

Synthesis, photolysis studies and *in vitro* photorelease of photolabile TRPV1 agonists and antagonists

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Electronic Supplementary Information

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General experimental methods

¹H NMR spectra were recorded at 300 MHz or 400 MHz, on Bruker Avance spectrometers, using deuteriochloroform (or other indicated solvent) as reference and internal deuterium lock. The chemical shift data for each signal are given as δ in units of parts per million (ppm) relative to tetramethylsilane (TMS) where $\delta_{\text{TMS}} = 0.00$ ppm. The multiplicity of each signal is indicated by: s (singlet); br s (broad singlet); d (doublet); dd (doublet of doublets); m (multiplet) etc. ¹³C NMR spectra were recorded at 75.5 MHz or 100 MHz using the DEPT Q pulse sequence with broadband proton decoupling and internal deuterium lock. The chemical shift data for each signal are given as δ in units of ppm relative to TMS where $\delta_{\text{TMS}} = 0.00$ ppm. Identical proton coupling constants (*J*) are averaged in each spectrum and reported to the nearest 0.1 Hz. Low and high resolution mass spectra were recorded on a Micromass LCT spectrometer using electrospray ionisation in either positive or negative polarity (ES⁺ or ES⁻). Certain samples were submitted to the National Mass Spectrometry Centre, Swansea. *m/z* values are reported in Daltons and followed by their percentage abundance in parentheses. Microanalyses were obtained on a Carlo Erber EA1110 analyser by the St Andrews University microanalysis service. IR spectra were recorded on a Perkin-Elmer GX FT-IR spectrometer as thin films between sodium chloride discs or as potassium bromide disks as indicated. Absorption maxima are reported in wavenumbers (cm⁻¹). Melting points were determined on an Electrothermal 9100 and are uncorrected. Optical rotations were measured on a Perkin-Elmer 341 polarimeter using cells with a path length of 1 dm. The concentration (*c*) is expressed in g/100 mL (equivalent to g/0.1 dm³). Specific rotations are denoted $[\alpha]_D^T$ and are given in implied units of 10⁻¹ deg cm² g⁻¹ (T= ambient temperature in °C). Analytical thin-layer chromatography (TLC) was carried out on Merck silica gel 60 F₂₅₄ pre-coated aluminium-backed plates. Visualisation of the plates was achieved using a UV lamp (λ_{max} 254 or 365 nm) or thermal development after dipping in either an ethanolic solution of phosphomolybdic acid or an ethanolic solution of 4-anisaldehyde, sulfuric acid and acetic acid. Flash column chromatography was carried out using Merck silica gel 60 (240-400 mesh), under a positive pressure of compressed air. Anhydrous CH₂Cl₂, THF, diethyl ether and hexane were obtained using a MBRAUN GmbH MB SPS-800 solvent purification system. Anhydrous DMF was purchased from Sigma Aldrich, UK and used without further purification. Where appropriate and if not stated otherwise, all non aqueous reactions were performed under an inert atmosphere of nitrogen or argon in flame-dried glassware, using a vacuum manifold with nitrogen or argon passed through 4 Å molecular sieves and self-indicating silica gel. *In vacuo* refers to the use of a rotary evaporator attached to a diaphragm pump. Hexane refers to a mixture of hexanes and brine refers to a saturated aqueous solution of sodium chloride.

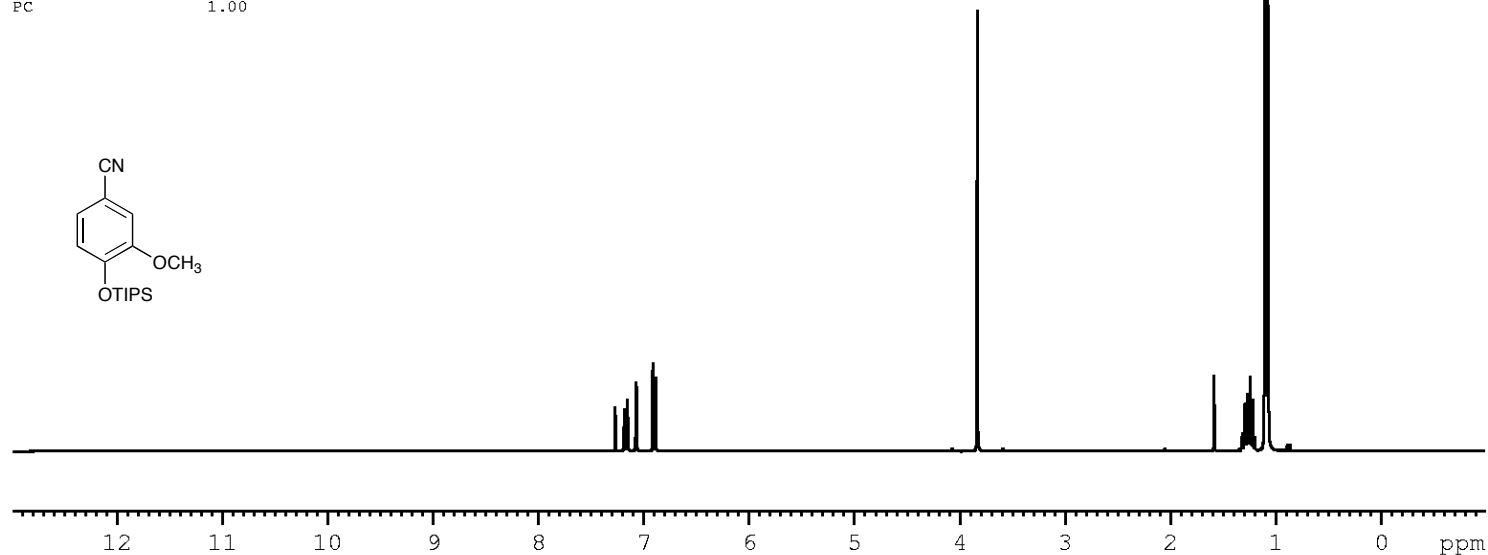
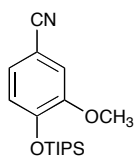
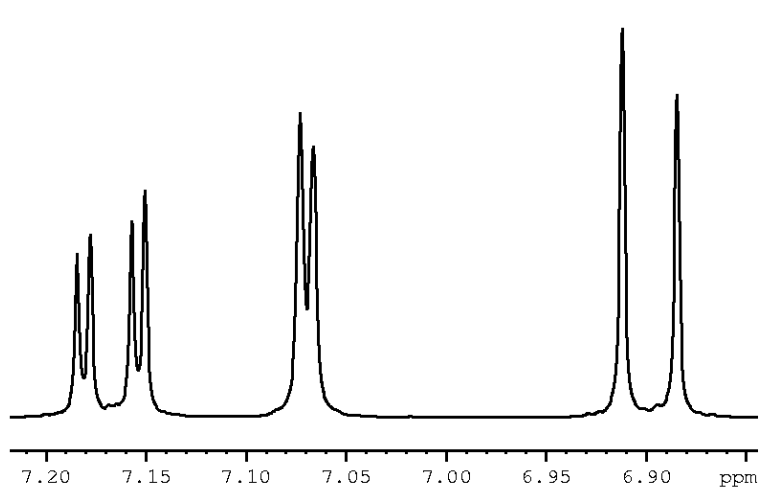
3-Methoxy-4-(triisopropoxy)benzonitrile (12) ¹H NMR spectrum

