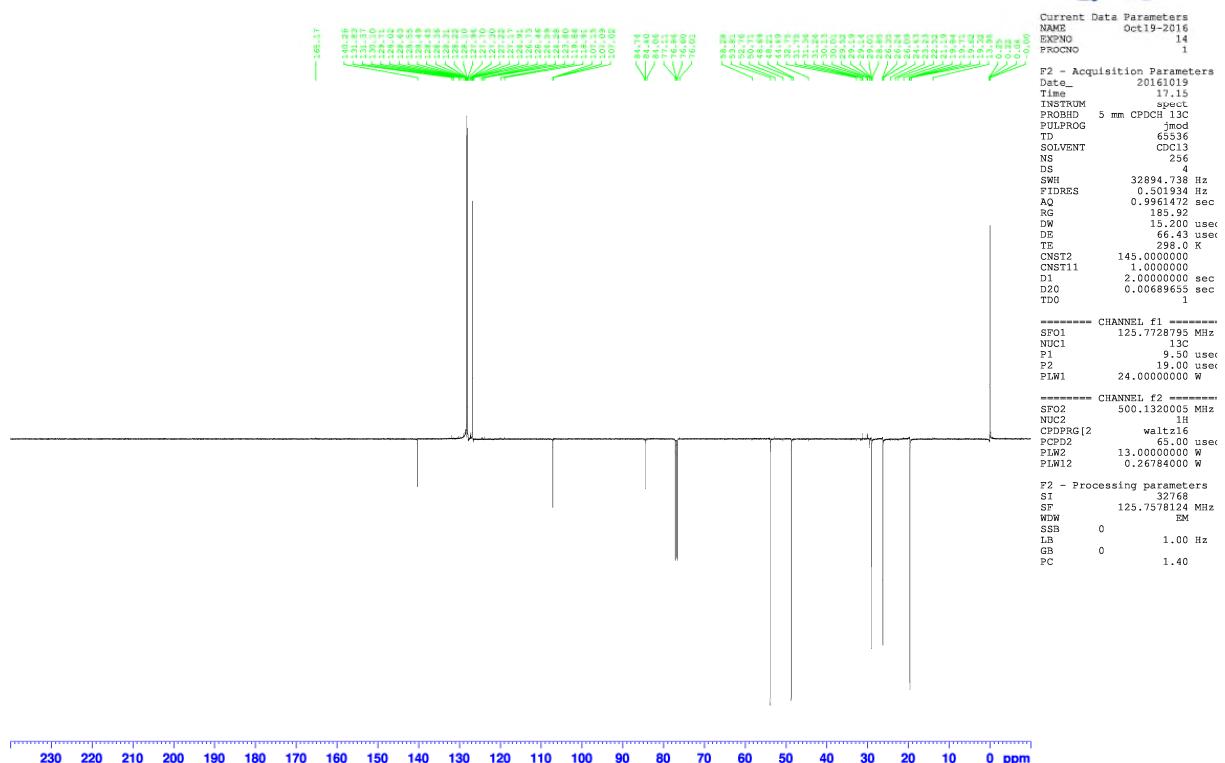


$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ).

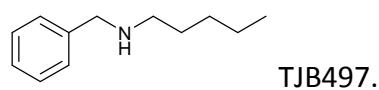


<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>).

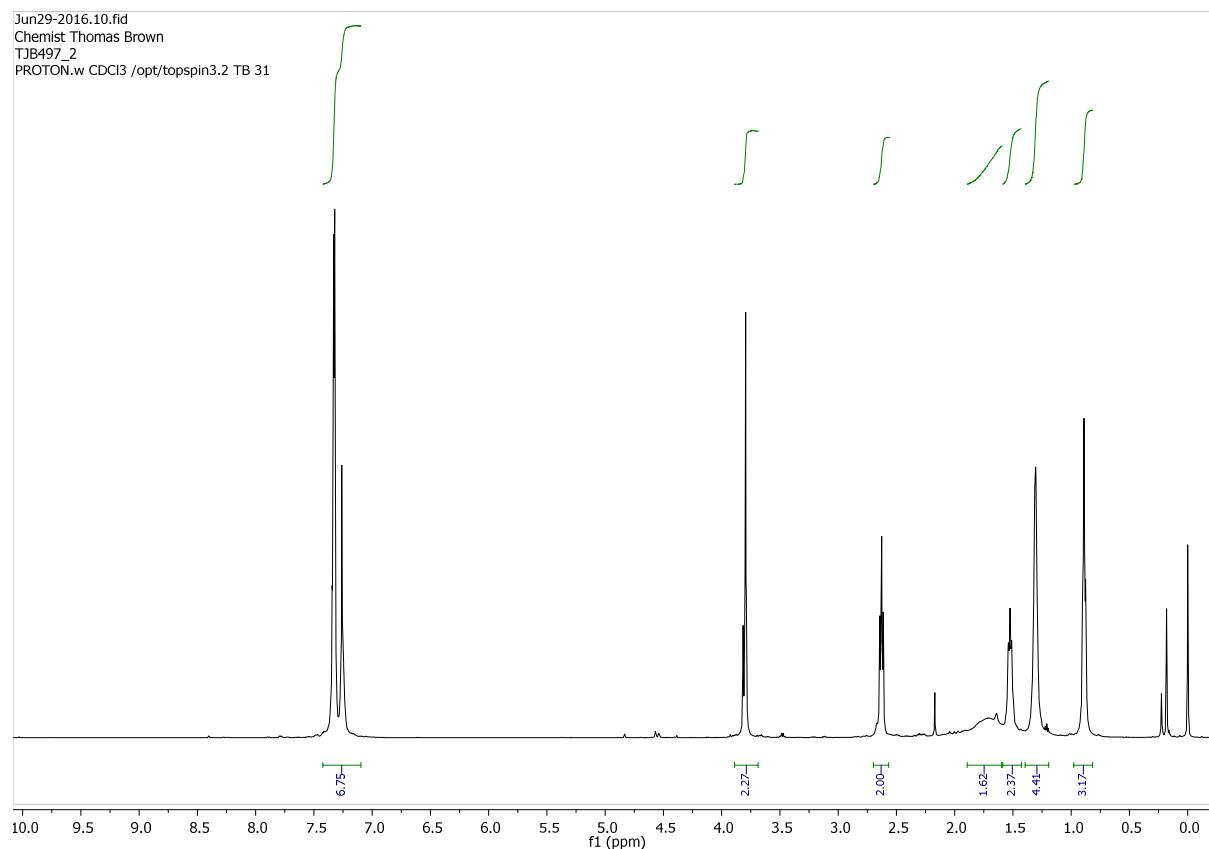
Chemist Thomas Brown  
TJB527-1\*  
C13APT.w CDCl<sub>3</sub> /opt/topspin3.2 TB 22



*N*-Benzylpentan-1-amine **43**.

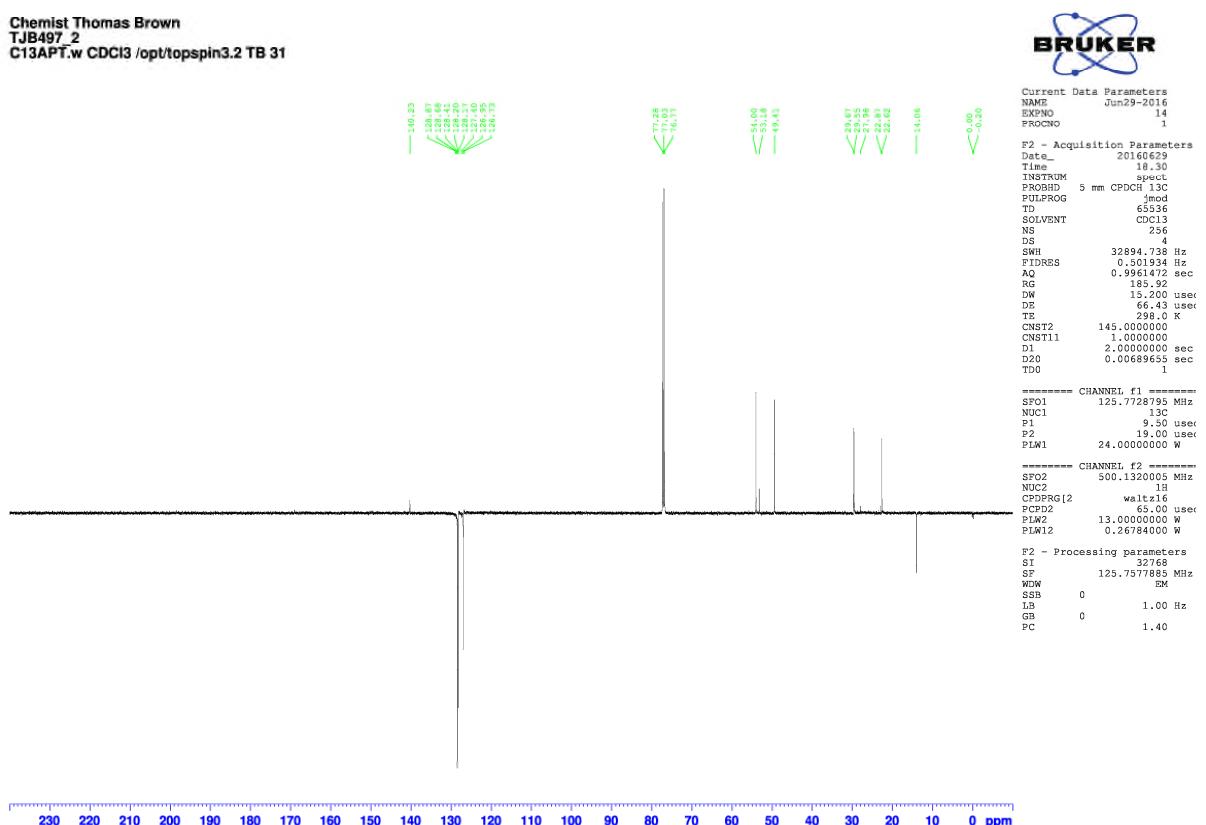


$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ).

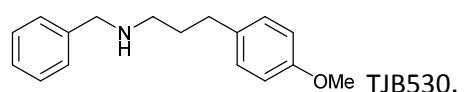


<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>).

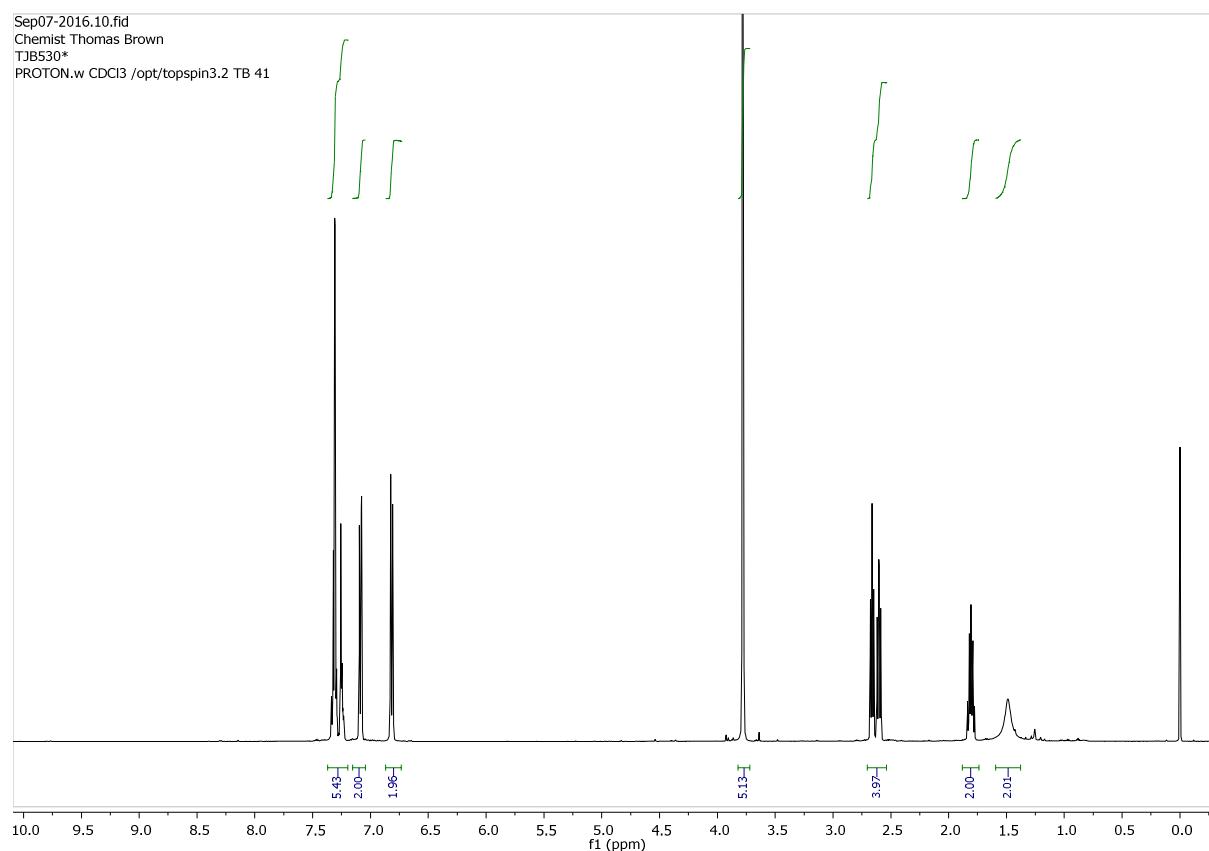
Chemist Thomas Brown  
TJB497\_2  
C13APT.w CDCl<sub>3</sub> /opt/topspin3.2 TB 31



*N*-Benzyl-3-(4-methoxyphenyl)propan-1-amine **44**.



$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ).



<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>).

Chemist Thomas Brown  
TJB530\*

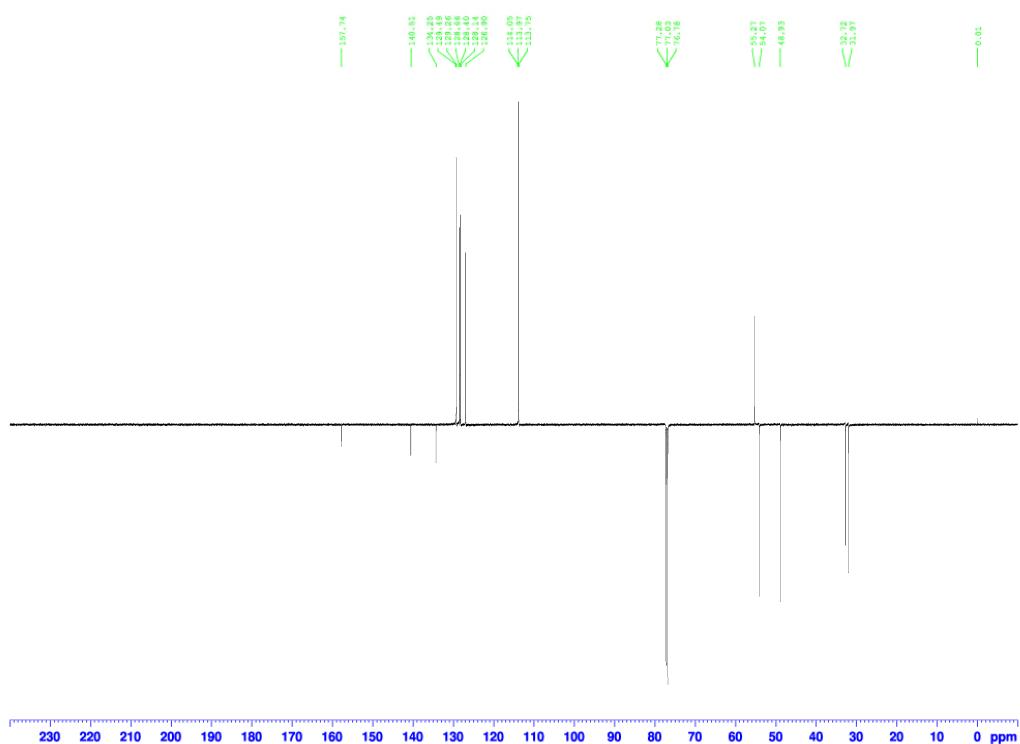


Current Data Parameters  
NAME Sep7-2016  
EXPNO 14  
PROCNO 1  
  
F2 - Acquisition Parameters  
Data\_2D 2016907  
Time\_ 16.51  
INSTRUM spect  
PROBHD 5 mm CPDCH DQCP  
PULPROG f2d  
TD 65536  
SOLVENT CDCl<sub>3</sub>  
NS 256  
DS 4  
SWH 32894.738 Hz  
FIDRES 0.501934 Hz  
AQ 0.9961472 sec  
RG 127  
DW 13.200 usec  
DE 66.43 usec  
TE 298.0 K  
CNUST2 145.000000 Hz  
CNST11 1.0000000  
D1 2.00000000 sec  
D20 0.00689655 sec  
TD0 1

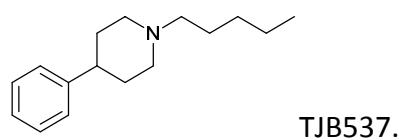
===== CHANNEL f1 =====  
SFO1 125.7728795 MHz  
NUC1 <sup>13</sup>C  
P1 5.0 usec  
P2 19.00 usec  
PLW1 24.00000000 W

===== CHANNEL f2 =====  
SFO2 500.1320005 MHz  
NUC2 <sup>1</sup>H  
CPDPGR2 waltz16  
PDPGR2 65.00 usec  
PLW2 13.00000000 W  
PLW12 0.26784000 W

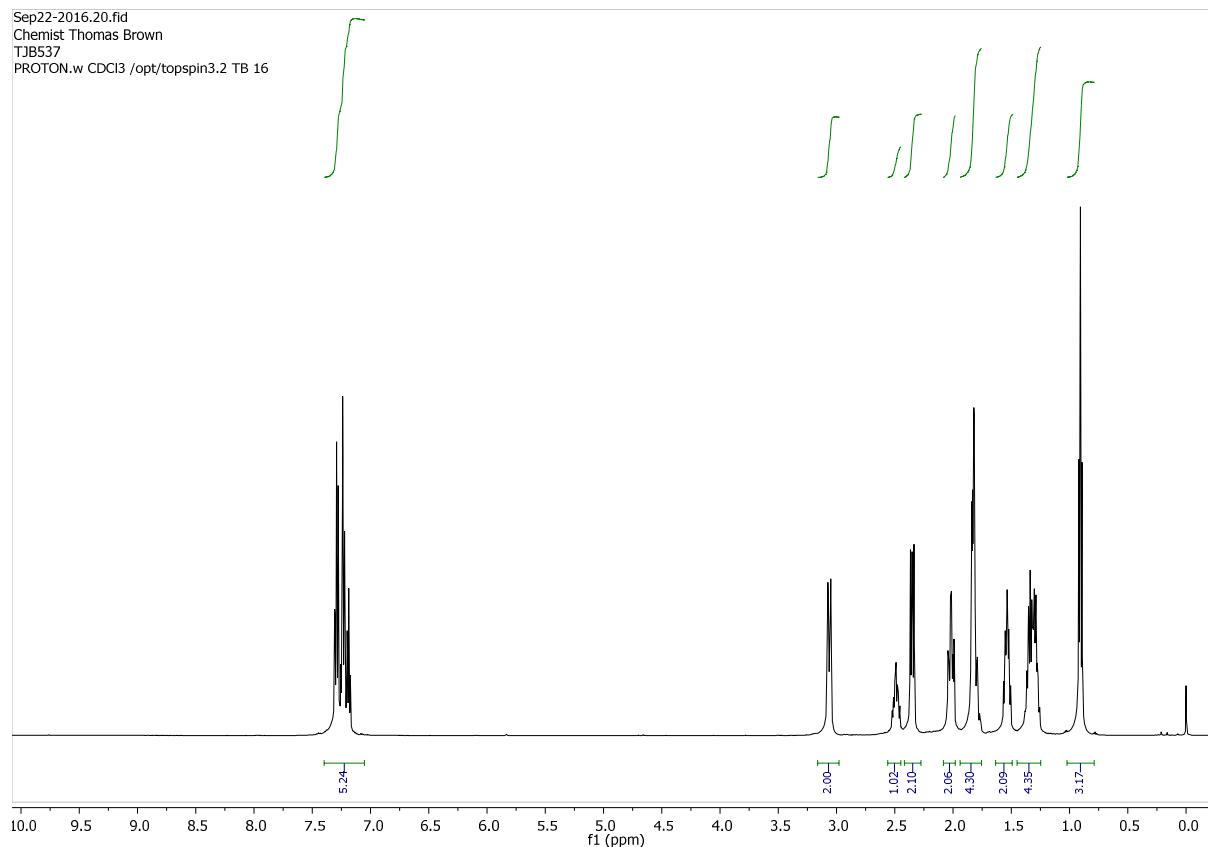
F2 - Processing parameters  
SI 32768  
SF 125.7577885 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40



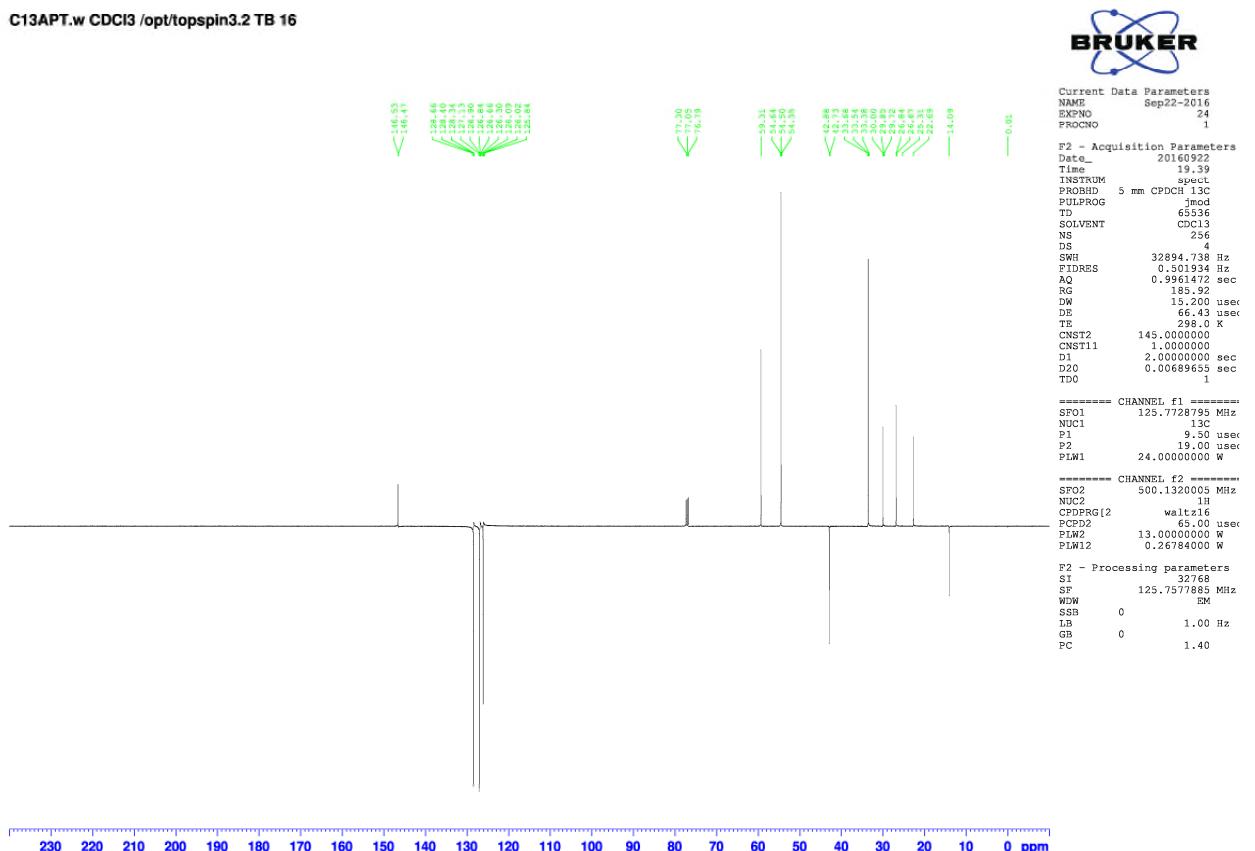
**1-Pentyl-4-phenylpiperidine 45.**



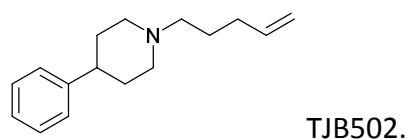
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ).



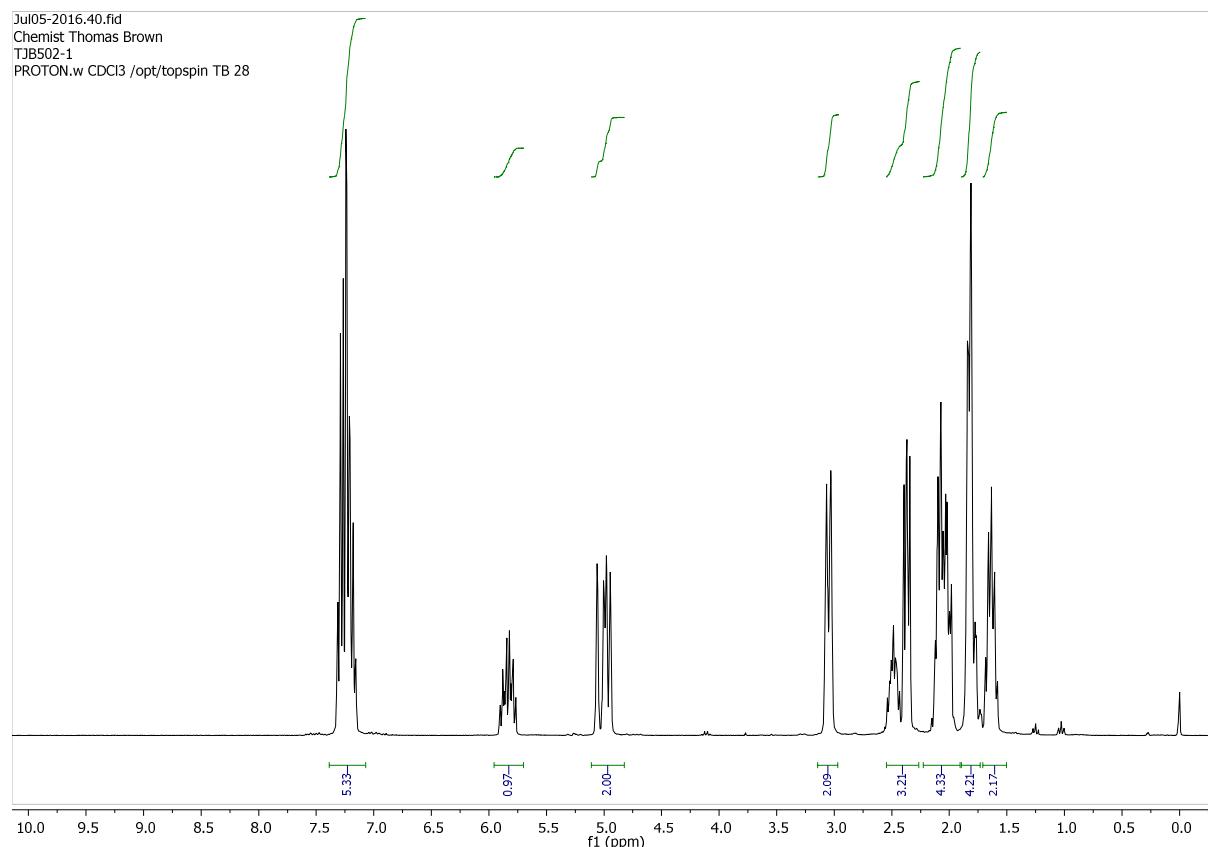
<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>).



**1-(Pent-4-en-1-yl)-4-phenylpiperidine **46**.**



$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ).



<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>).

Chemist Thomas Brown  
TJB502-1  
C13APT.w CDCl<sub>3</sub> /opt/topspin3.2 TB 13

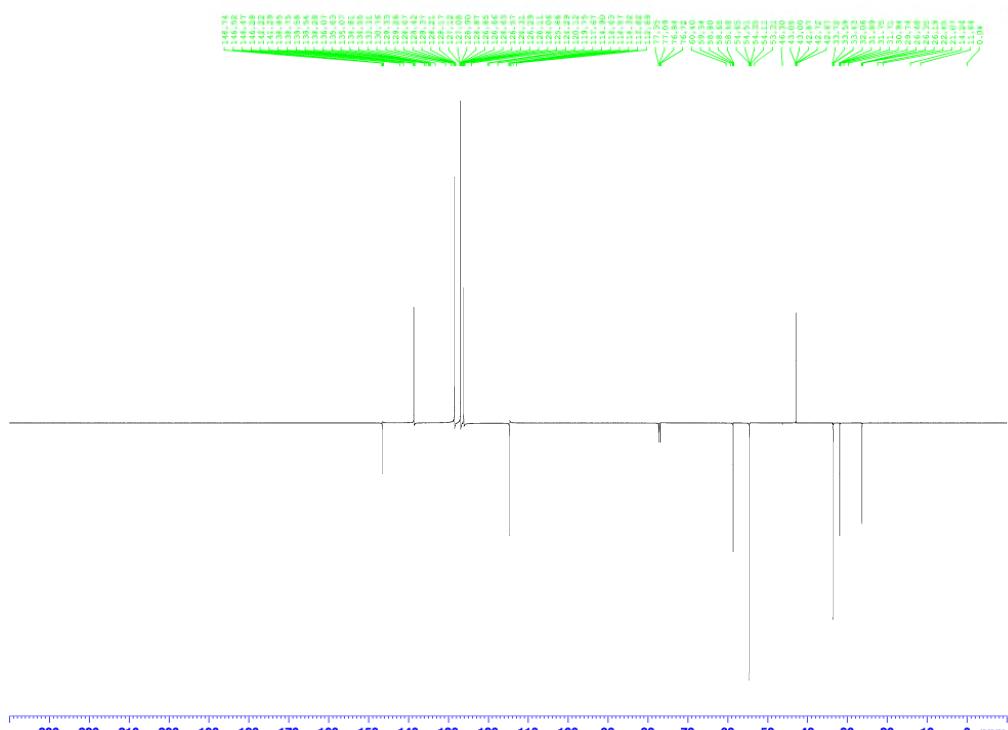


```
Current Data Parameters
NAME      Jul06-2016
EXPNO     14
PROCNO    1
P1        14.43
TD        65536
SOLVENT   CDCl3
NS         256
DS         4
SWH       32894.738 Hz
FIDRES   0.501934 Hz
AQ        0.983800 sec
RG        185.82
DW        15.200 usec
DE        66.43 usec
TE        29.58 K
CNS12    145.000000
CNS11    1.000000
D1        6.0000000 sec
D20      0.00689655 sec
TD0      1

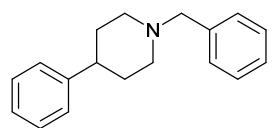
***** CHANNEL f1 *****
SFO1    125.728795 MHz
NUC1     1H
P1       9.50 usec
P2       19.00 usec
PLW1    24.0000000 W

***** CHANNEL f2 *****
SFO2    500.1320005 MHz
NUC2     1H
CPDPFG[2]  waltz16
P1Z2    60.00 usec
PLW2    13.0000000 W
PLW12   0.26784000 W

F2 - Acquisition parameters
SI        32768
SF       125.757785 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB      0
PC       1.40
```

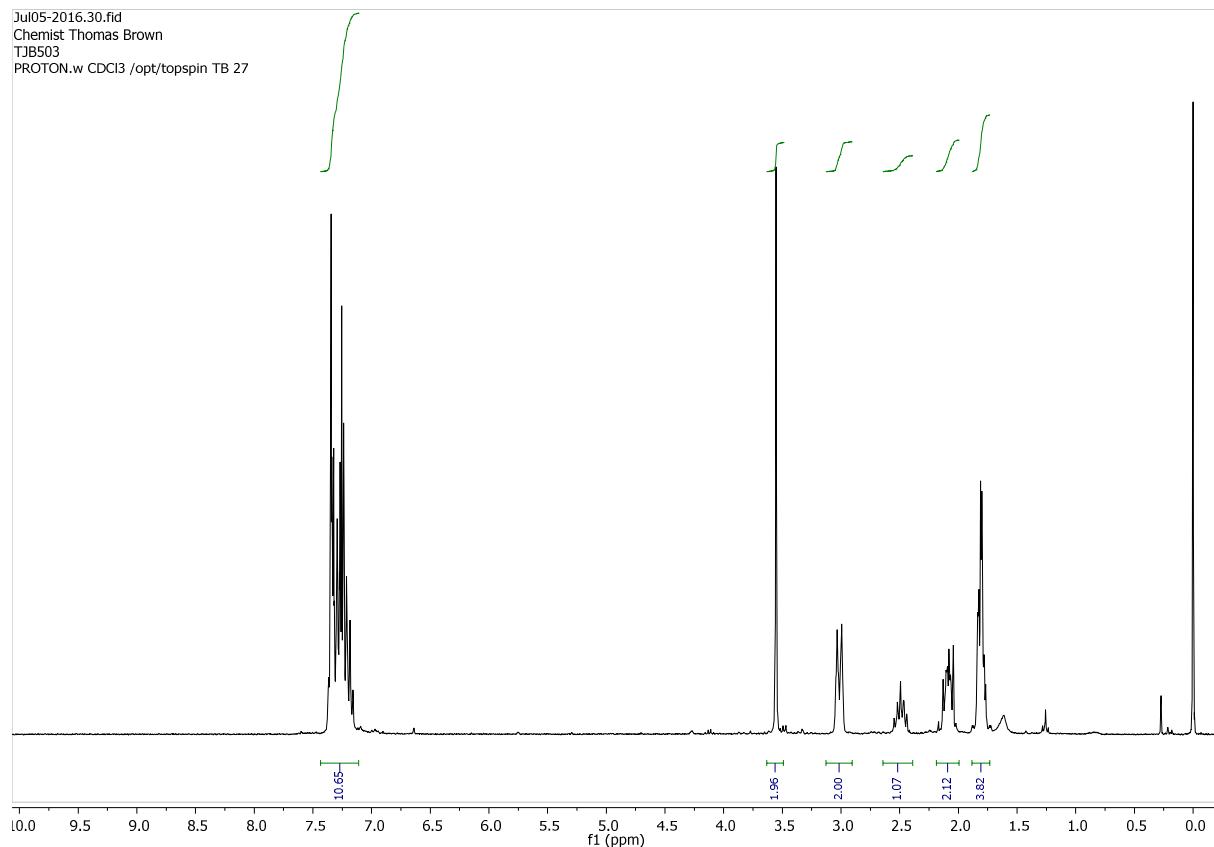


**1-Benzyl-4-phenylpiperidine 47.**



TJB503.

$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ).

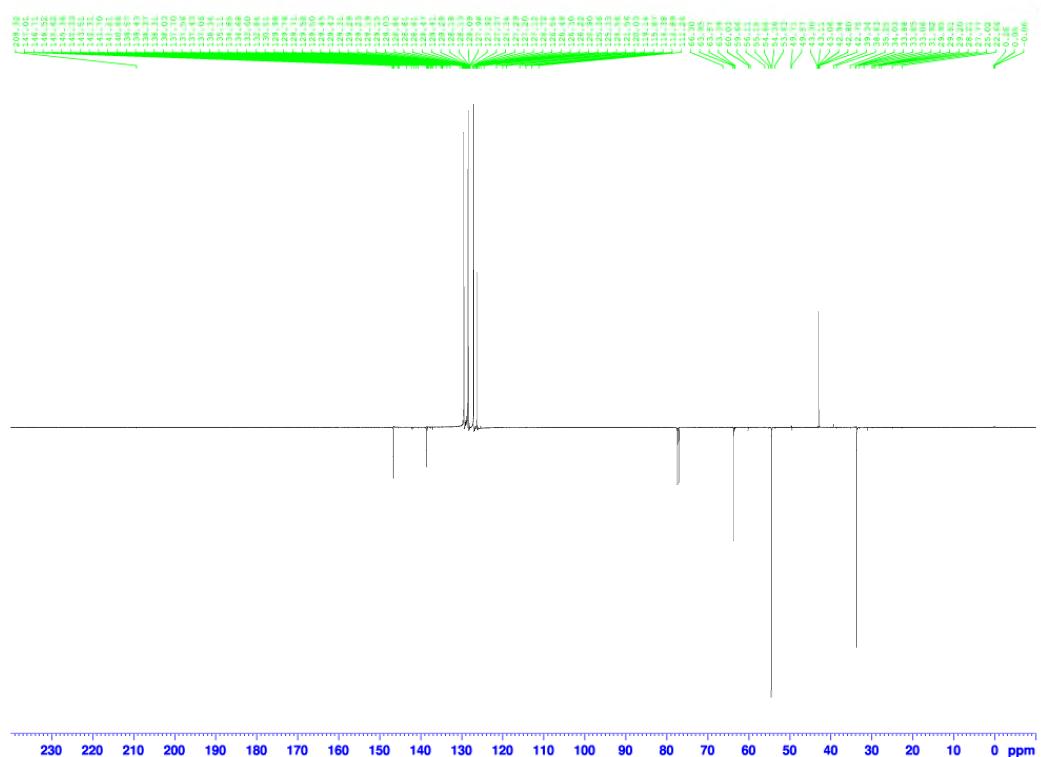


<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>).

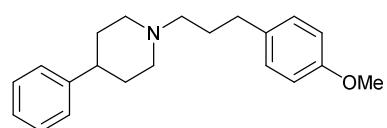
Chemist Thomas Brown  
TJB503  
C13APT.w CDCl<sub>3</sub> /opt/topspin3.2 TB 14



Current Data Parameters  
NAME: Jul106-2016  
EXPNO: 24  
PROCNO: 1  
  
F2 - Acquisition Parameters  
D1 - 2016.00 sec  
Time: 16.09  
INSTRUM: spect  
PROBHD: 5 mm CPDCH 13C  
PULPROG: jmd4  
TD: 65536  
SOLVENT: CDCl<sub>3</sub>  
NS: 256  
DS: 4  
SWB: 32894.76 Hz  
FIDRES: 0.501934 Hz  
AQ: 0.9961472 sec  
RG: 185.92  
DW: 15.40 usec  
DE: 66.45 usec  
TE: 298.0 K  
CNST2: 145.0000000  
CNST11: 1.0000000  
D1: 6.0000000 sec  
D2: 0.00639655 sec  
TD0:  
  
===== CHANNEL f1 =====  
SFO1: 125.7728795 MHz  
NUC1: 13C  
P1: 9.50 usec  
P2: 19.00 usec  
PLW1: 24.0000000 W  
  
===== CHANNEL f2 =====  
SFO2: 500.1320005 MHz  
NUC2: 1H  
CPDPRG[2]: waltz16  
PCPD2: 65.00 usec  
PLW2: 13.0000000 W  
PLW12: 0.26784000 W  
  
F2 - Processing parameters  
SI: 32768  
SF: 125.7577661 MHz  
WDW:  
SSB: 0  
LB: 1.00 Hz  
GB: 0  
PC: 1.40

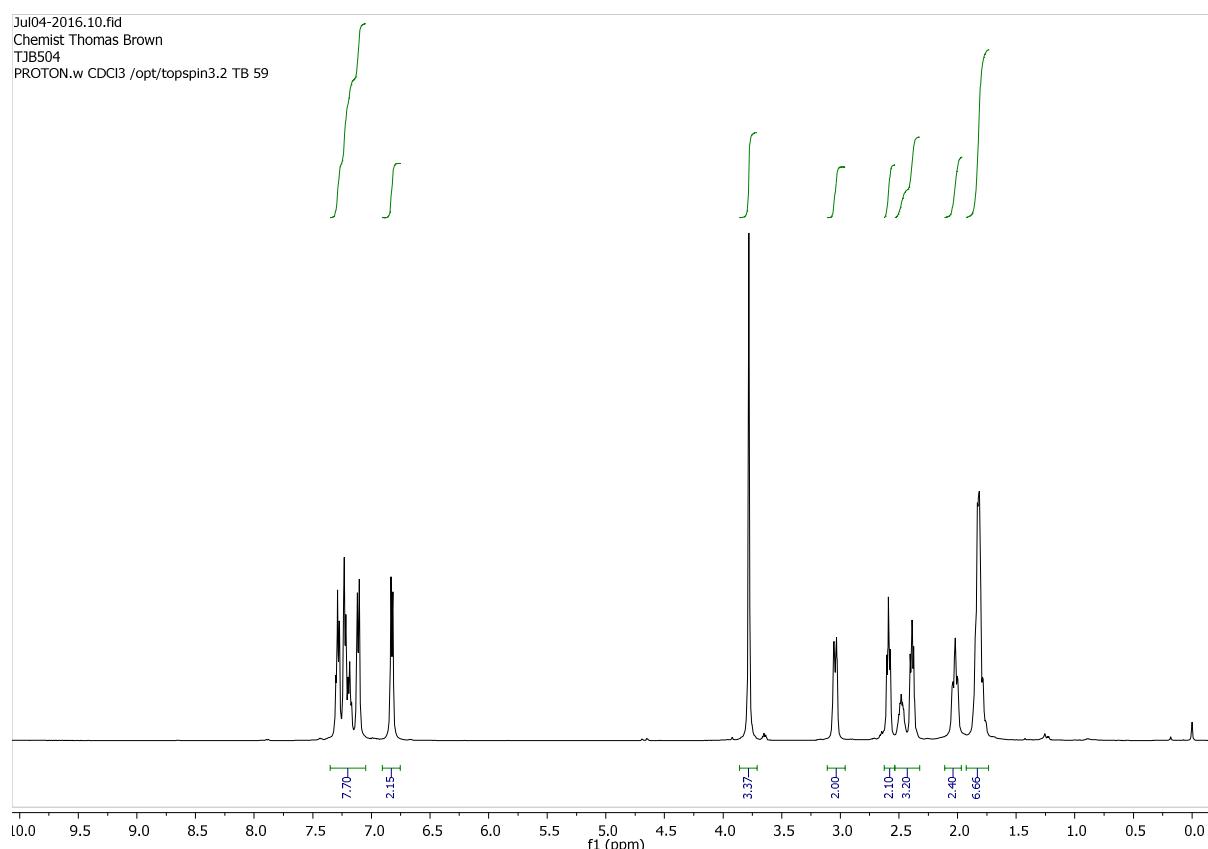


1-(3-(4-Methoxyphenyl)propyl)-4-phenylpiperidine **48**.



TJB504.

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ).

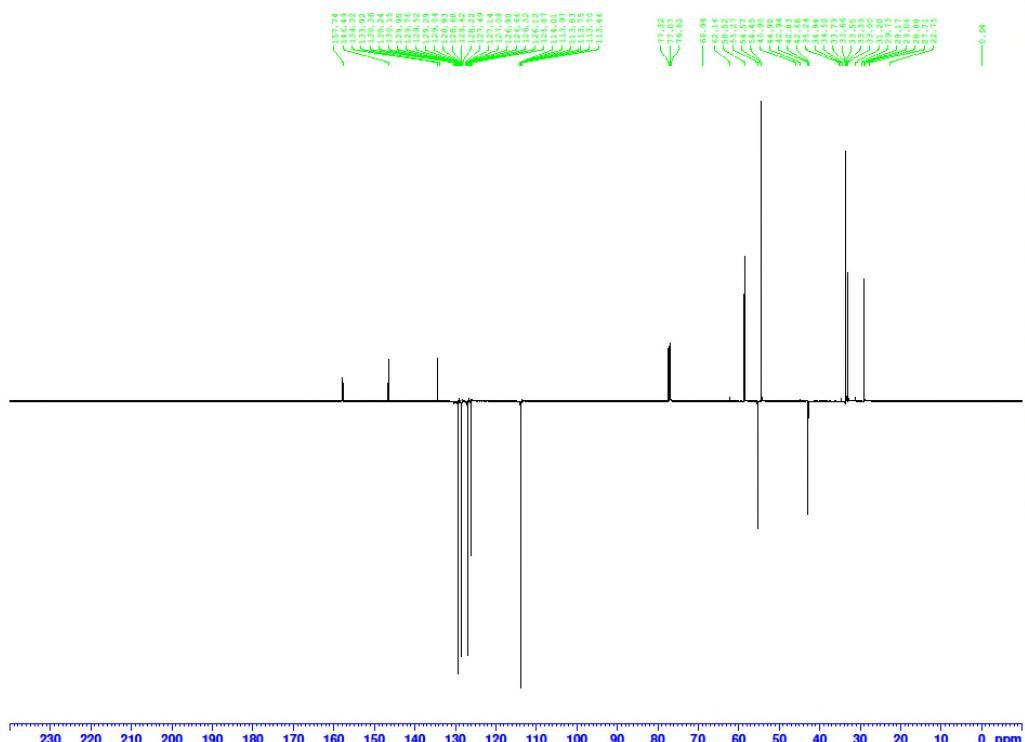


<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>).

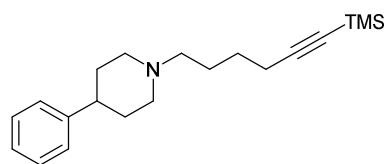
C13APT.w CDCl<sub>3</sub> /opt/topspin3.2 TB 59



Current Data Parameters  
 NAME Ju104-2016  
 EXPNO 14  
 PROCNO 1  
  
 F2 - Acquisition Parameters  
 Data\_ 20160704  
 Time 18.56  
 INSTRUM spect  
 PROBHD 5 mm CPDMR13C  
 PULPROG jmod  
 TD 65536  
 SOLVENT CDCl<sub>3</sub>  
 NS 256  
 DS 4  
 SWH 32894.738 Hz  
 FIDRES 0.501934 Hz  
 AQ 0.9961472 sec  
 RG 185.92  
 DW 15.00 usec  
 DE 66.43 usec  
 TE 298.0 K  
 CNSTT2 145.000000  
 CNSTT11 1.000000  
 D1 2.0000000 sec  
 D20 0.00689655 sec  
 TDO 1  
  
 ----- CHANNEL f1 -----  
 SF01 125.7728795 MHz  
 NUC1 13C  
 P1 9.50 usec  
 P2 19.00 usec  
 PLW1 24.0000000 W  
  
 ----- CHANNEL f2 -----  
 SF02 500.1320009 MHz  
 NUC2 1H  
 CPDPRG[2 waltz16  
 PCPD2 65.00 usec  
 PLW2 13.00000000 W  
 PLW2 0.267840000 W  
  
 F2 - Processing parameters  
 ST 32768  
 SF 125.7577885 MHz  
 WM 2M  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

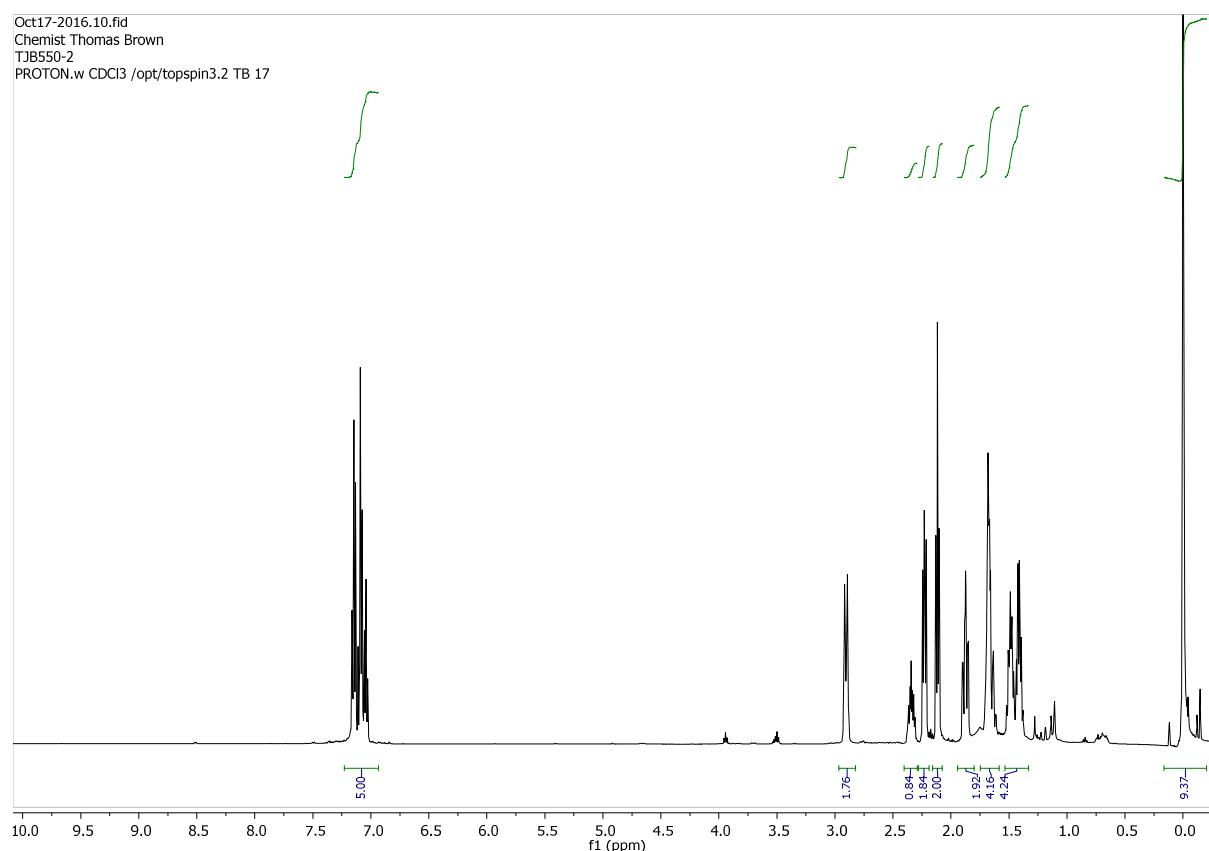


4-Phenyl-1-(6-(trimethylsilyl)hex-5-yn-1-yl)piperidine **49**.

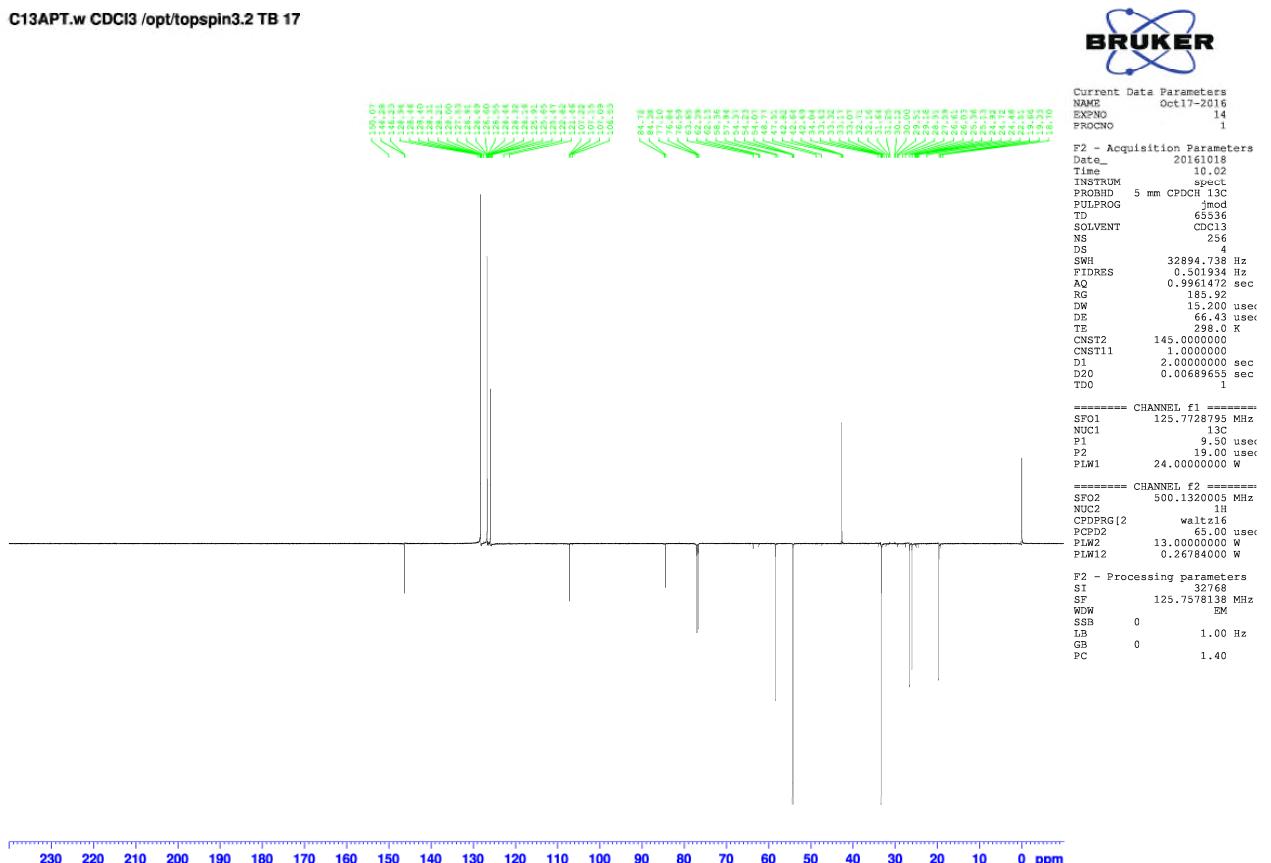


TJB550.

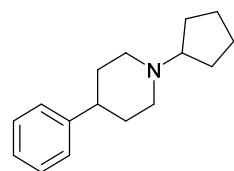
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ).



<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>).

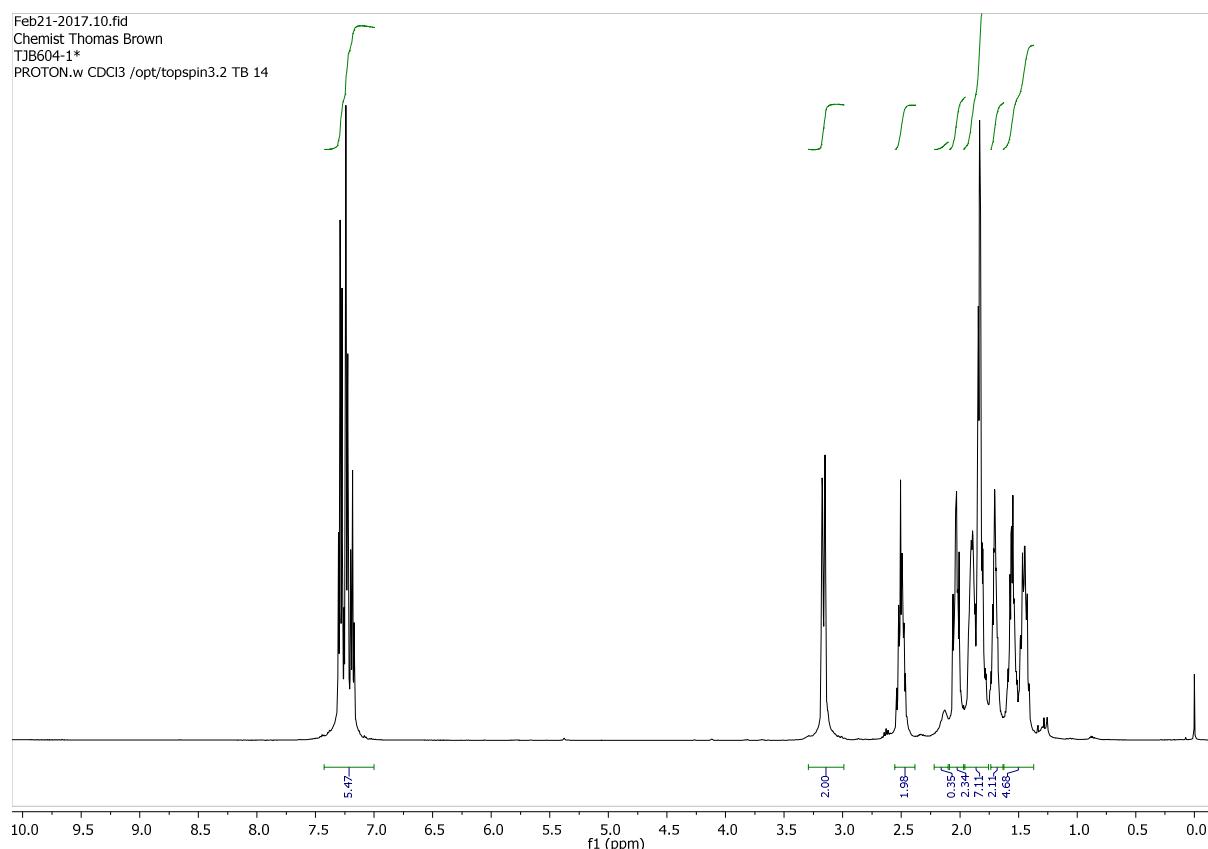


1-Cyclopentyl-4-phenylpiperidine **50**.



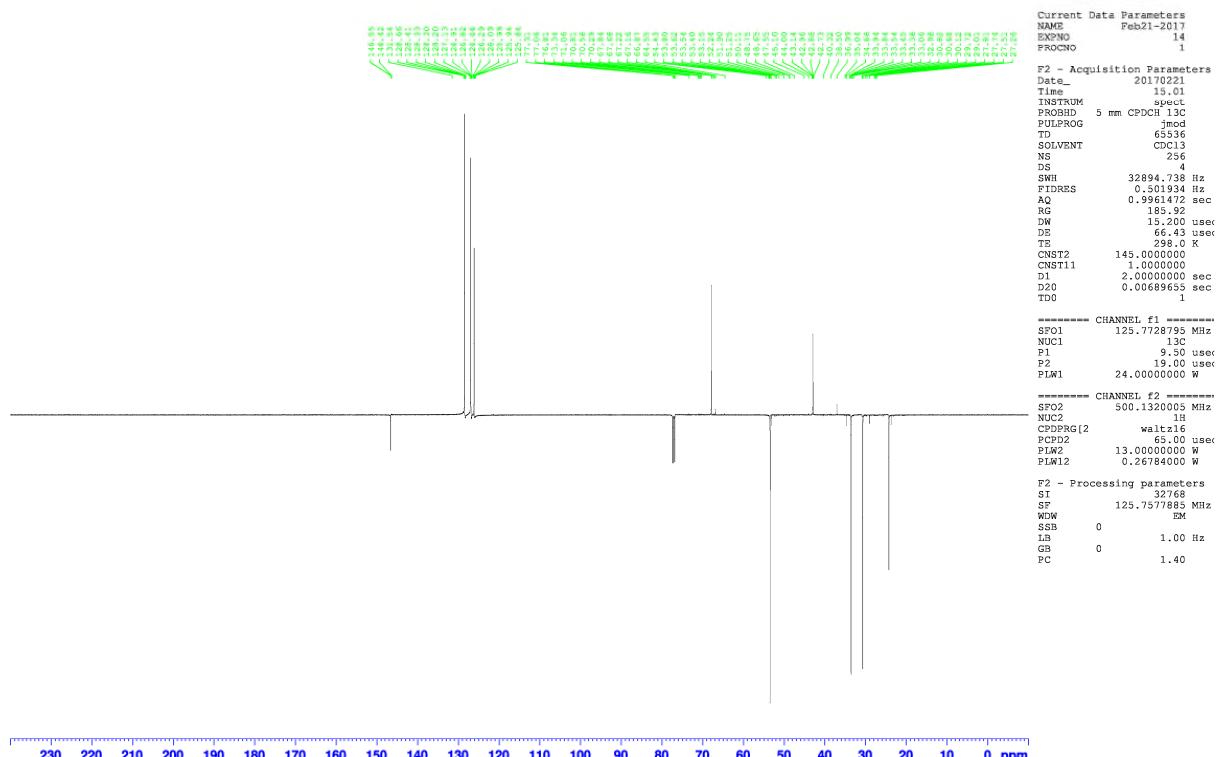
TJB604.

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ).



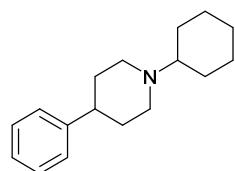
<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>).

Chemist Thomas Brown  
TJB604-1\*  
C13APT.w CDCl<sub>3</sub> /opt/topspin3.2 TB 14



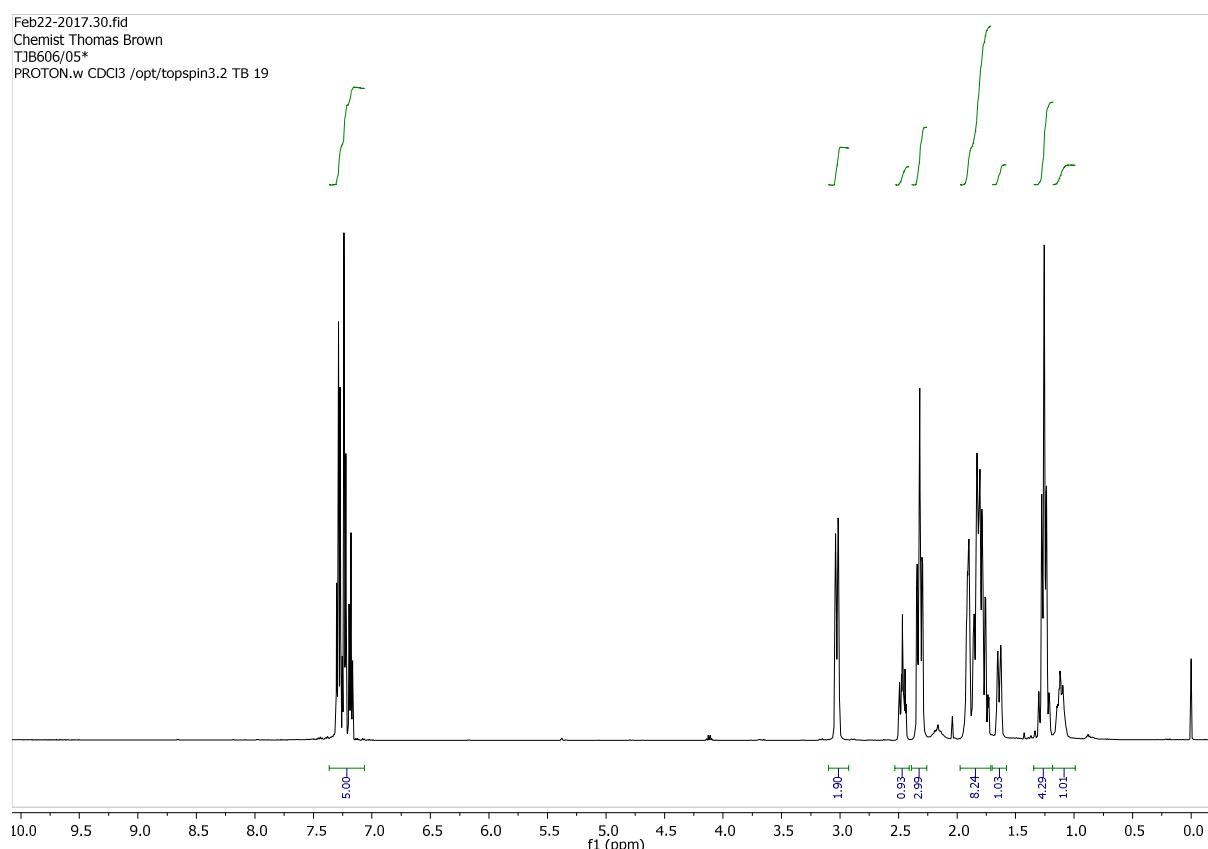
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

1-Cyclohexyl-4-phenylpiperidine **51**.



TJB605.

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ).



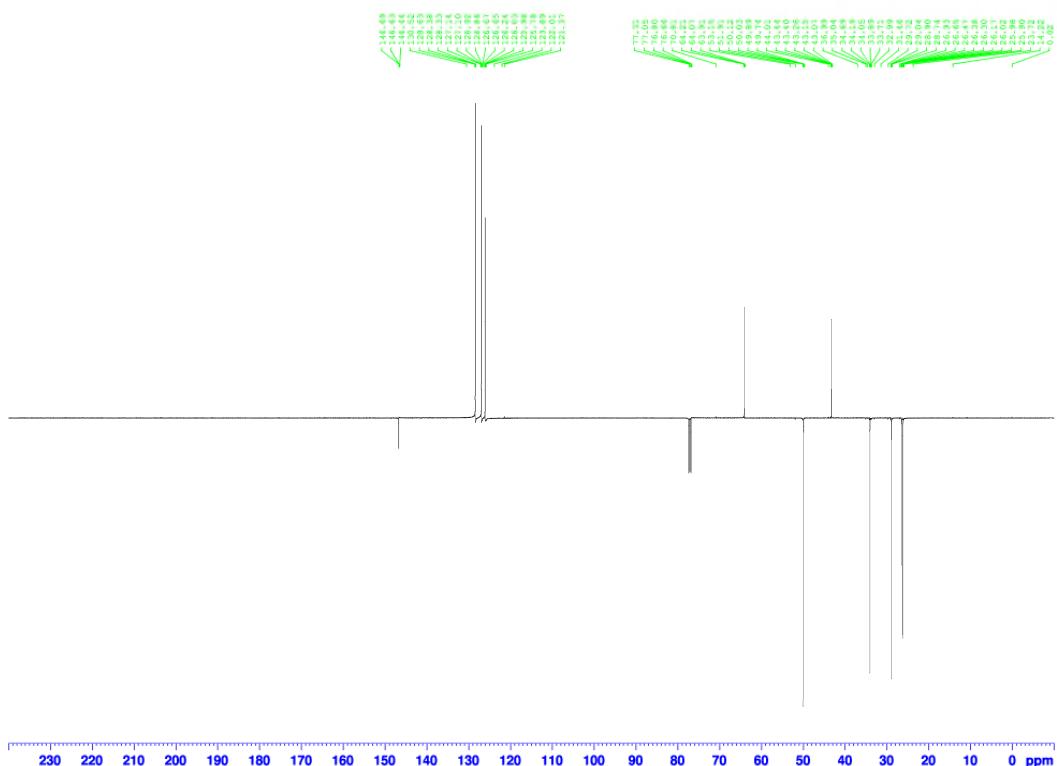
<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>).

Chemist Thomas Brown  
TJB606/05\*  
C13APT.w CDCl<sub>3</sub> /opt/topspin3.2 TB 19

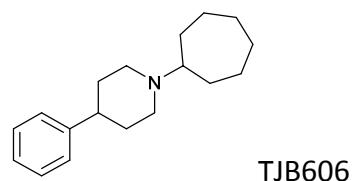


Current Data Parameters  
NAME Feb22-2017  
EXPNO 34  
PROCNO 1  
  
P2 - Acquisition Parameters  
Date\_ 20170222  
Time\_ 16.10  
INSTRUM w90  
PROBHD 5 mm CPDCH 13C  
PULPROG jmod  
TD 65536  
SOLVENT CDCl<sub>3</sub>  
NS 256  
DS 4  
SWH 32894.738 Hz  
TPRDES 0.501934 Hz  
AQ 0.999 sec  
RG 185.92  
DW 15.200 usec  
DE 66.43 usec  
TE 298.0 K  
CNST11 145.000000  
CNST11 1.0000000  
D1 2.0000000 sec  
D20 0.00689655 sec  
TD0 1

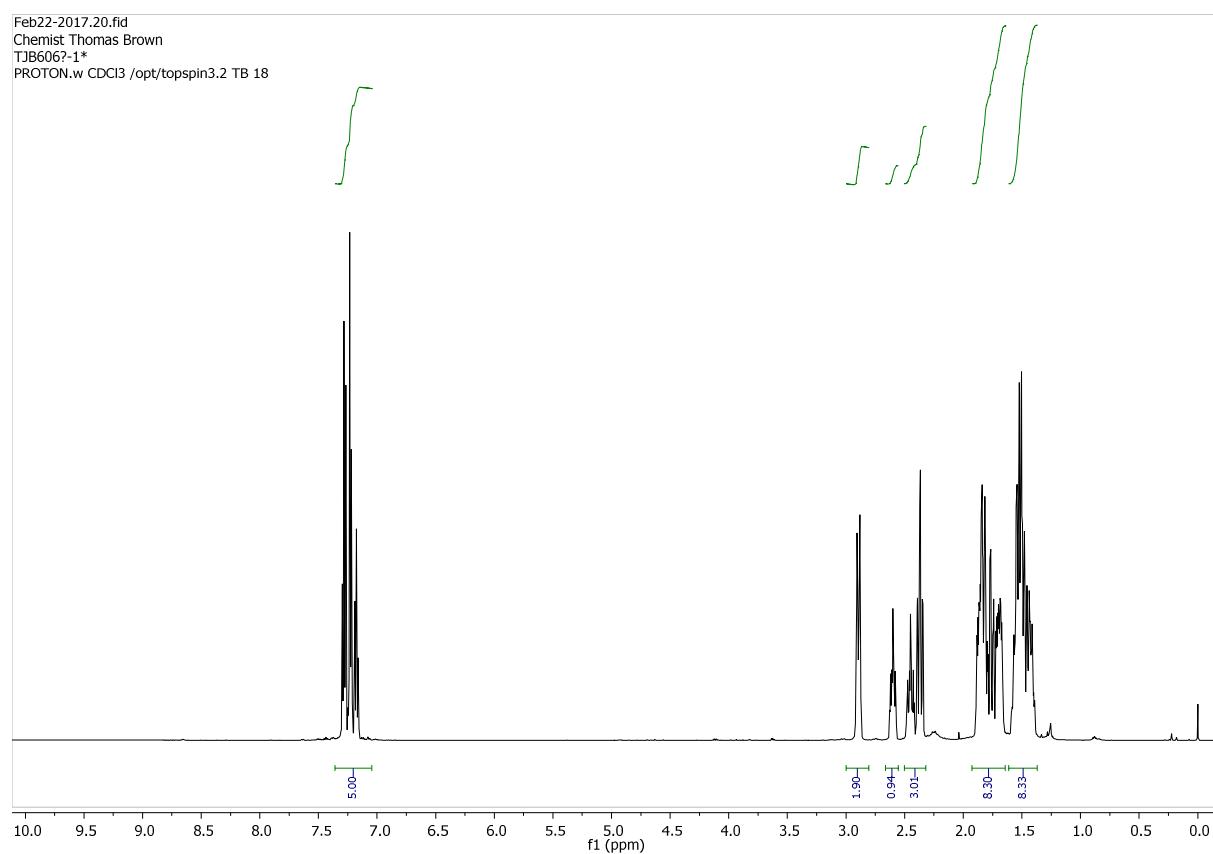
===== CHANNEL f1 =====  
SFO1 125.728795 MHz  
NUC1 13C  
P1 9.50 usec  
P2 19.00 usec  
PLW1 24.0000000 W  
  
===== CHANNEL f2 =====  
SFO1 500.1320000 MHz  
NUC1 1H  
CPDPG12 waltz16  
PCPD2 65.00 usec  
PLW1 13.0000000 W  
PLW12 0.26784000 W  
  
P2 - Processing parameters  
SI 32768  
SF 125.7577885 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40



1-Cycloheptyl-4-phenylpiperidine **52**.



$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ).

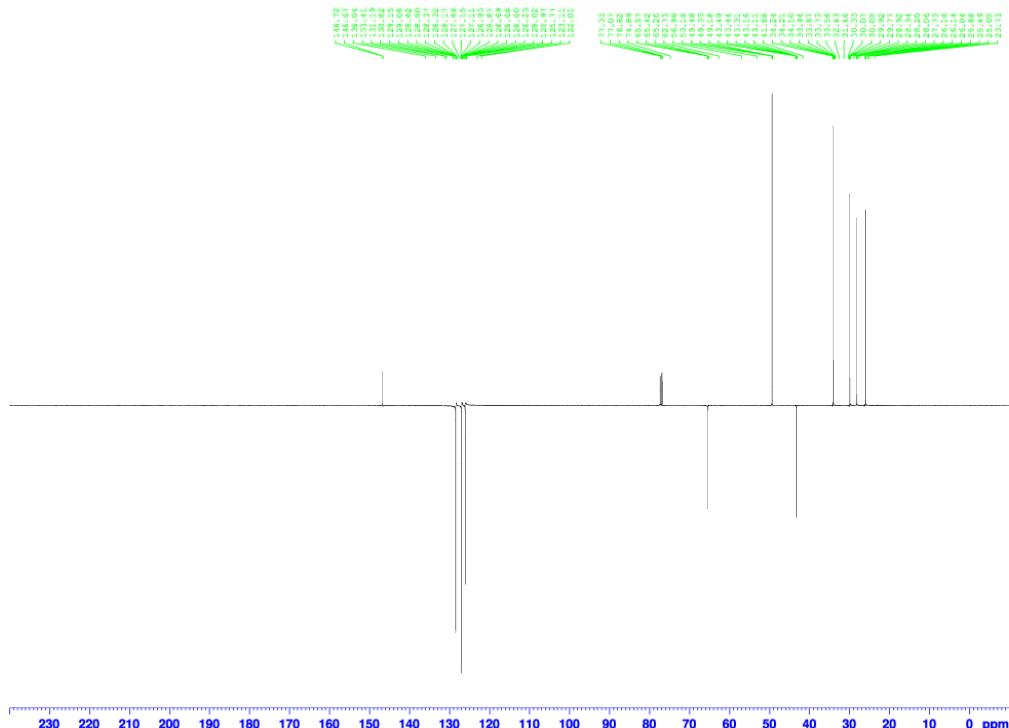


<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>).

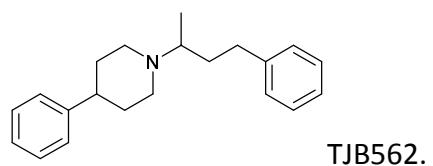
C13APT.w CDCl<sub>3</sub> /opt/topspin3.2 TB 18



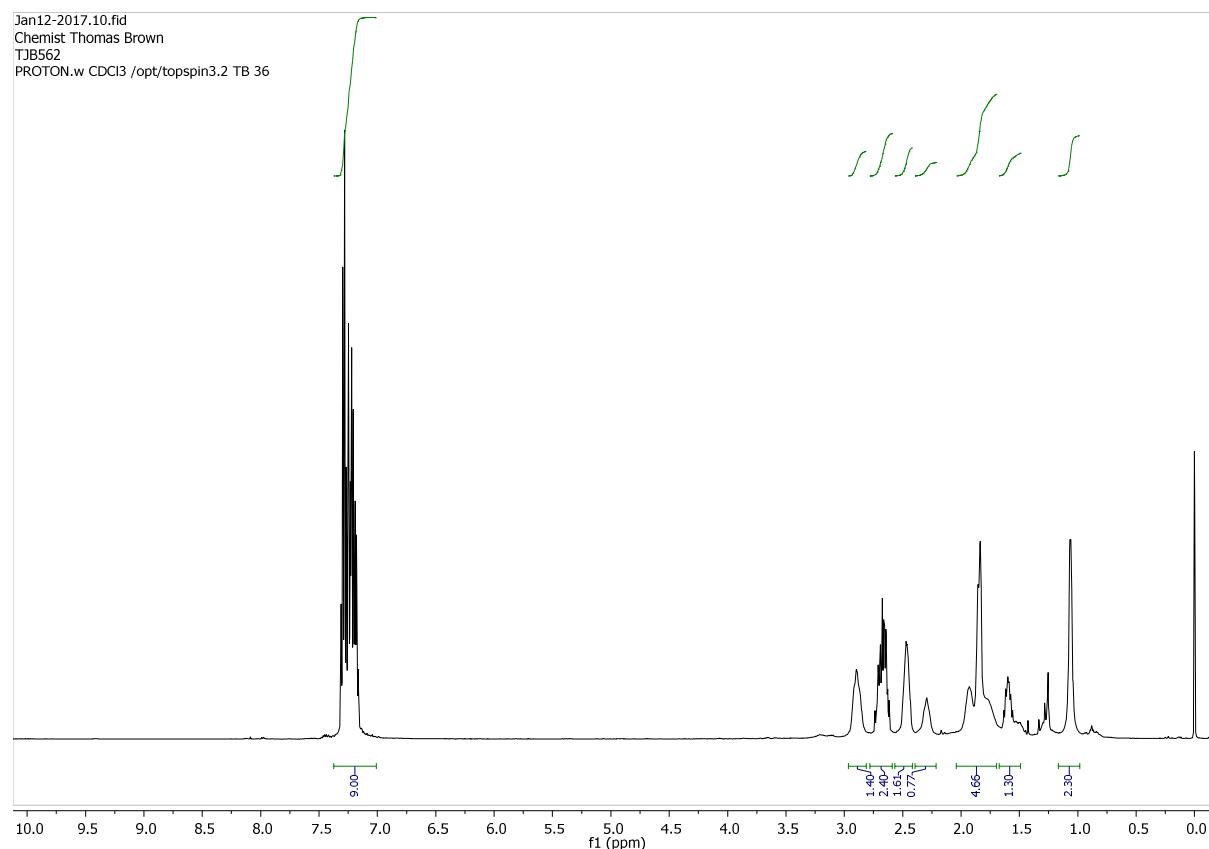
Current Data Parameters  
 NAME Feb22-2017  
 EXPNO 24  
 PROCNO 1  
  
 F2 - Acquisition Parameters  
 Date 20170222  
 Time 15.01  
 INSTRUM spect  
 PROBODIM 5 mm CPDCH3  
 PULPROG jmod  
 TD 65536  
 SOLVENT CDCl3  
 NS 256  
 DS 4  
 SWH 32894.738 Hz  
 FIDRES 0.501934 Hz  
 AQ 0.95144 sec  
 RG 18.92  
 DW 15.200 used  
 DE 66.43 used  
 TS 2.00 K  
 CNTS2 145.000000  
 CNST11 1.0000000  
 D1 2.0000000 sec  
 D20 0.00689655 sec  
 TDO 1  
  
 ===== CHANNEL f1 =====  
 SF01 125.7728795 MHz  
 NUC1 1H  
 P1 9.50 used  
 P2 19.00 used  
 PLW1 24.0000000 W  
  
 ===== CHANNEL f2 =====  
 SF02 500.1320005 MHz  
 NUC2 1H  
 CCP2 waltz16  
 P1 0.00 used  
 PLW2 13.0000000 W  
 PLW12 0.26784000 W  
  
 F2 - Processing parameters  
 SI 32768  
 SF 125.7577885 MHz  
 WDW 0 EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40



4-Phenyl-1-(4-phenylbutan-2-yl)piperidine **53**.



<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>).



<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>).

Chemist Thomas Brown  
TJB562  
C13APT.w CDCl<sub>3</sub> /opt/topspins.2 TB 25



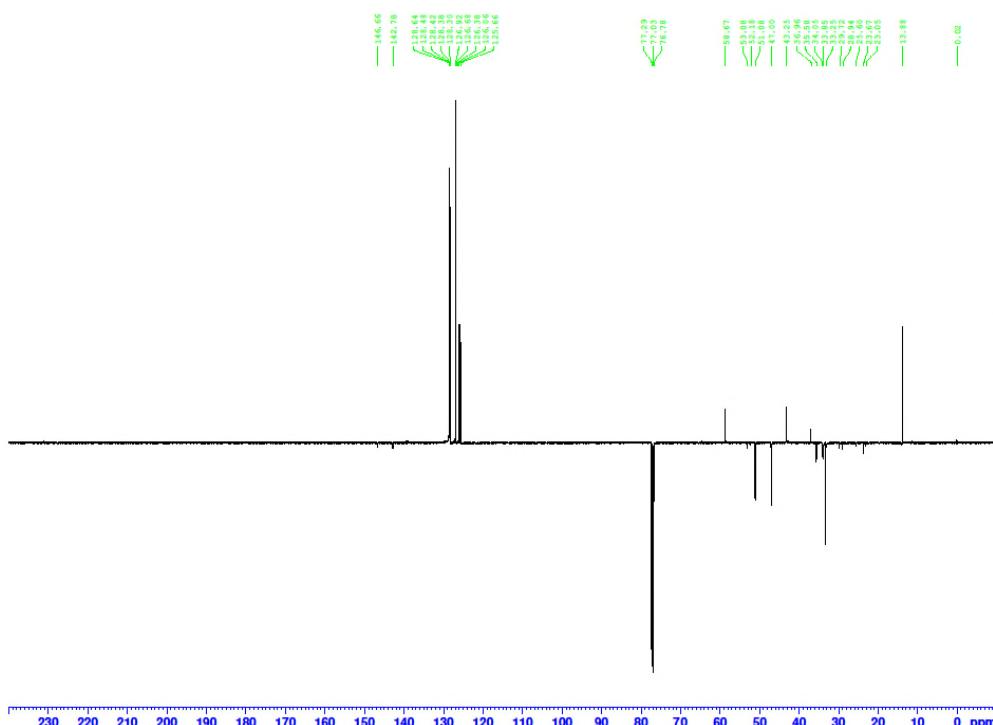
```
Current Data Parameters
NAME      Decl16-2016
EXPNO    14
PROCNO   1

F2 - Acquisition Parameters
Date_   20161216
Time    12.29
DURATION 60.000 sec
PROBHD  5 mm CPDPCH 13C
PULPROG jmod
TD      65536
SOLVENT  CDCl3
NUC1    13C
NS      256
DS      4
SWH    32894.738 Hz
FIDRES 0.553125 Hz
TD0    0.001472 sec
RG      185.92
DW      15.200 usec
DE      1.200 usec
TE      299.0 K
CNS1T2  145.000000
CNS1T1  1.000000
DT      2.0000000 sec
D20    0.0069655 sec
IDO    1

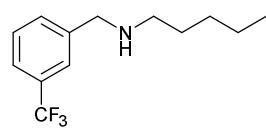
CHANNEL F1
SF01  125.7728795 MHz
NUC1  13C
P1    9.50 usec
P2    19.00 usec
PLW1  24.0000000 W

CHANNEL F2
SF02  500.1320095 MHz
NUC2  1H
CPDPG[2  waltz16
PCPD2  65.00 usec
PLW2  13.0000000 W
PLW12  0.26784000 W

F2 - Processing parameters
SI      32768
SF      125.7728785 MHz
WDW    EM
SSB    0
LB      1.00 Hz
GB      0
PC      1.40
```

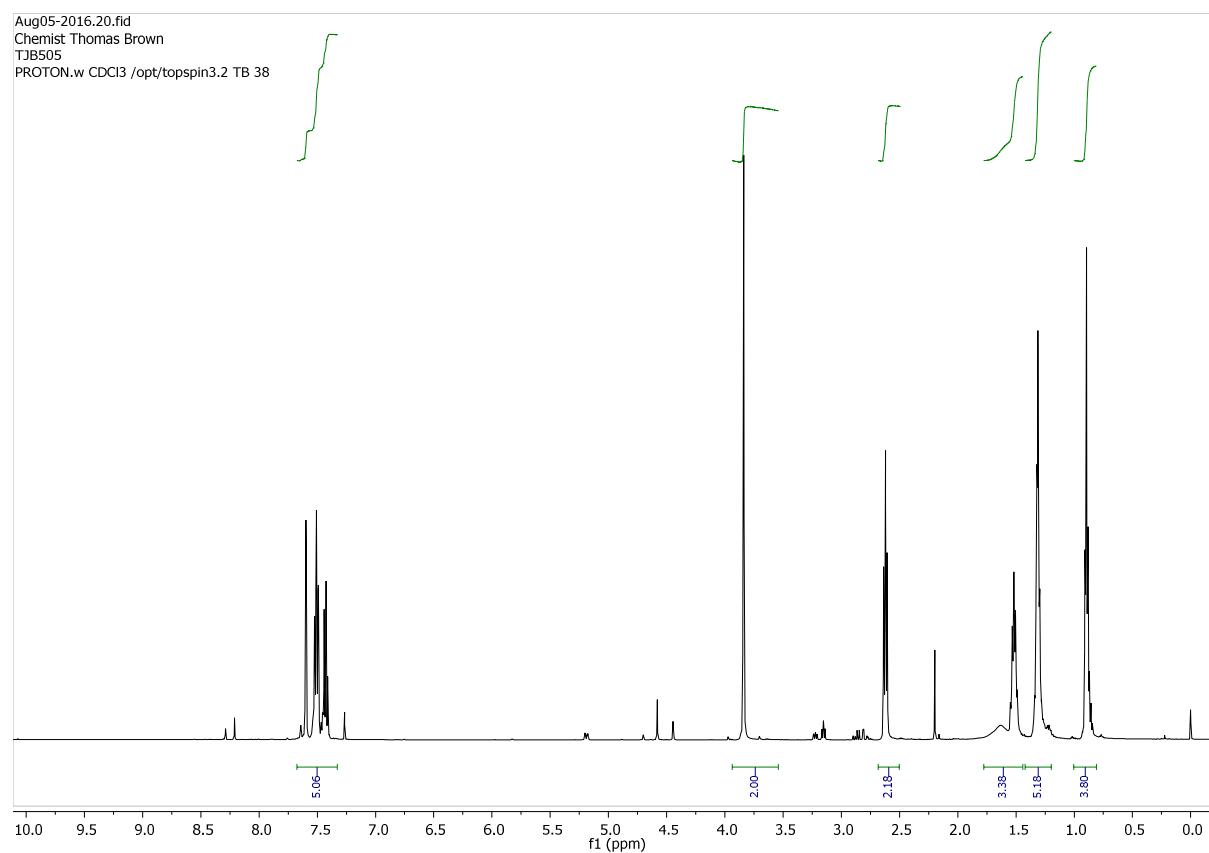


*N*-(3-(Trifluoromethyl)benzyl)pentan-1-amine **54**.

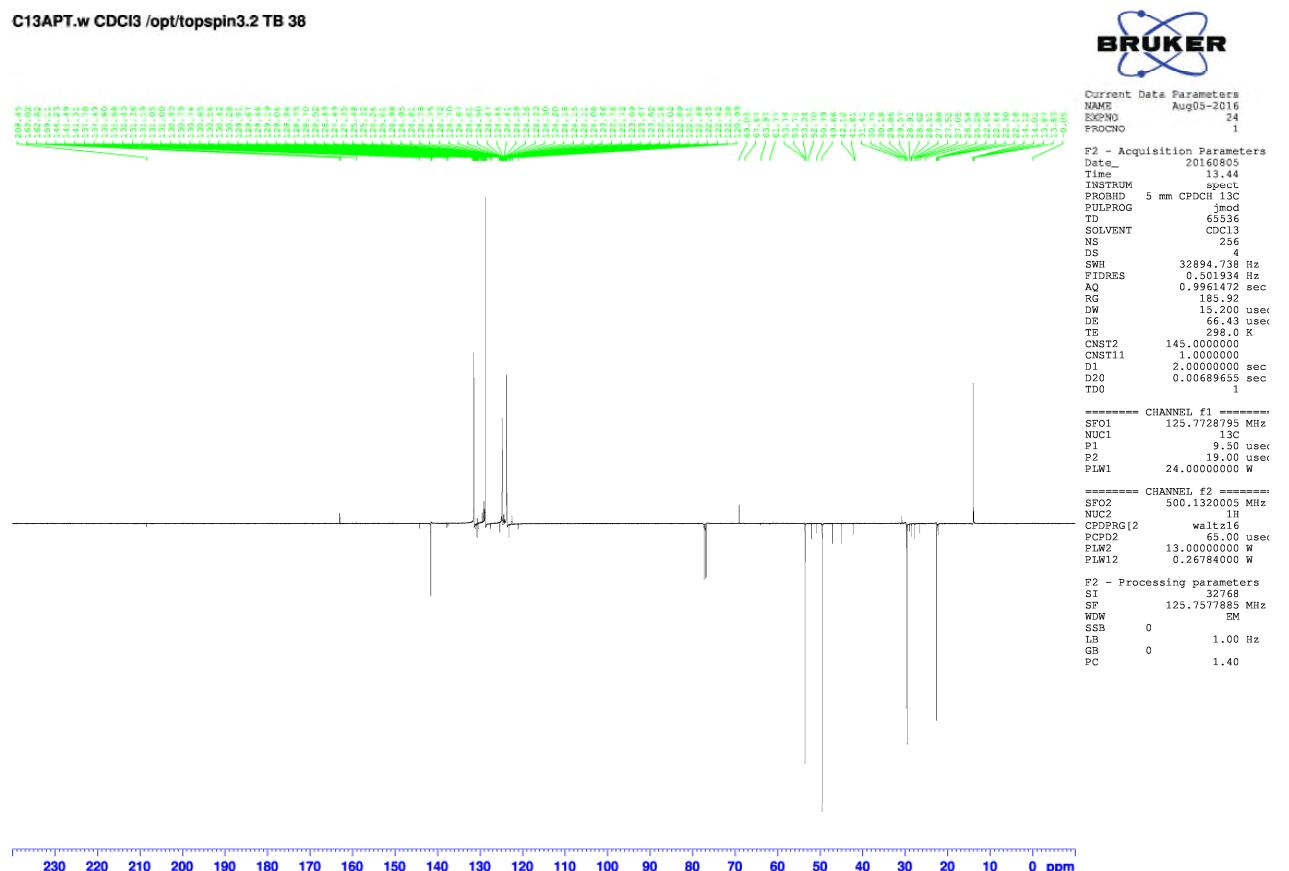


TJB505

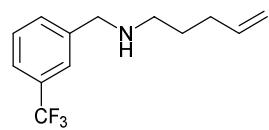
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ).



<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>).

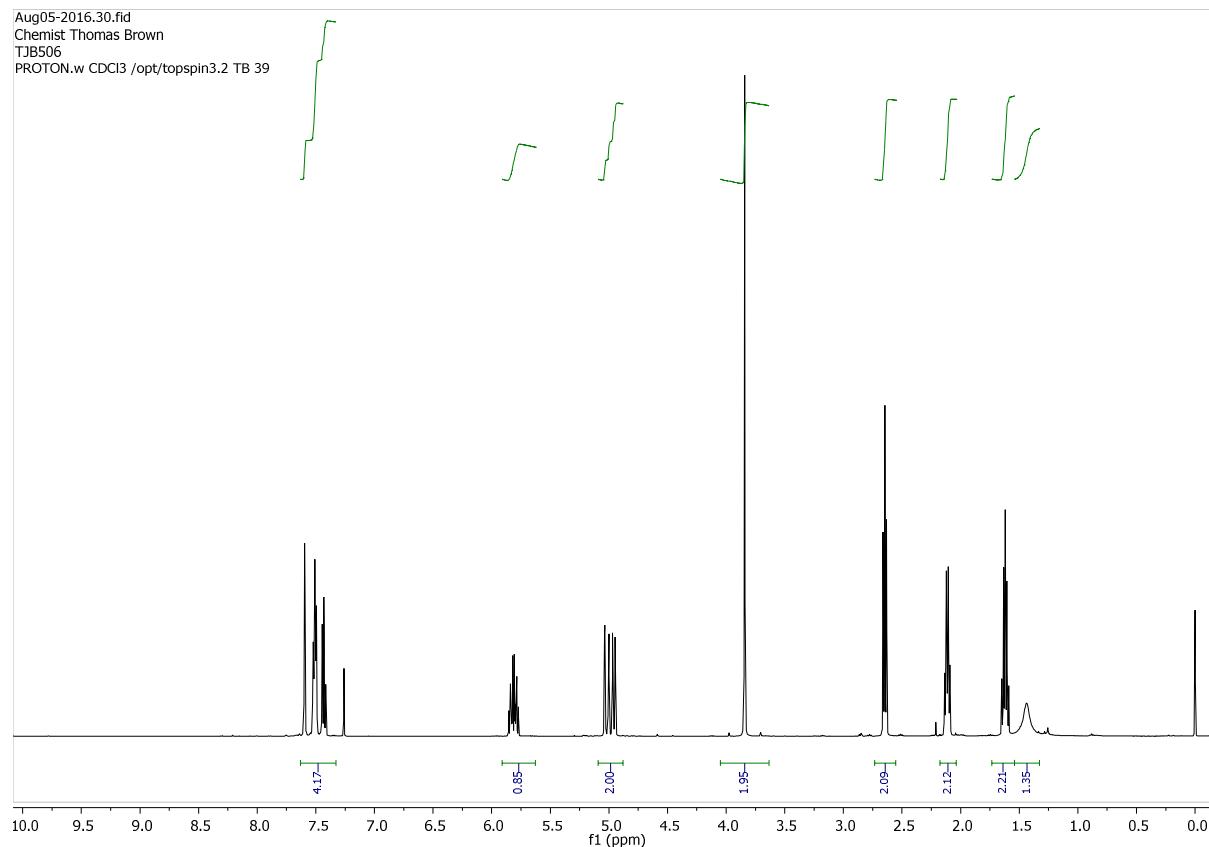


*N*-(3-(trifluoromethyl)benzyl)pent-4-en-1-amine **55**.



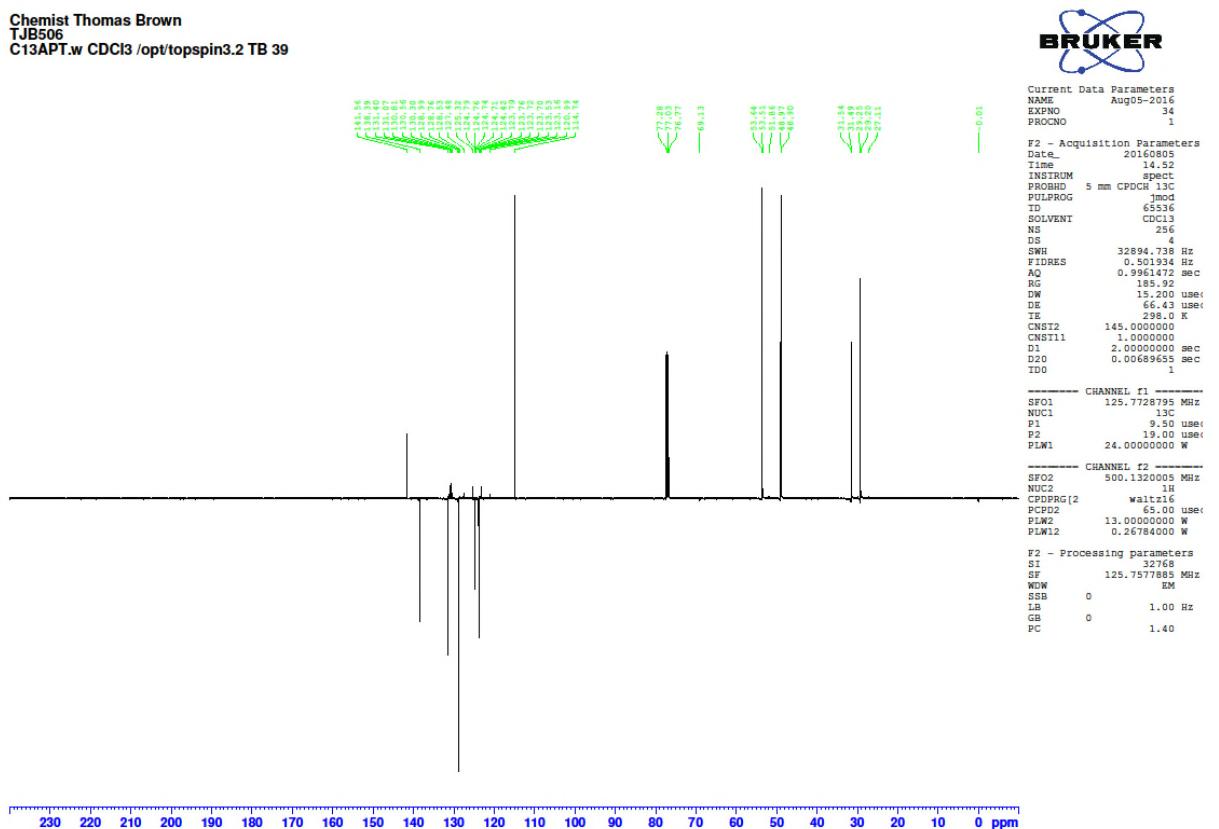
TJB506.

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ).

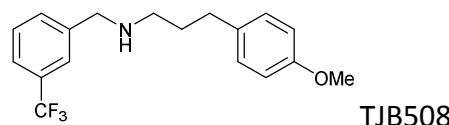


<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>).

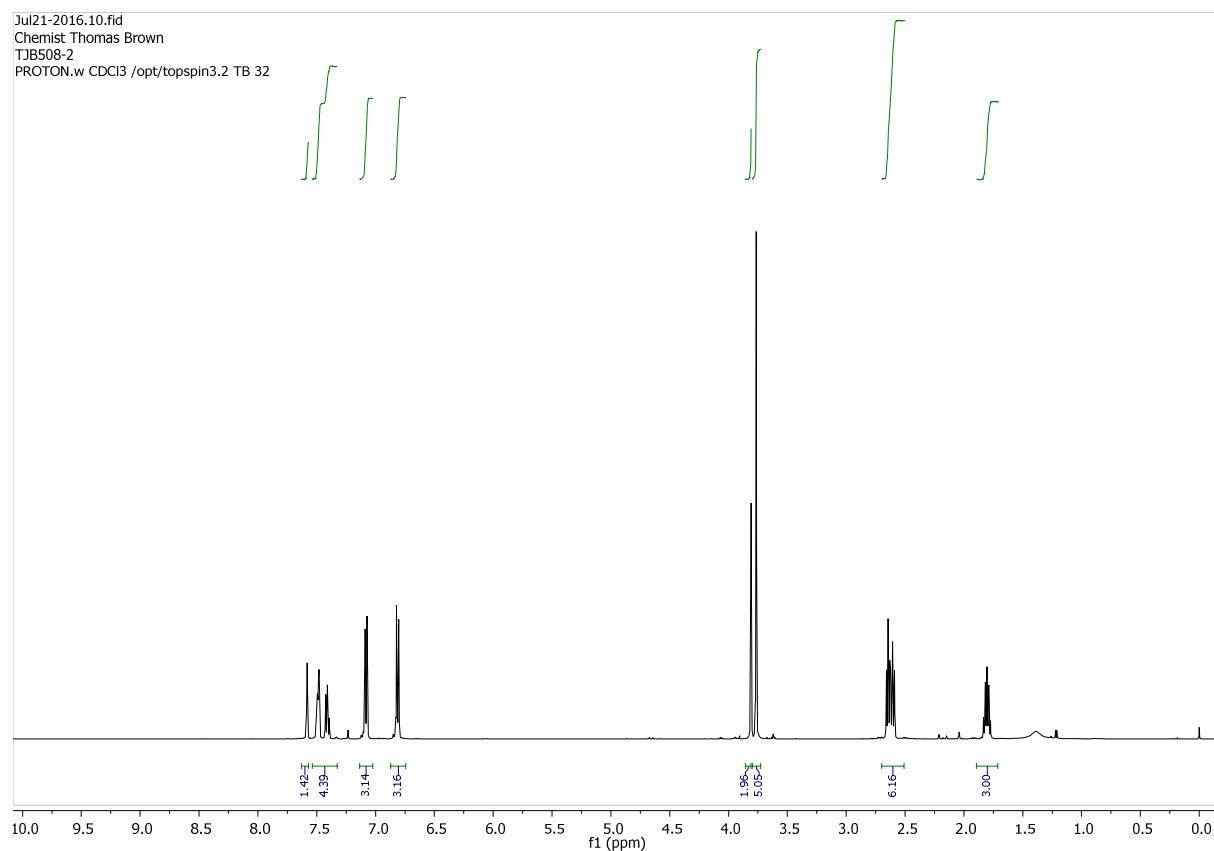
Chemist Thomas Brown  
TJB506  
C13APT.w CDCl<sub>3</sub> /opt/topspin3.2 TB 39



3-(4-Methoxyphenyl)-N-(3-(trifluoromethyl)benzyl)propan-1-amine **56**.



$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ).



<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>).

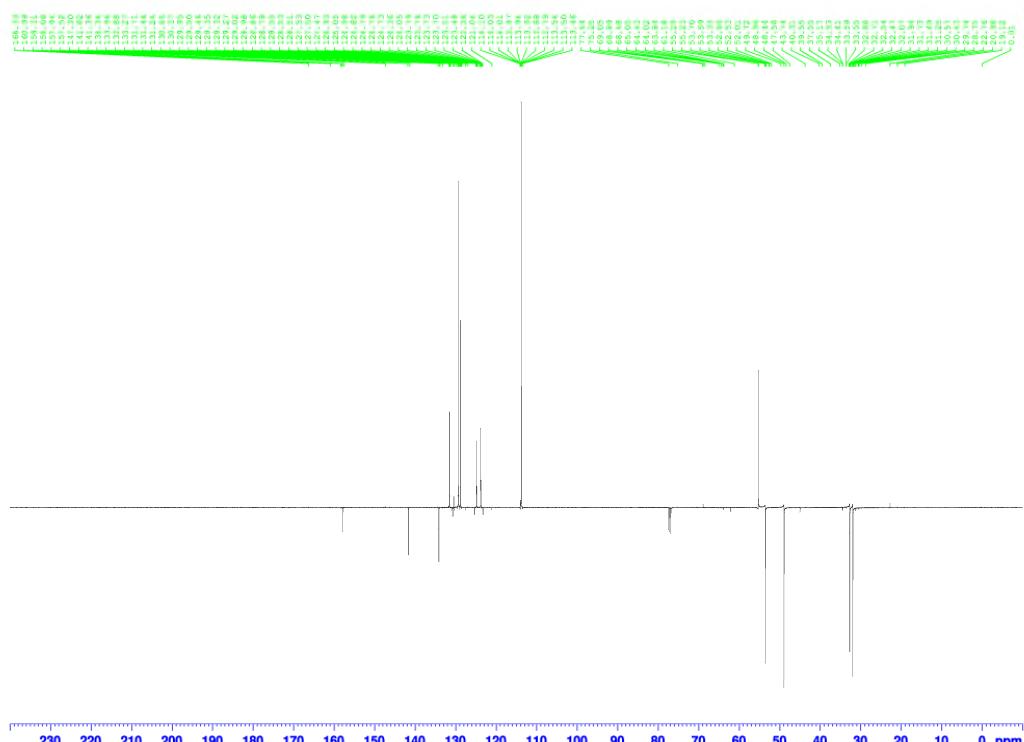
Chemist Thomas Brown

TJB508-2

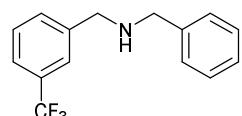
C13APT.w CDCl<sub>3</sub> /opt/topspin3.2 TB 32



Current Data Parameters  
 NAME Jc121-2010  
 EXPNO 14  
 PROBNO 1  
 P2 - Acquisition Parameters  
 Data 20160721  
 Time 12.46  
 INSTRUM spect  
 PROBHD 5 mm CPDCH 13C  
 PULPROG 90deg  
 TD 65536  
 SOLVENT CDCl<sub>3</sub>  
 NS 256  
 DS 1  
 SWH 32894.738 Hz  
 FIDRES 0.501934 Hz  
 AQ 0.9961472 sec  
 RG 185.92  
 DW 15.00 used  
 DE 66.43 used  
 TE 298.0 K  
 CNTST2 145.000000  
 CNSTT11 1.0000000  
 D1 2.0000000 sec  
 D20 0.00689655 sec  
 T00 1  
 ===== CHANNEL f1 ======  
 SF01 125.7728795 MHz  
 NUC1 13C  
 P1 9.50 used  
 P2 15.00 used  
 PLW1 24.0000000 W  
 ===== CHANNEL f2 ======  
 SF02 500.1320005 MHz  
 NUC2 1H  
 CPDPRG12 waltz16  
 PCPD2 65.00 used  
 PLW2 13.0000000 W  
 PLW2 0.26784000 W  
 F2 - Processing parameters  
 SI 32768  
 SF 125.7577885 MHz  
 WDW  
 SSB 0 2M  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

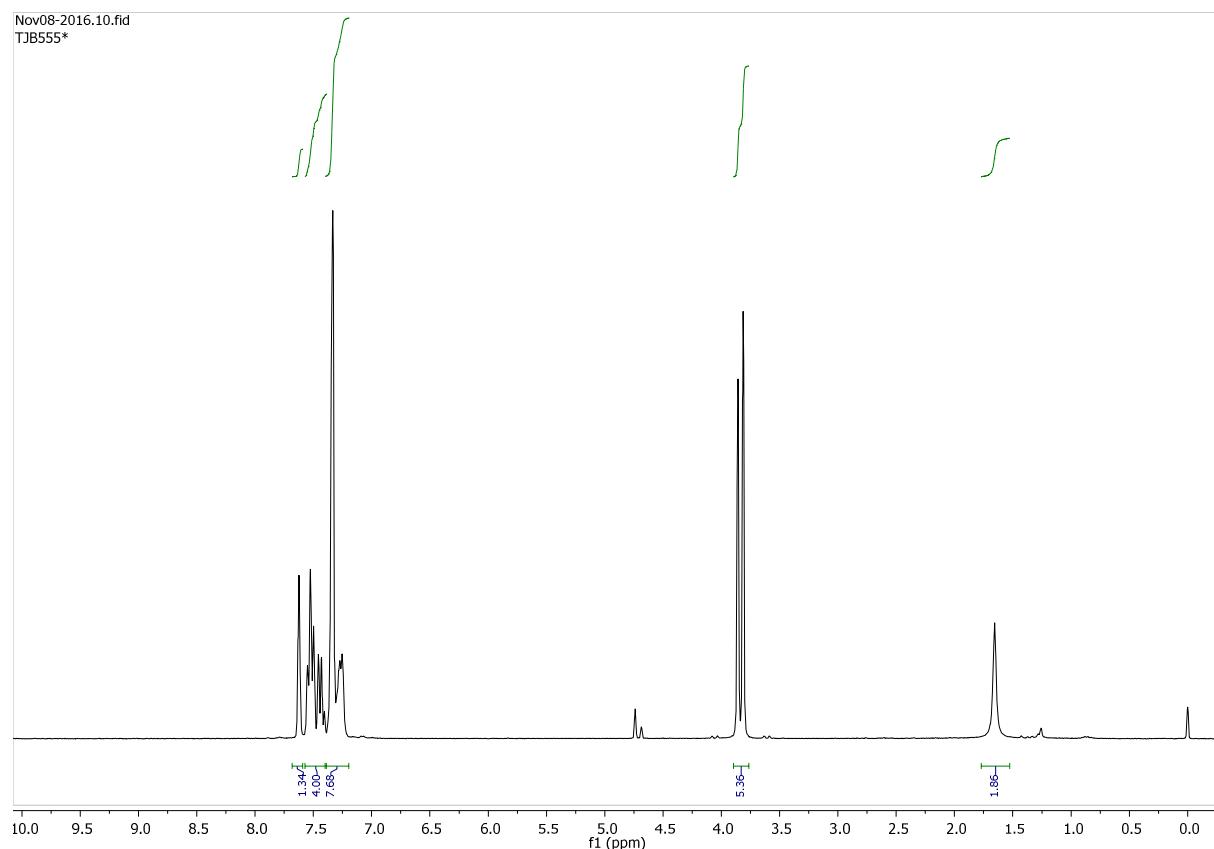


*N*-Benzyl-1-(3-(trifluoromethyl)phenyl)methanamine **57**.



TJB555.

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ).

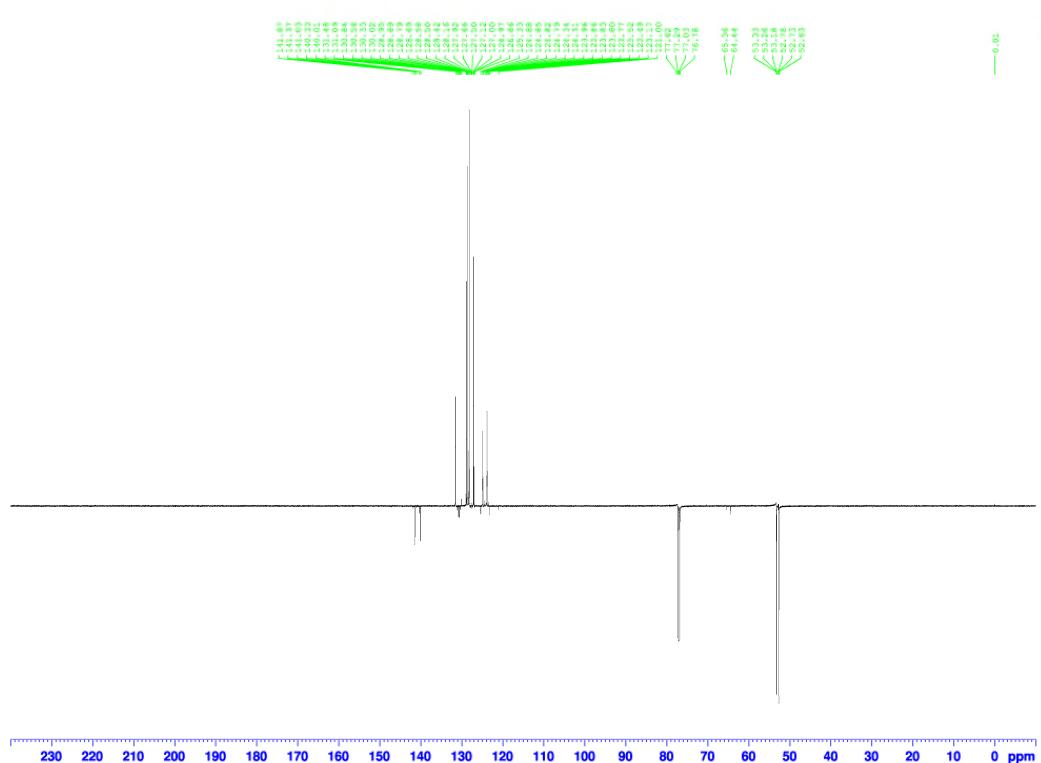


<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>).

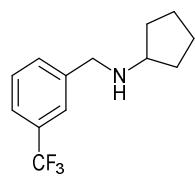
Chemist Thomas Brown  
TJB555\*  
C13APT.w CDCl<sub>3</sub> /opt/topspin3.2 TB 10



Current Data Parameters  
 NMRA Nov08-2016  
 EXPNO 14  
 PROCHD 1  
  
 F2 - Acquisition Parameters  
 DPPG 2016132  
 Time 2.00  
 INSTRUM spect  
 PROBHD 5 mm CPDPCH 13C  
 PULPROG jmd4  
 TD 65536  
 SOLVENT CDCl<sub>3</sub>  
 NS 256  
 DS 4  
 SWB 32894.7 Hz  
 FIDRES 0.501334 Hz  
 AQ 0.9961472 sec  
 RG 185.92  
 DW 15.40 usec  
 DE 66.45 usec  
 TE 298.0 K  
 CNST2 145.000000  
 CNST11 1.000000  
 D1 2.0000000 sec  
 D2 0.0063965 sec  
 TDO  
  
 ===== CHANNEL f1 =====  
 SFO1 125.7728795 MHz  
 NUC1 13C  
 P1 9.50 usec  
 P2 19.00 usec  
 PLW1 24.0000000 W  
  
 ===== CHANNEL f2 =====  
 SFO2 500.1320005 MHz  
 NUC2 1H  
 CCPDPRG2 waltz16  
 PCPD2 65.00 usec  
 PLW2 13.0000000 W  
 PLW12 0.26784000 W  
  
 F2 - Processing parameters  
 SI 32768  
 SF 125.7577885 MHz  
 WDW EM  
 SSBB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

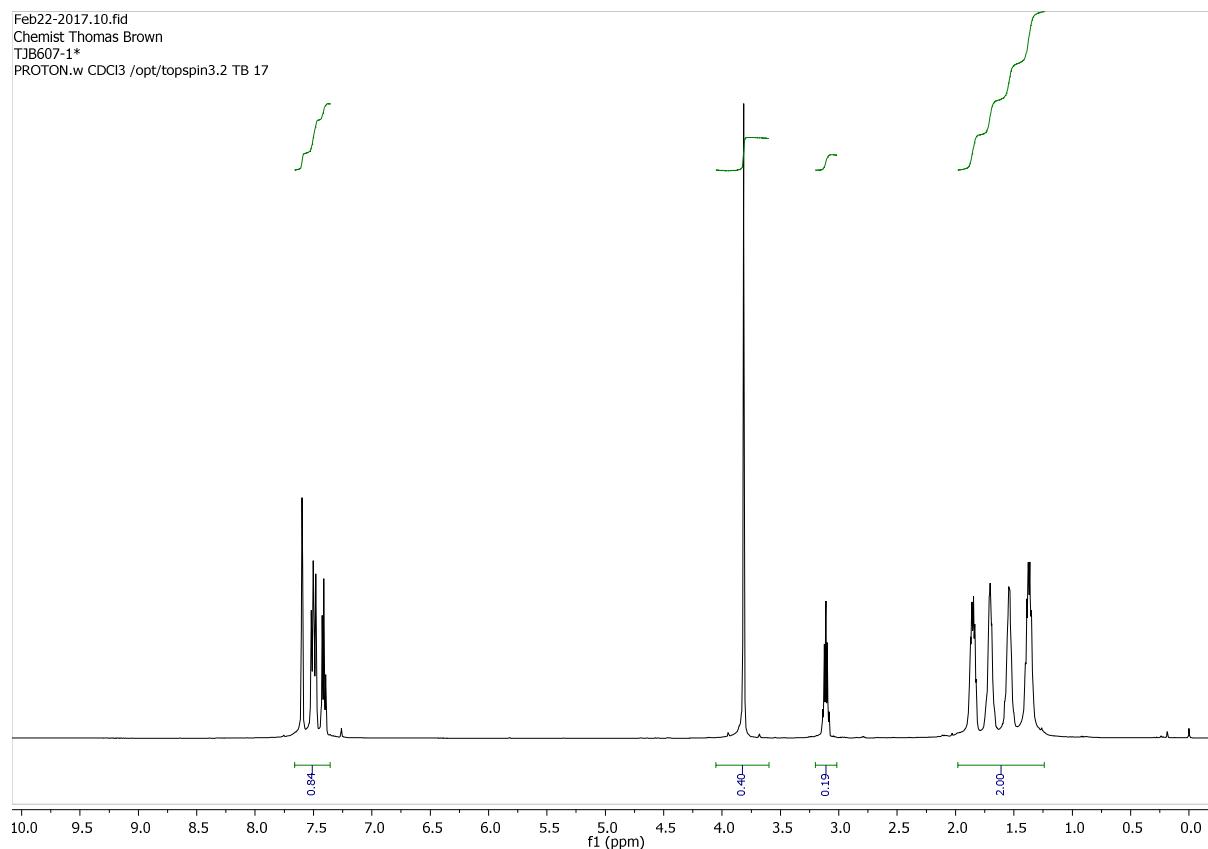


*N*-(3-(trifluoromethyl)benzyl)cyclopentanamine **58**.



TJB607.

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ).



<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>).

Chemist Thomas Brown

TJB607-1\*

C13APT.w CDCl<sub>3</sub> /opt/topspin3.2 TB 17



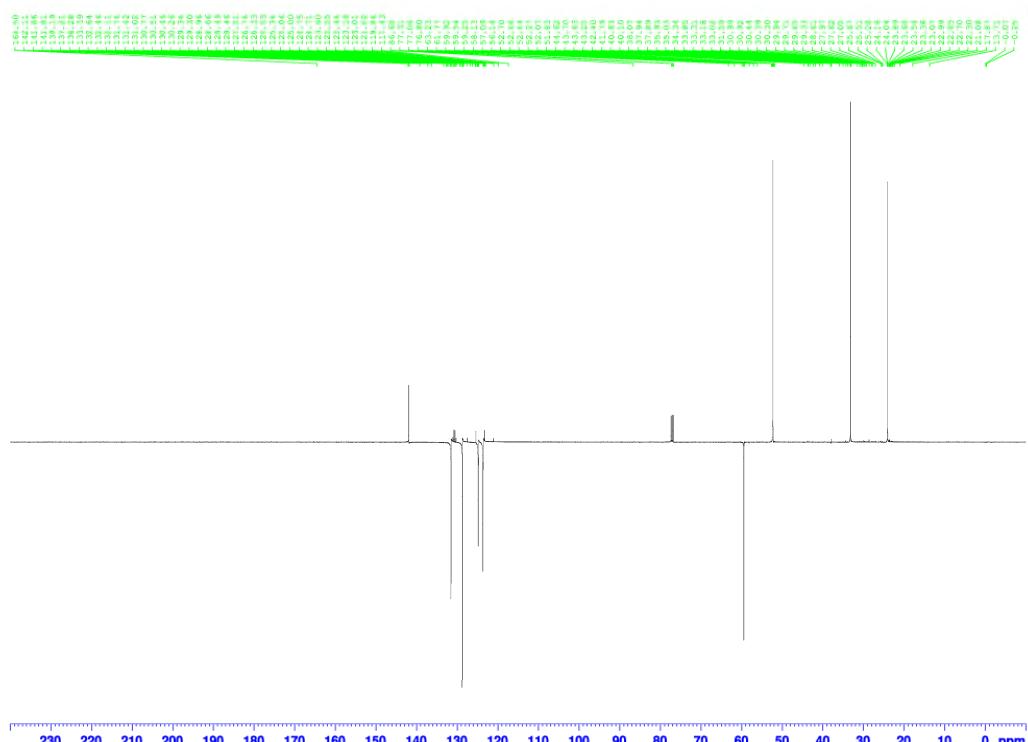
Current Data Parameters  
NAME Feb22-2017  
EXNO 14  
PRCNO 1

P2 - Acquisition Parameters  
Date\_ 20170222  
Time\_ 13.53  
INSTRUM spect  
PROBHD 5 mm CPDCT-13C  
EPPROG僵  
TD 65536  
SOLVENT CDCl<sub>3</sub>  
NS 256  
DS 4  
SWH 32894.738 Hz  
FIDRES 0.501934 Hz  
AQ 0.9961472 sec  
RG 185.95  
DW 15.00 usec  
DE 66.43 usec  
TE 298.0 K  
CNST2 145.000000  
CNSST11 1.000000  
D1 2.0000000 sec  
D20 0.00689655 sec  
TD0 1

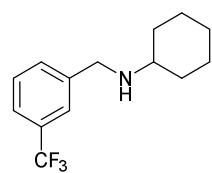
===== CHANNEL f1 =====  
SF01 125.7728795 MHz  
NUC1 <sup>13</sup>C  
P1 9.50 usec  
P2 19.00 usec  
PLW1 24.0000000 W

===== CHANNEL f2 =====  
SF02 500.1320005 MHz  
NUC2 <sup>1</sup>H  
CPDPRG[2] waltz16  
PCPD2 65.00 usec  
PLW2 13.0000000 W  
PLW2 0.26784000 W

P2 - Processing parameters  
SI 32768  
SF 125.7577885 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

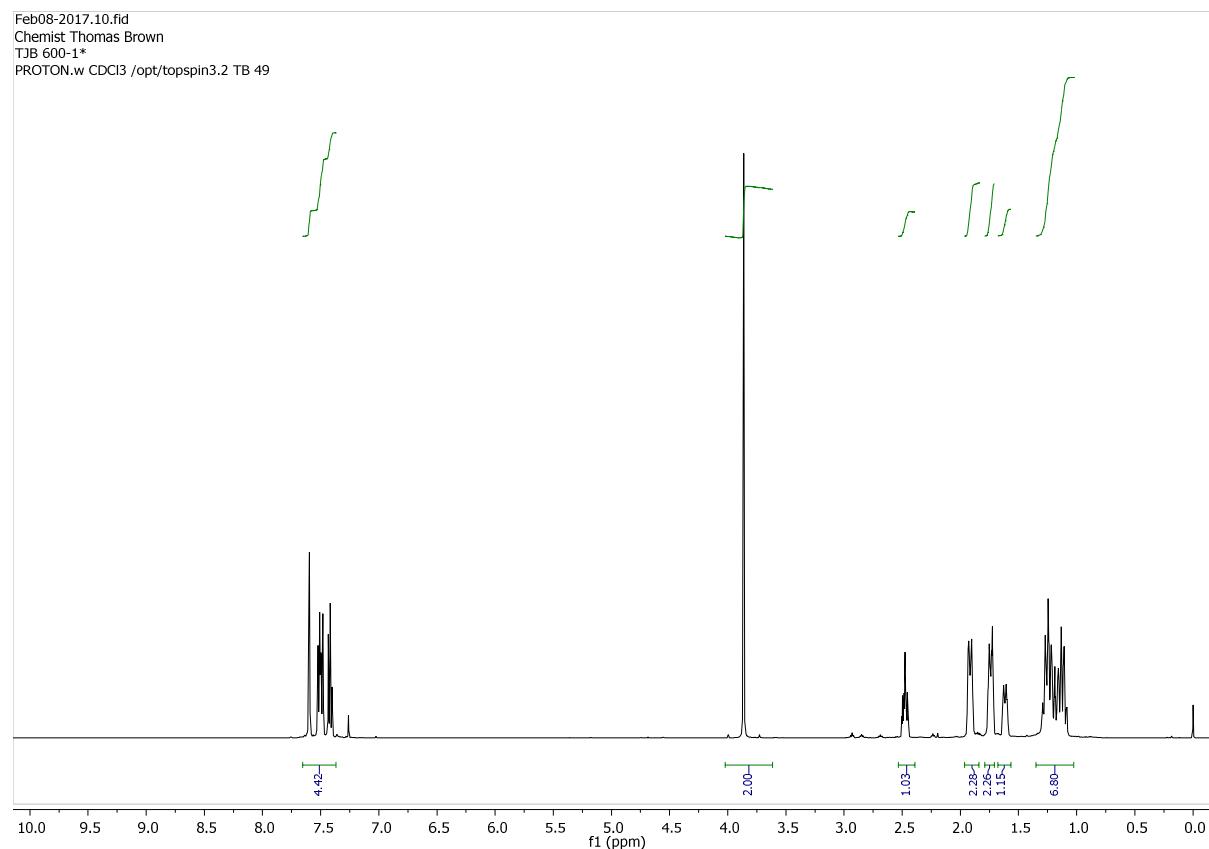


*N*-(3-(Trifluoromethyl)benzyl)cyclohexanamine **59**.



TJB600.

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ).



<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>).

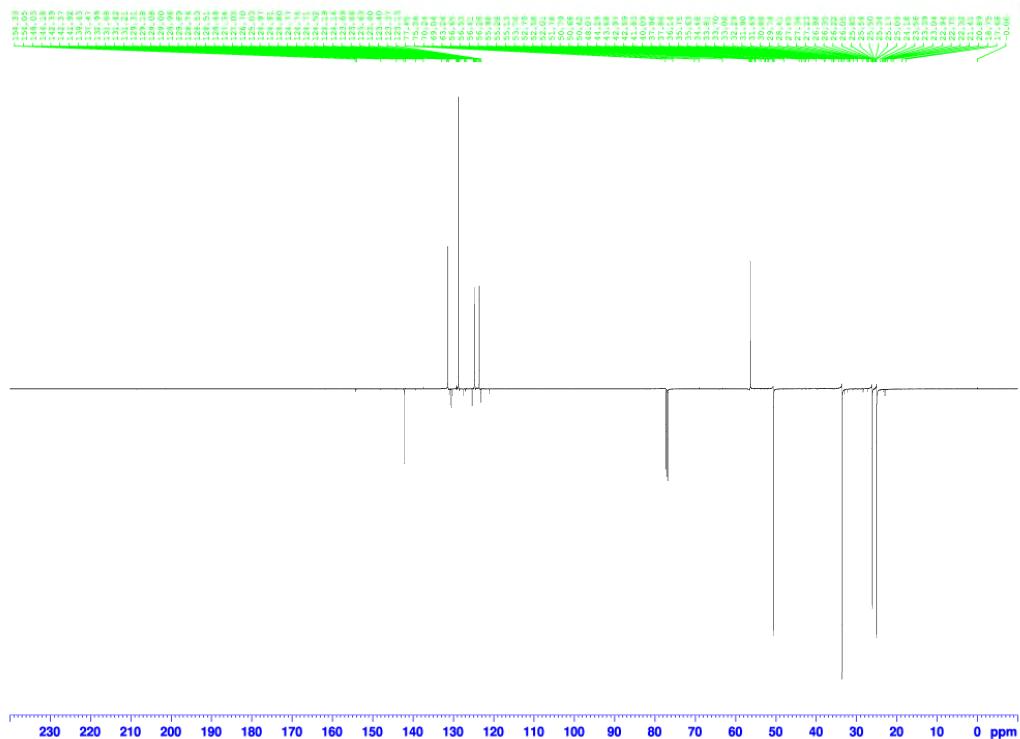
Chemist Thomas Brown

TJB 600-1\*

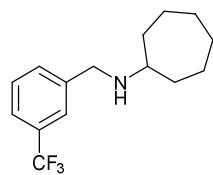
C13APT.w CDCl<sub>3</sub> /opt/topspin3.2 TB 49



Current Data Parameters  
 NAME Feb06-2017  
 EXPNO 14  
 PROCNO 1  
  
 F2 - Acquisition Parameters  
 Data 20170206  
 Time 10.56  
 INSTRUM spect  
 PROBHD 5 mm CPDCH 13C  
 PULPROG zg3d  
 TD 65536  
 SOLVENT CDCl<sub>3</sub>  
 NS 256  
 D1 4  
 SWH 32894.738 Hz  
 FIDRES 0.501934 Hz  
 AQ 0.9961472 sec  
 RG 128  
 DW 13.200 usec  
 DS 66.43 usec  
 TS 298.0 K  
 CNST1 145.00000000  
 CNST11 1.0000000  
 D1 6.00000000 sec  
 D20 0.00689655 sec  
 TDD 1  
  
 ===== CHANNEL f1 =====  
 SFO1 125.7728795 MHz  
 NUC1 <sup>13</sup>C  
 P1 5 usec  
 P2 19.00 usec  
 PLW1 24.00000000 W  
  
 ===== CHANNEL f2 =====  
 SFO2 500.1320005 MHz  
 NUC2 <sup>1</sup>H  
 CPDPRG[2 waltz16  
 CPDPG2 65.00 usec  
 PLR2 13.00000000 W  
 PLW2 0.26784000 W  
  
 F2 - Processing parameters  
 SI 32768  
 SF 125.7577853 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

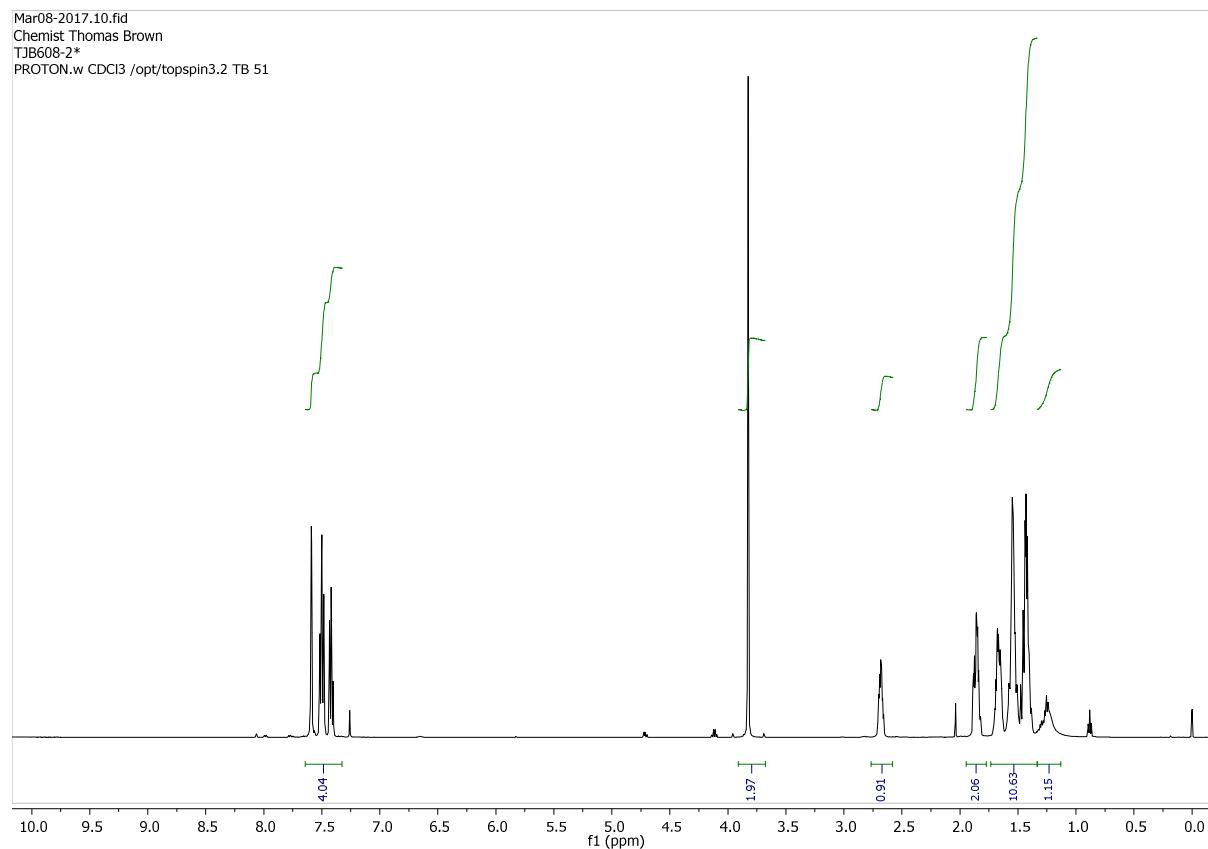


*N*-(3-(Trifluoromethyl)benzyl)cycloheptanamine **60**.



TJB608.

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ).

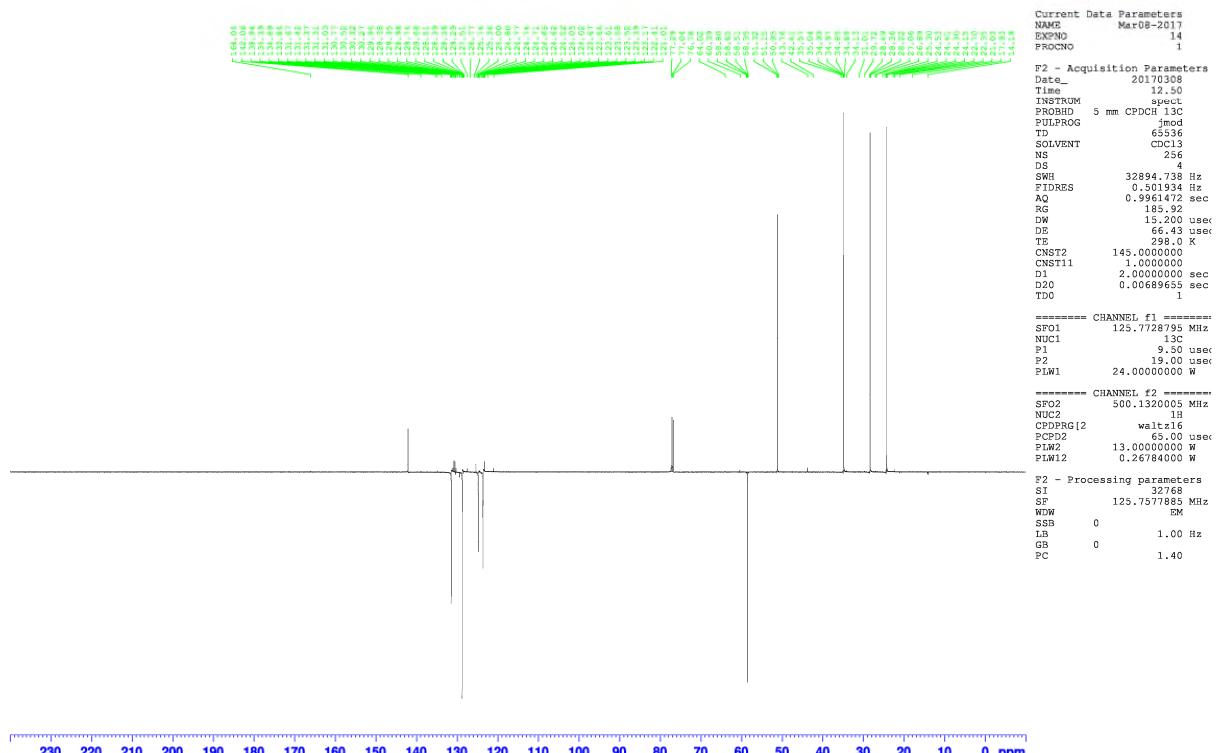


<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>).

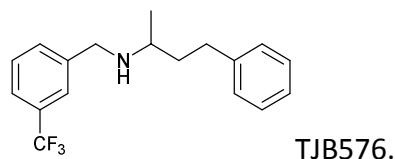
Chemist Thomas Brown

TJB608-2\*

C13APT.w CDCl<sub>3</sub> /opt/topspin3.2 TB 51

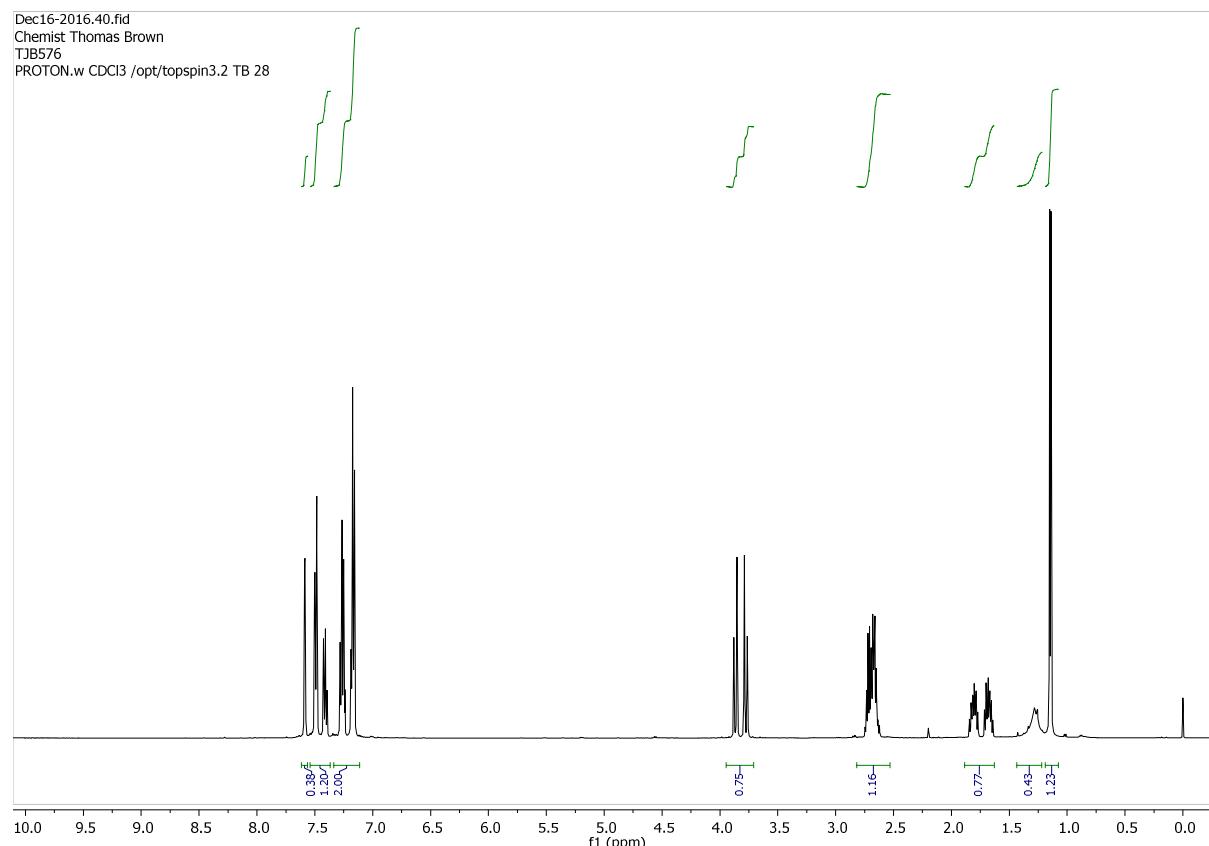


4-Phenyl-N-(3-(trifluoromethyl)benzyl)butan-2-amine **61**.



TJB576.

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ).



<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>).

Chemist Thomas Brown  
TJB576  
C13APT.w CDCl<sub>3</sub> /opt/topspin3.2 TB 28

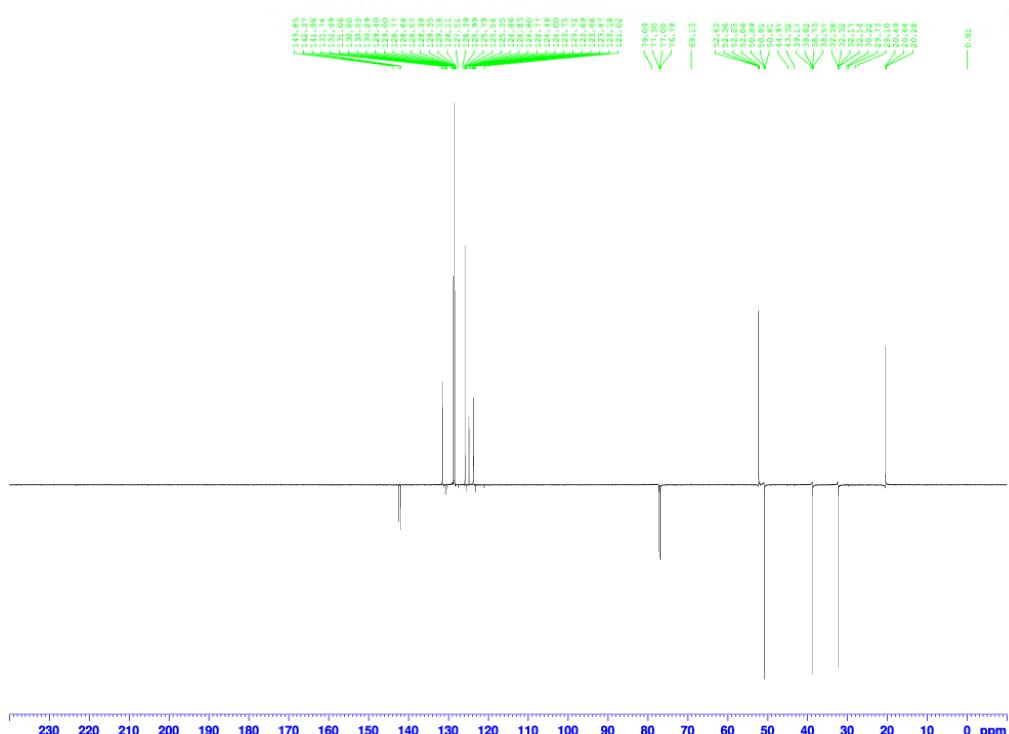


Current Data Parameters  
NAME Dec16-2016  
EXPNO 44  
PROCNO 1  
  
F2 - Acquisition Parameters  
Date 20161216  
Time 15.55  
INSTRUM spect  
PROBHD 5 mm CPDCH BBO  
PULPROG jmod  
TD 65536  
SOLVENT CDCl<sub>3</sub>  
NS 256  
DS 4  
SWH 32894.738 Hz  
FIDRES 0.501934 Hz  
AQ 0.9800 sec  
RG 185.92  
DW 15.200 usec  
DE 66.43 usec  
TE 220.0 K  
CNS1 145.000000  
CNS11 1.000000  
D1 2.000000 sec  
D20 0.00689655 sec  
TD0 1

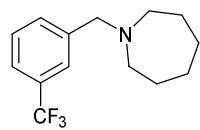
----- CHANNEL f1 -----  
SF01 125.7720000 MHz  
NUC1 <sup>13</sup>C  
P1 9.50 usec  
P2 19.00 usec  
PLW1 24.0000000 W

----- CHANNEL f2 -----  
SF02 500.1320005 MHz  
NUC2 <sup>1</sup>H  
CPDPR2c waltz16  
PCPD2 65.00 usec  
PLW2 13.0000000 W  
PLW12 0.26784000 W

F3 - Processing parameters  
SI 32768  
SF 125.7577885 MHz  
SW 1.0000000 sec  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

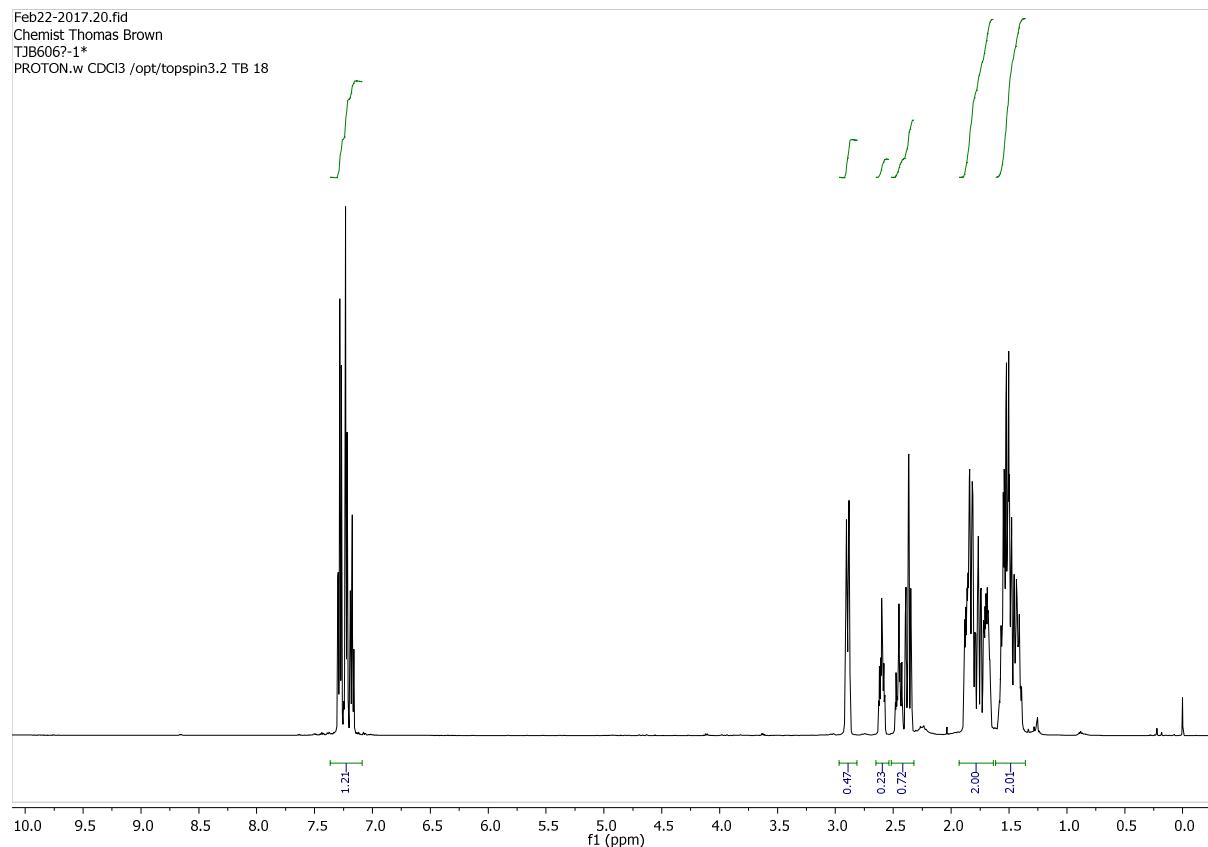


**1-(3-(Trifluoromethyl)benzyl)azepane **62**.**



TJB585.

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ).



<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>).

Chemist Thomas Brown  
TJB585-1  
C13APT.w CDCl<sub>3</sub> /opt/topspin3.2 TB 54



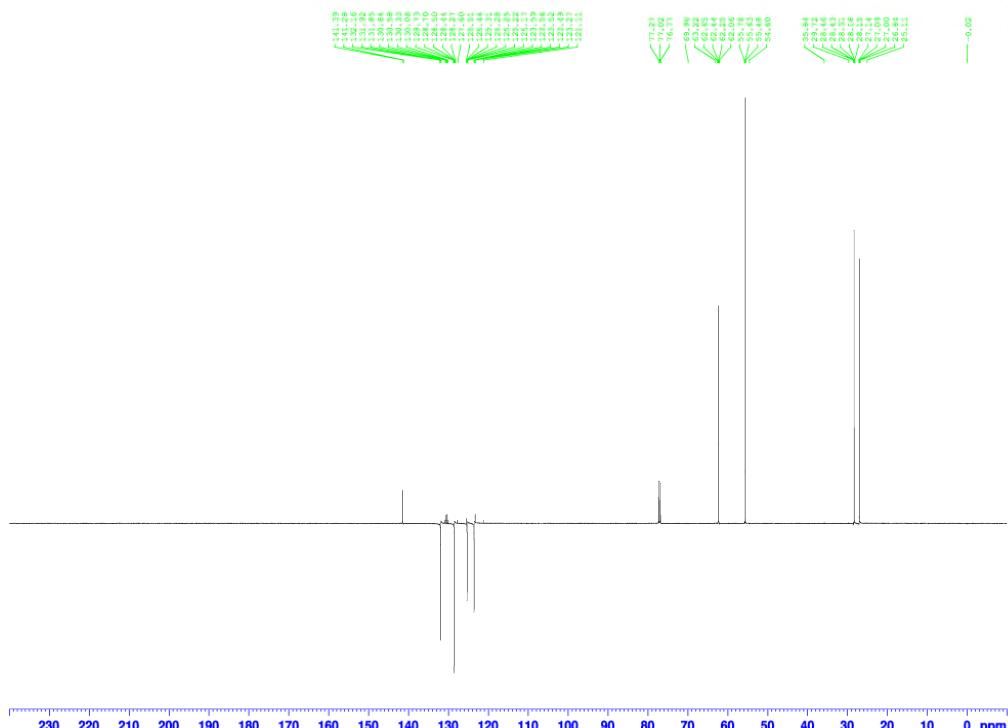
```
Current Data Parameters
NAME      Jan17-2017
EXPNO     14
PROCNO    1

F2 - Acquisition Parameters
Date       20170117
Time       17.34
INSTRUM   spect
PROBHD   5 mm CPDCH3OC
PULPROG  jmod
TD        65536
SOLVENT   CDCl3
NS         256
DS          4
SWH       32894.738 Hz
FIDRES   0.501934 Hz
AQ        0.9838 sec
RG        185.92
DW        15.200 usec
DE        66.43 usec
TE        220.0 K
CNST1    145.000000
CNST11   1.000000
D1        2.0000000 sec
D2D      0.00689655 sec
TD0           1

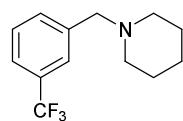
----- CHANNEL f1 -----
SF01      125.722000 MHz
NUC1      13C
P1        9.50 usec
P2        19.00 usec
PLW1     24.0000000 W

----- CHANNEL f2 -----
SF02      500.1320005 MHz
NUC2      1H
CPDPRG[2]  waltz16
PCPD2    65.00 usec
PLW2     13.0000000 W
PLW12    0.26784000 W

F2 - Processing parameters
SI        32768
SF        125.7577885 MHz
WDW      0
SSB      0
LB        1.00 Hz
GB      0
PC        1.40
```

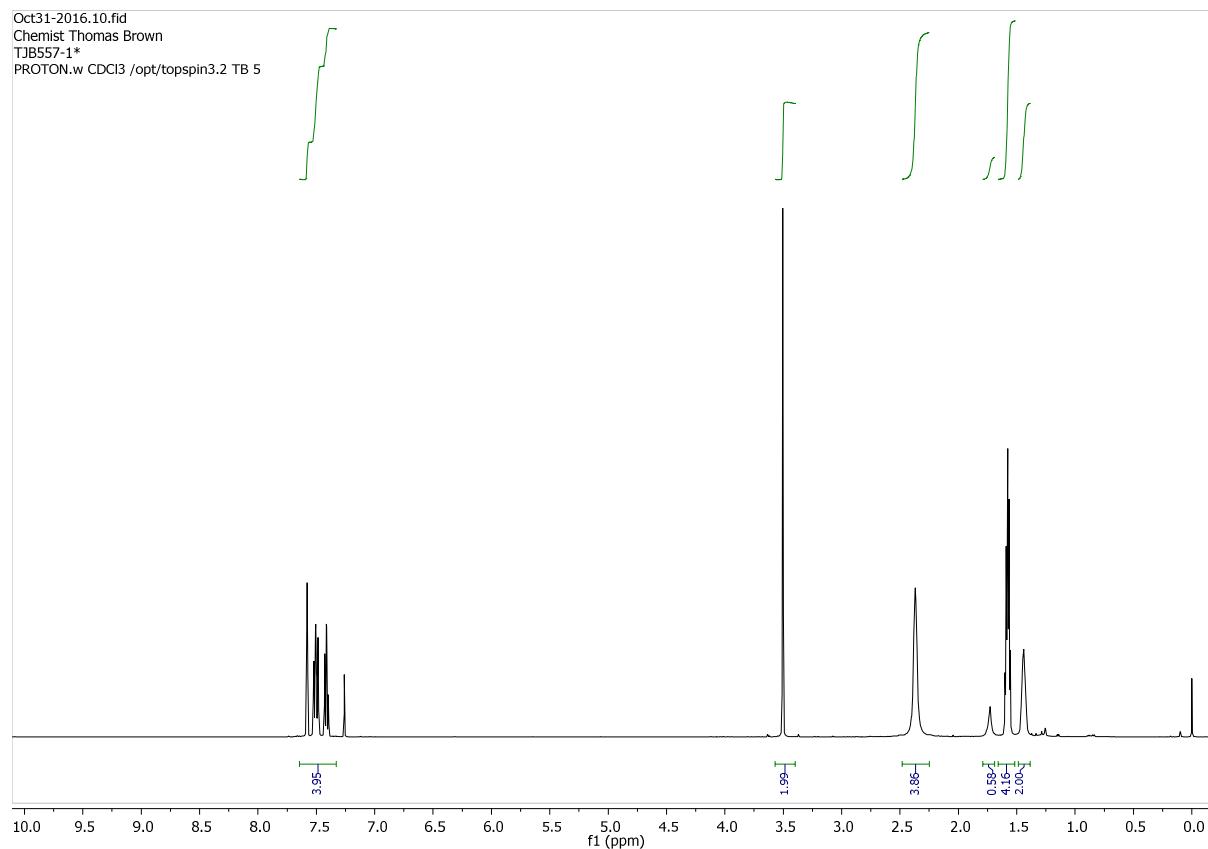


1-(3-(Trifluoromethyl)benzyl)piperidine **63**.



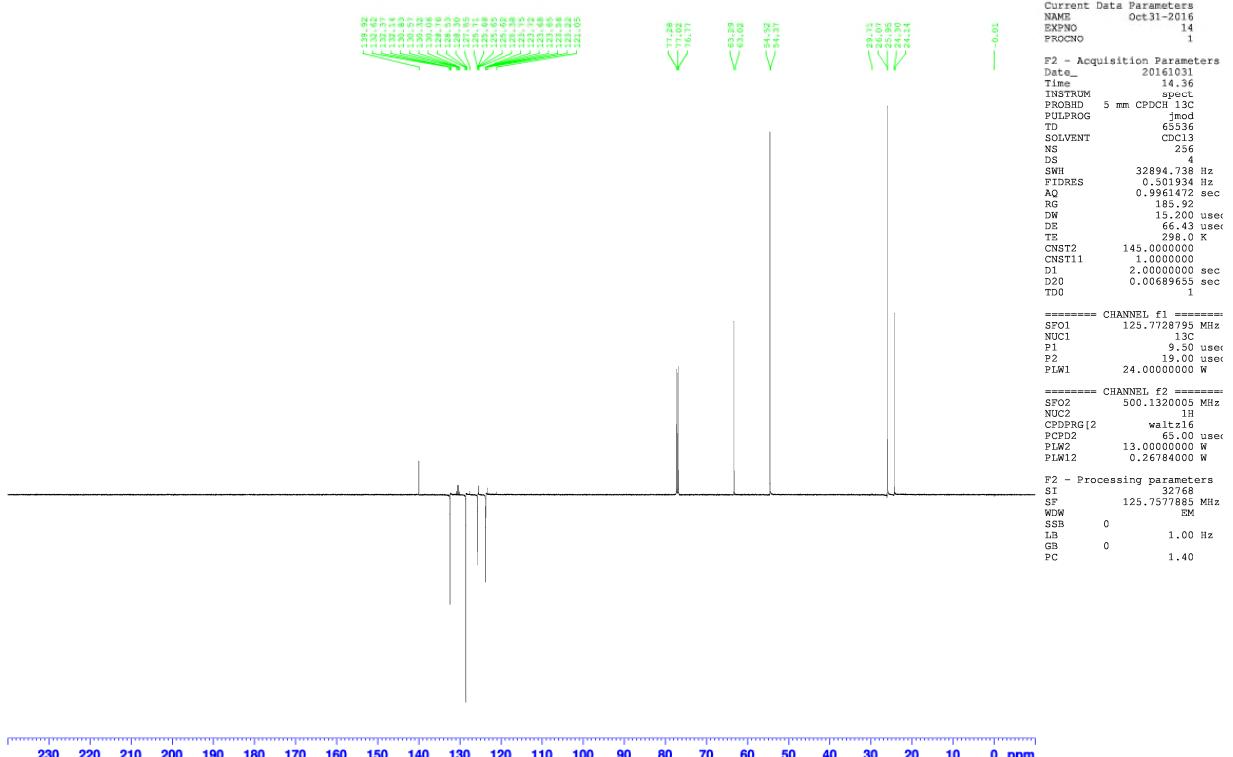
TJB557.

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>).

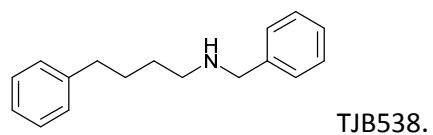


<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>).

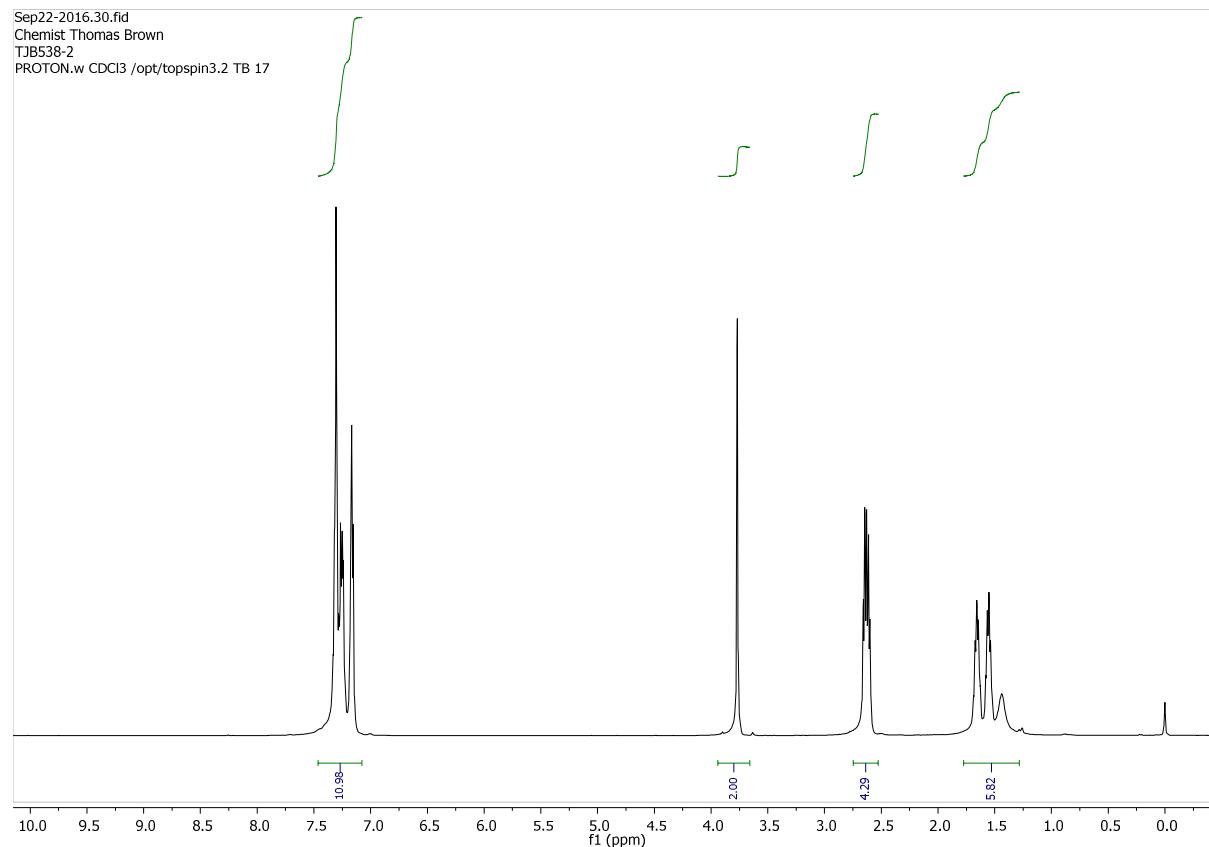
Chemist Thomas Brown  
TJB557-1\*  
C13APT.w CDCl3 /opt/topsim3.2 TB 5



*N*-Benzyl-4-phenylbutan-1-amine **64**.

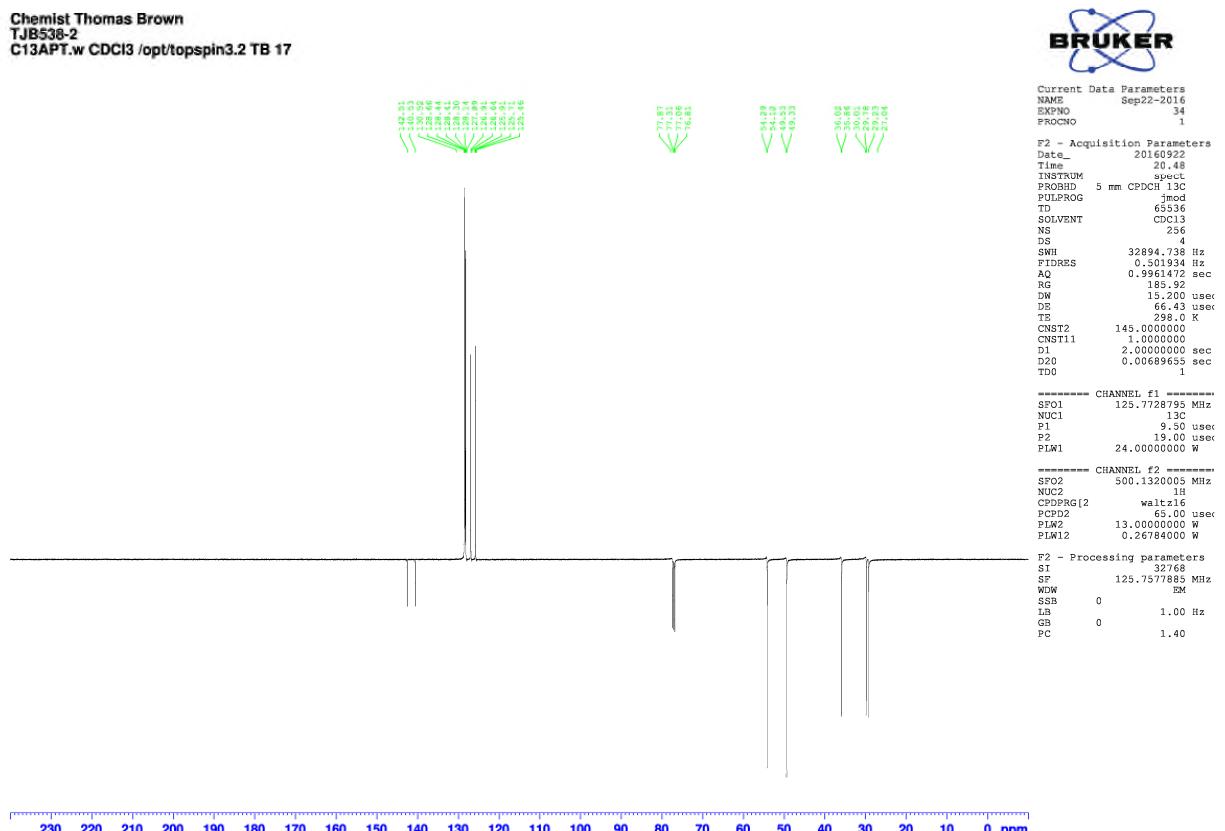


$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ).

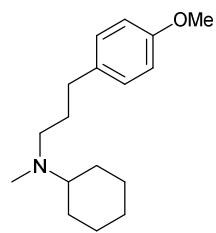


<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>).

Chemist Thomas Brown  
TJB538-2  
C13APT.w CDCl3 /opt/topspin3.2 TB 17

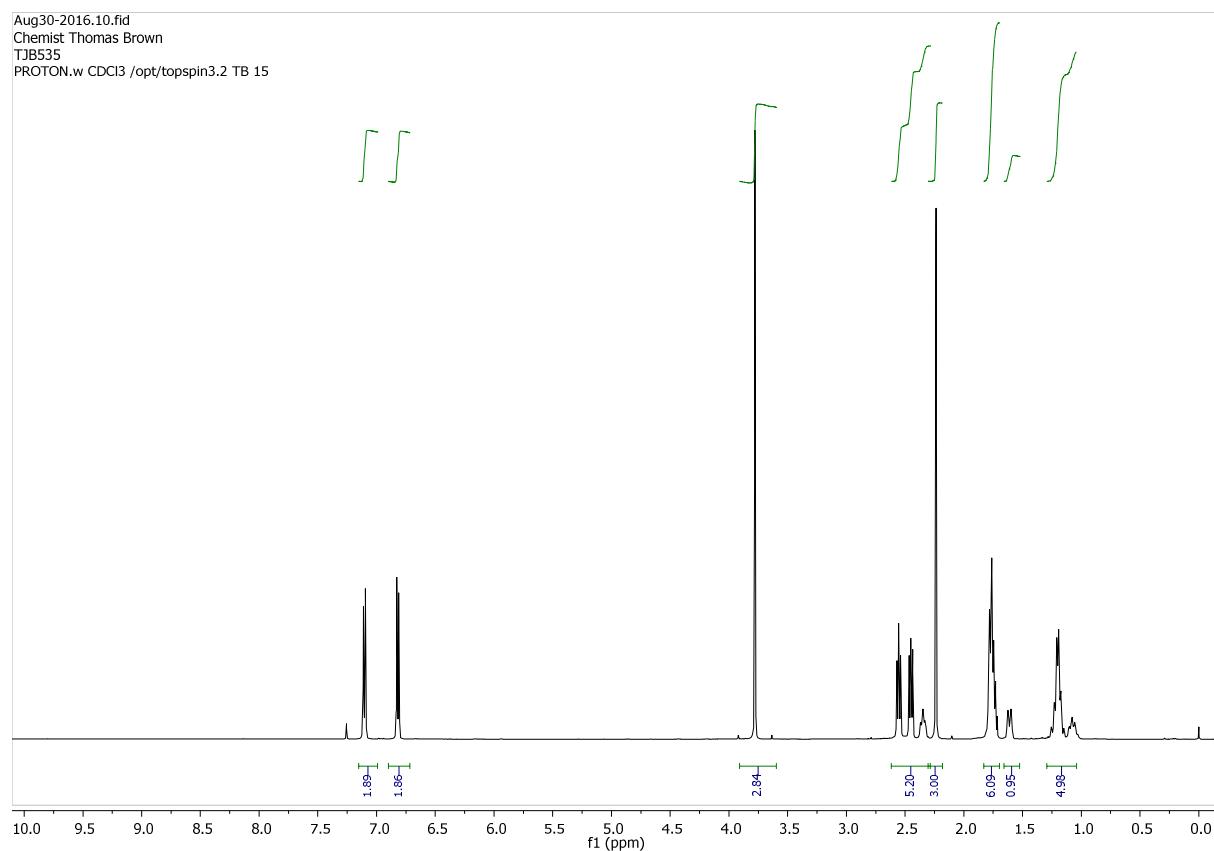


*N*-(3-(4-Methoxyphenyl)propyl-*N*-methylcyclohexanamine **65**.



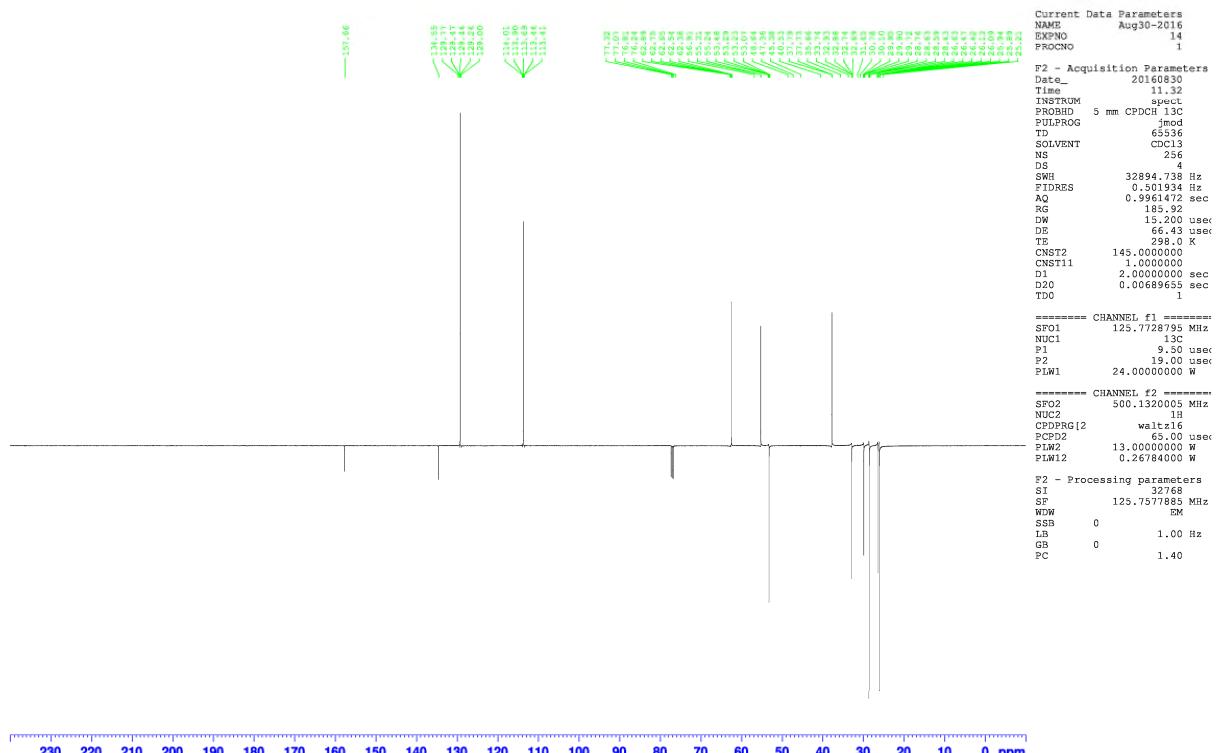
TJB535.

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ).

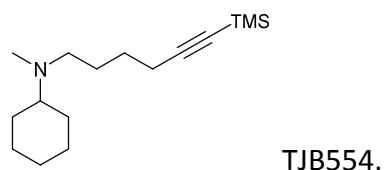


<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>).

Chemist Thomas Brown  
TJB535  
C13APT.w CDCl<sub>3</sub> /opt/topspin3.2 TB 15

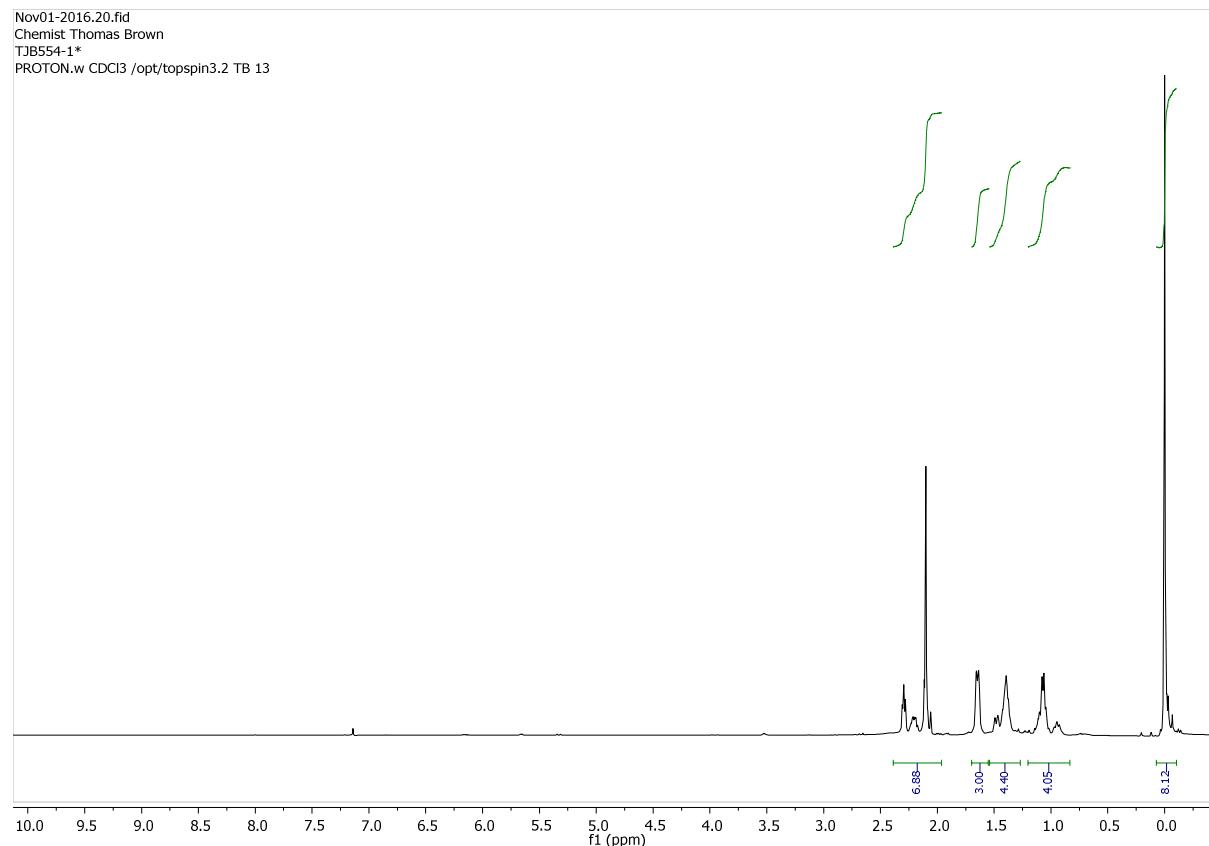


*N*-Methyl-*N*-(6-trimethylsilyl)hex-5-yn-1-yl)cyclohexanamine **66**.



TJB554.

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>).



<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>).

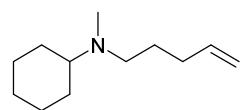
C13APT.w CDCl<sub>3</sub> /opt/topspin3.2 TB 13



Current Data Parameters  
 NAME Nov01-2016  
 EXPNO 24  
 PROCNO 1  
  
 F2 - Acquisition Parameters  
 Data 20161101  
 Time 17.03  
 INSTRUM spect  
 PROBHD 5 mm CPDMR 13C  
 PULPROG 6000  
 TD 65536  
 SOLVENT CDCl<sub>3</sub>  
 NS 256  
 DS 4  
 SWH 32894.738 Hz  
 FIDRES 0.501934 Hz  
 AQ 0.9961472 sec  
 RG 185.32  
 DW 15.00 usec  
 DE 66.43 usec  
 T2 298.0 K  
 CNTST2 145.0000000  
 CNSTT11 1.0000000  
 D1 2.00000000 sec  
 D20 0.00689655 sec  
 TDO 1  
  
 ----- CHANNEL F1 -----  
 SFO1 125.7728795 MHz  
 NUC1 13C  
 P1 9.50 usec  
 P2 19.00 usec  
 PLW1 24.0000000 W  
  
 ----- CHANNEL F2 -----  
 SFO2 500.1320009 MHz  
 NUC2 1H  
 CPDPRG[2] Waltz16  
 PCPD2 65.00 usec  
 PLW2 13.00000000 W  
 PLW2 0.26784000 W  
  
 F2 - Processing parameters  
 ST 32768  
 SF 125.7578094 MHz  
 WM 0  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

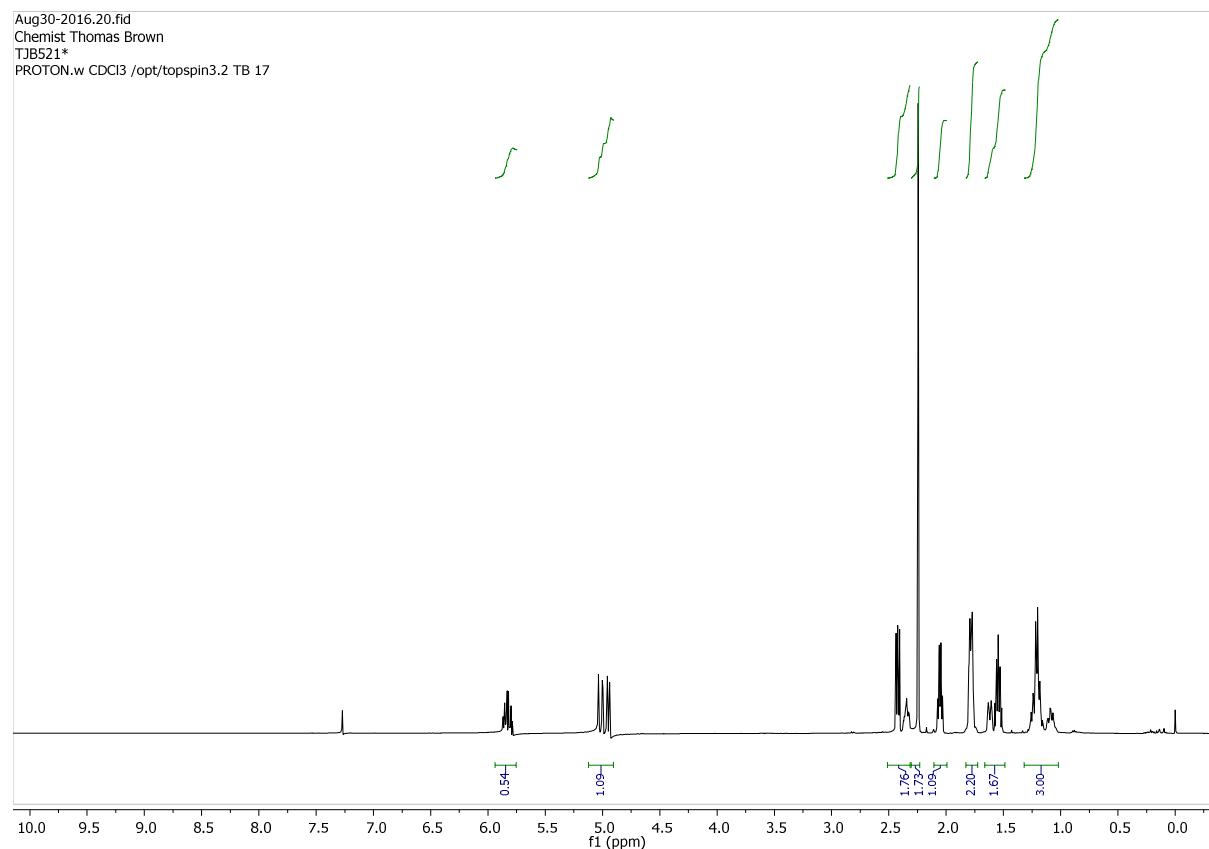


*N*-Methyl-*N*-(pent-4-en-1-yl)cyclohexanamine **67**.

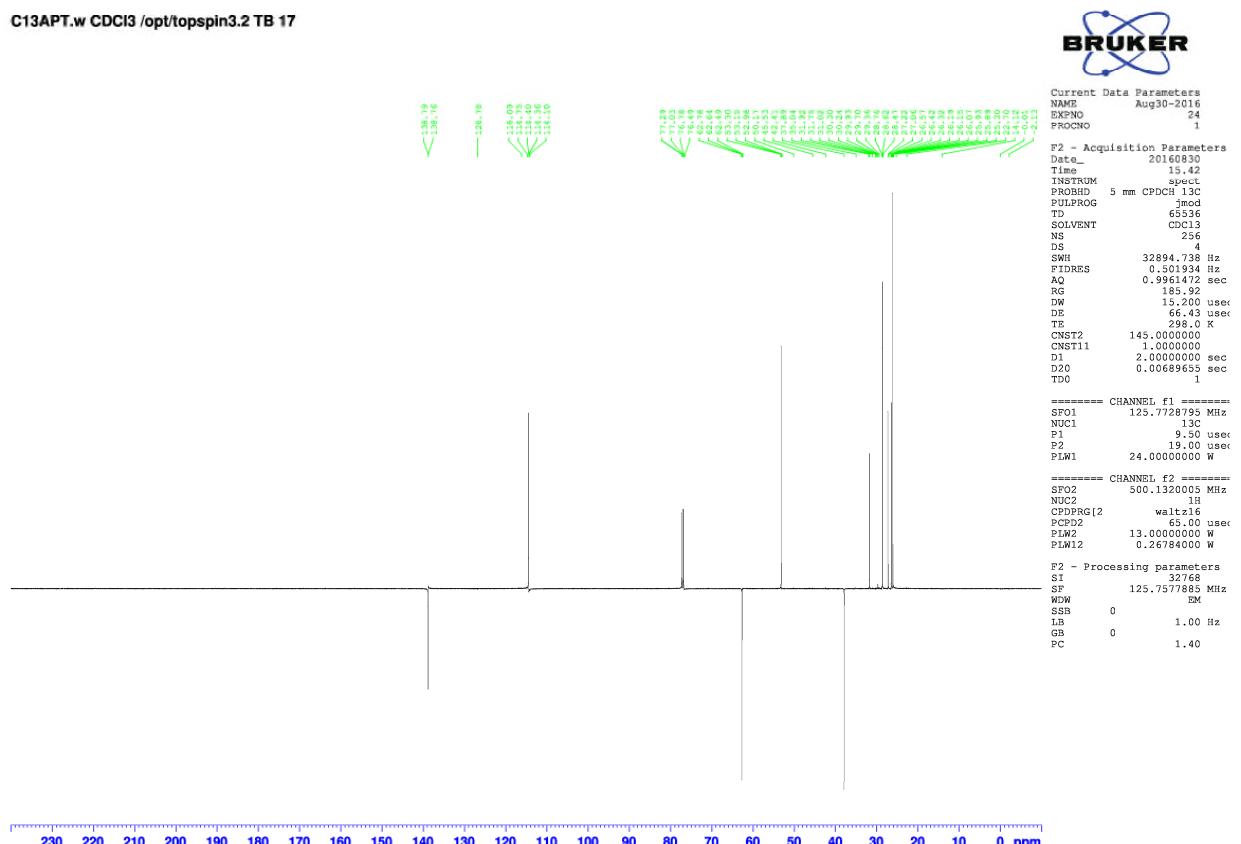


TJB521.

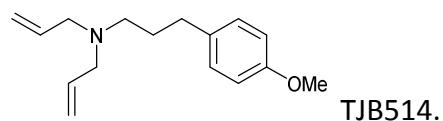
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ).



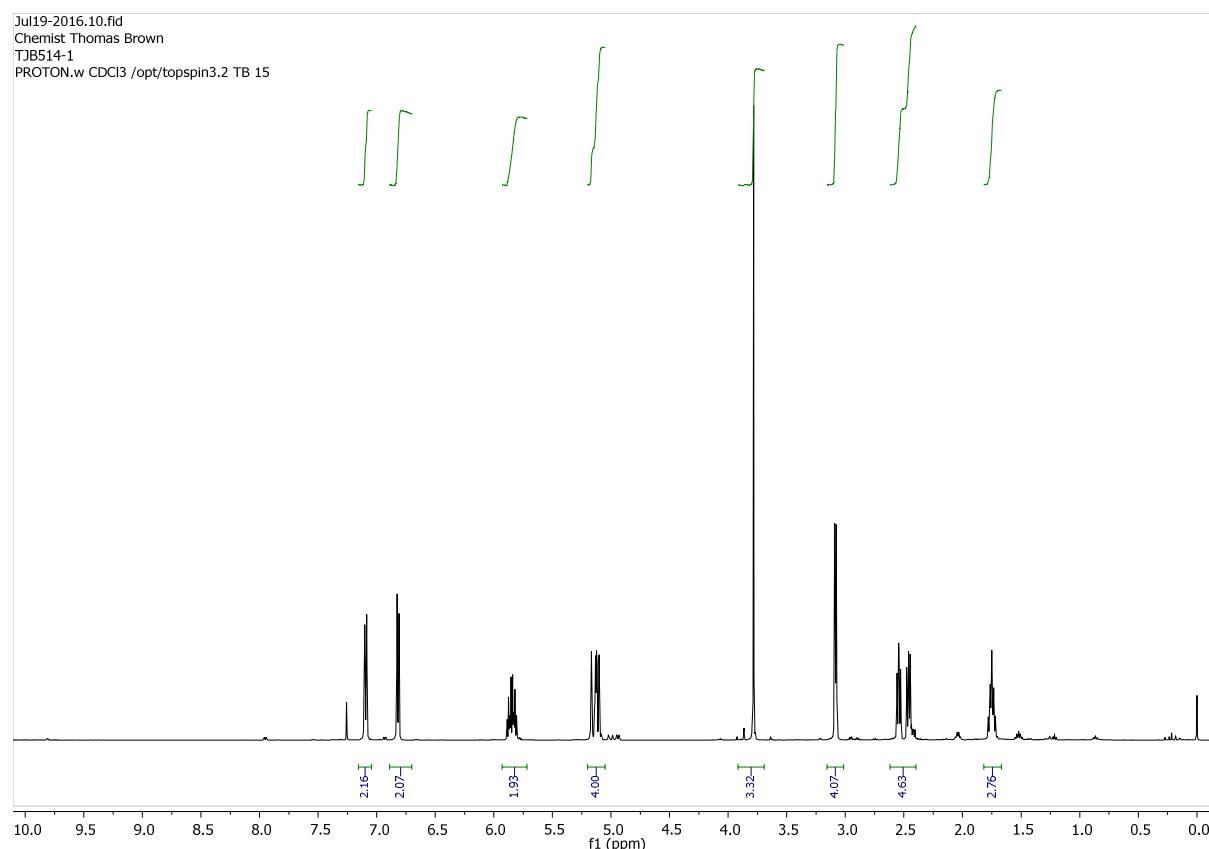
<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>).



*N*-Allyl-*N*-(3-(4-Methoxyphenyl)propyl)prop-2-en-1-amine **68**.



$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ).



<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>).

Chemist Thomas Brown  
TJB514-1  
C13APT.w CDCl<sub>3</sub> /opt/topspin3.2 TB 15



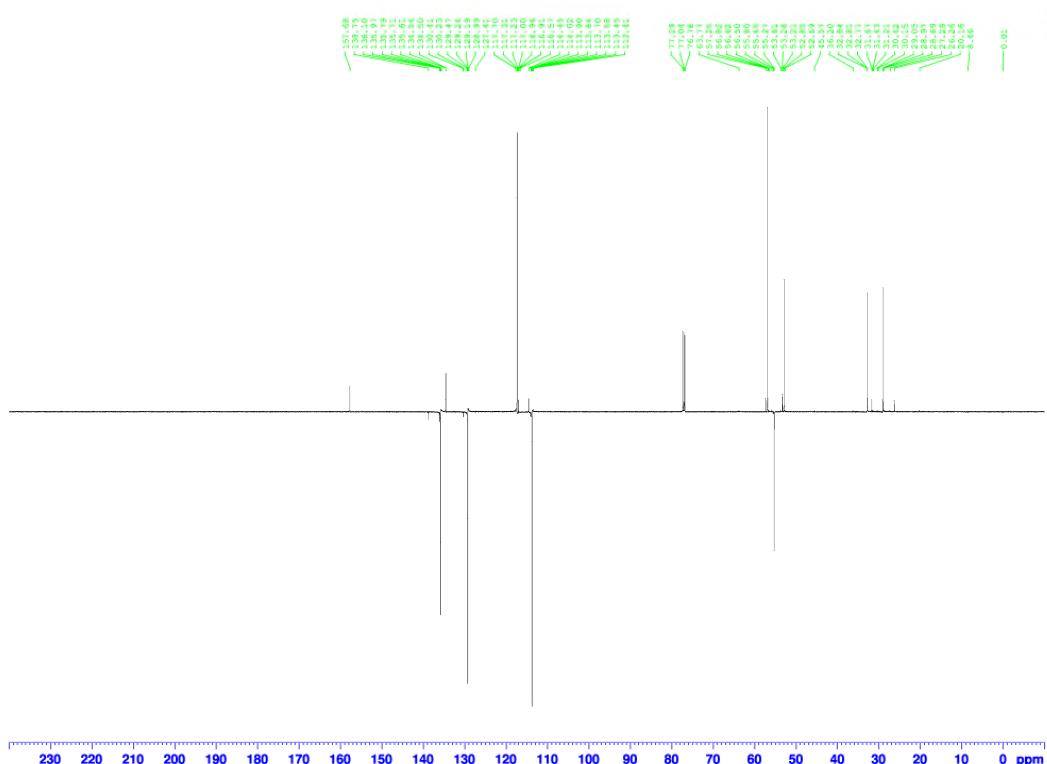
Current Data Parameters  
NAME July19-2016  
EXPNO 14  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20160719  
Time 15.19  
INSTRUM spect  
PROBHD 5 mm CPDCH 13C  
PULPROG jmod  
TD 65536  
SOLVENT CDCl<sub>3</sub>  
NS 256  
DS 4  
SWH 32894.738 Hz  
FIDRES 0.501934 Hz  
AQ 0.9900 sec  
RG 185.92  
DW 15.200 usec  
DE 66.43 usec  
TE 290.0 K  
CNST2 145.0000000  
CNST11 1.0000000  
D1 2.0000000 sec  
D20 0.00689655 sec  
TD0 1

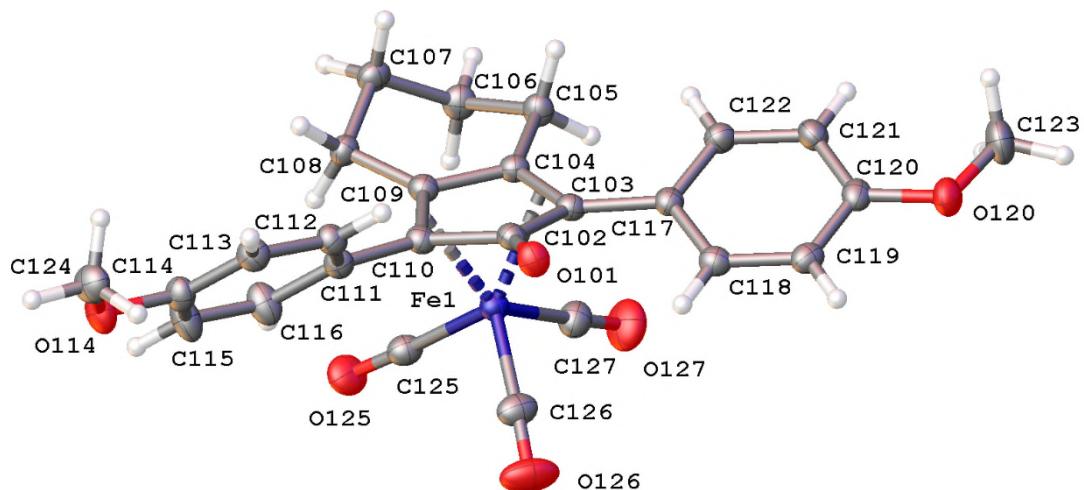
===== CHANNEL f1 =====  
SFO1 125.728795 MHz  
NUC1 <sup>13</sup>C  
P1 9.0 usec  
P2 19.00 usec  
PLW1 24.0000000 W

===== CHANNEL f2 =====  
SFO2 500.1320005 MHz  
NUC2 <sup>1</sup>H  
CPDPRG12 waltz16  
PCPD2 65.00 usec  
PLW2 13.0000000 W  
PLW12 0.26784000 W

F2 - Processing parameters  
SI 32768  
SF 125.757785 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

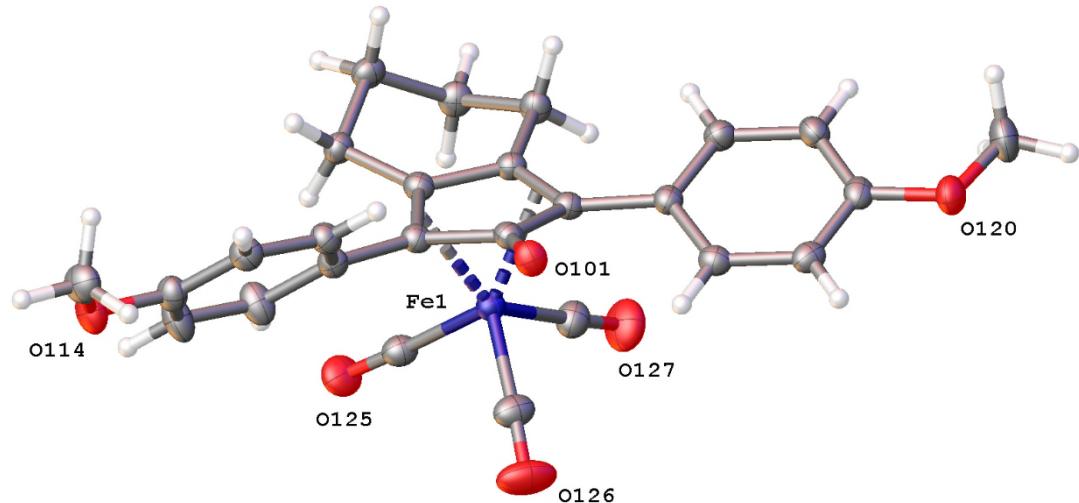


X-ray crystallographic data for tricarbonyl(1,8-bis(4-methoxyphenyl)octa-1,7-diyne)iron **6**  
(local code MC1), CCDC 1560655:



*Solid state structure of one of the crystallographically independent but chemically identical molecules in the asymmetric unit of mc1 with atom labeling and thermal ellipsoids drawn at 50% probability level.*

*Below is the same picture but just Fe and oxygen labeled.*



**Crystal structure determination of **6** (MC1).**

The asymmetric unit contains two crystallographically independent but chemically identical Fe complexes. Four molecules in the unit cell. Nothing unusual to report with regards to the refinement or crystal packing.

**Experimental**

Single crystals of  $C_{26}H_{22}FeO_6$  were grown from EtOAc. A suitable crystal was selected and mounted on a glass fibre with Fromblin oil and placed on an Xcalibur Gemini diffractometer with a Ruby CCD area detector. The crystal was kept at 150(2) K during data collection. Using Olex2 [1], the structure was solved with the ShelXT [2] structure solution program

using Direct Methods and refined with the XL [3] refinement package using Least Squares minimisation.

1 Dolomanov, O.V., Bourhis, L.J., Gildea, R.J., Howard, J.A.K. & Puschmann, H. (2009), *J. Appl. Cryst.* 42, 339-341.

2 Sheldrick, G.M. (2015). *Acta Cryst. A*71, 3-8.

3 Sheldrick, G.M. (2008). *Acta Cryst. A*64, 112-122.

**Crystal Data** for  $C_{26}H_{22}FeO_6$  ( $M = 486.28$  g/mol): triclinic, space group P-1 (no. 2),  $a = 11.3053(2)$  Å,  $b = 13.6069(3)$  Å,  $c = 16.3832(4)$  Å,  $\alpha = 105.526(2)^\circ$ ,  $\beta = 92.8266(19)^\circ$ ,  $\gamma = 109.973(2)^\circ$ ,  $V = 2254.47(10)$  Å<sup>3</sup>,  $Z = 4$ ,  $T = 150(2)$  K,  $\mu(\text{CuK}\alpha) = 5.709$  mm<sup>-1</sup>,  $D_{\text{calc}} = 1.433$  g/cm<sup>3</sup>, 23003 reflections measured ( $7.254^\circ \leq 2\Theta \leq 156.258^\circ$ ), 9502 unique ( $R_{\text{int}} = 0.0322$ ,  $R_{\text{sigma}} = 0.0383$ ) which were used in all calculations. The final  $R_1$  was 0.0359 ( $I > 2\sigma(I)$ ) and  $wR_2$  was 0.0994 (all data).

**Table S1 Crystal data and structure refinement for 6 (MC1).**

Identification code	MC1
Empirical formula	$C_{26}H_{22}FeO_6$
Formula weight	486.28
Temperature/K	150(2)
Crystal system	Triclinic
Space group	P-1
$a/\text{\AA}$	11.3053(2)
$b/\text{\AA}$	13.6069(3)
$c/\text{\AA}$	16.3832(4)
$\alpha/^\circ$	105.526(2)
$\beta/^\circ$	92.8266(19)
$\gamma/^\circ$	109.973(2)
Volume/Å <sup>3</sup>	2254.47(10)
Z	4
$\rho_{\text{calc}}/\text{g/cm}^3$	1.433
$\mu/\text{mm}^{-1}$	5.709
F(000)	1008.0
Crystal size/mm <sup>3</sup>	0.4 × 0.25 × 0.08 yellow block
Radiation	CuK $\alpha$ ( $\lambda = 1.54184$ )

2Θ range for data collection/°	7.254 to 156.258
Index ranges	-13 ≤ h ≤ 14, -17 ≤ k ≤ 16, -20 ≤ l ≤ 20
Reflections collected	23003
Independent reflections	9502 [ $R_{\text{int}} = 0.0322$ , $R_{\text{sigma}} = 0.0383$ ]
Data/restraints/parameters	9502/0/599
Goodness-of-fit on $F^2$	1.032
Final R indexes [ $I \geq 2\sigma(I)$ ]	$R_1 = 0.0359$ , $wR_2 = 0.0973$
Final R indexes [all data]	$R_1 = 0.0381$ , $wR_2 = 0.0994$
Largest diff. peak/hole / e Å <sup>-3</sup>	0.66/-0.39