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PULPROG    zg30
TD         32768
SOLVENT    CDCl3
NS         16
DS         2
SWH        4194.631 Hz
FIDRES     0.128010 Hz
AQ         3.9059956 sec
RG         228.1
DW         119.200 usec
DE         6.00 usec
TE         295.9 K
D1         1.00000000 sec
TD0        1
    
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PL1        10.00 dB
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SI         32768
SF         300.0600000 MHz
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3-Methoxy-4-(triisopropoxy)benzonitrile (12) ¹³C NMR spectrum

```

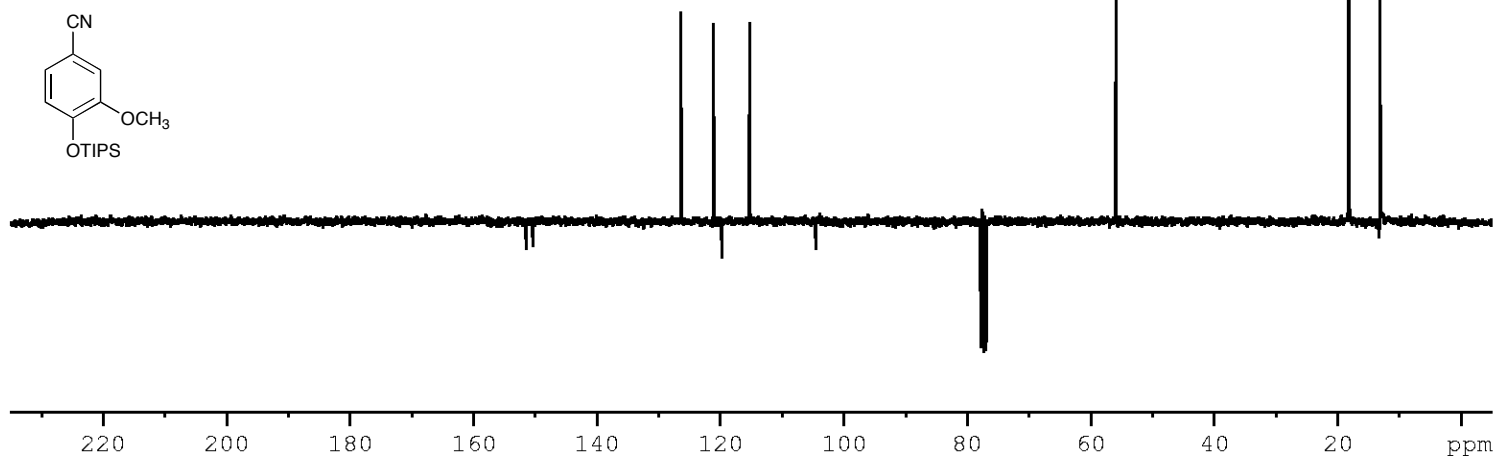
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FIDRES     0.276427 Hz
AQ         1.8088436 sec
RG         18390.4
DW         27.600 usec
DE         6.00 usec
TE         293.9 K
CNST2      145.0000000
CNST3      1.0000000
CNST4      5.0000000
D1         1.50000000 sec
d2         0.00172414 sec
d3         0.00431034 sec
D12        0.00002000 sec
D13        0.00000400 sec
    
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```

===== CHANNEL f1 =====
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p2         15.00 usec
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SFO1       75.4587938 MHz
    
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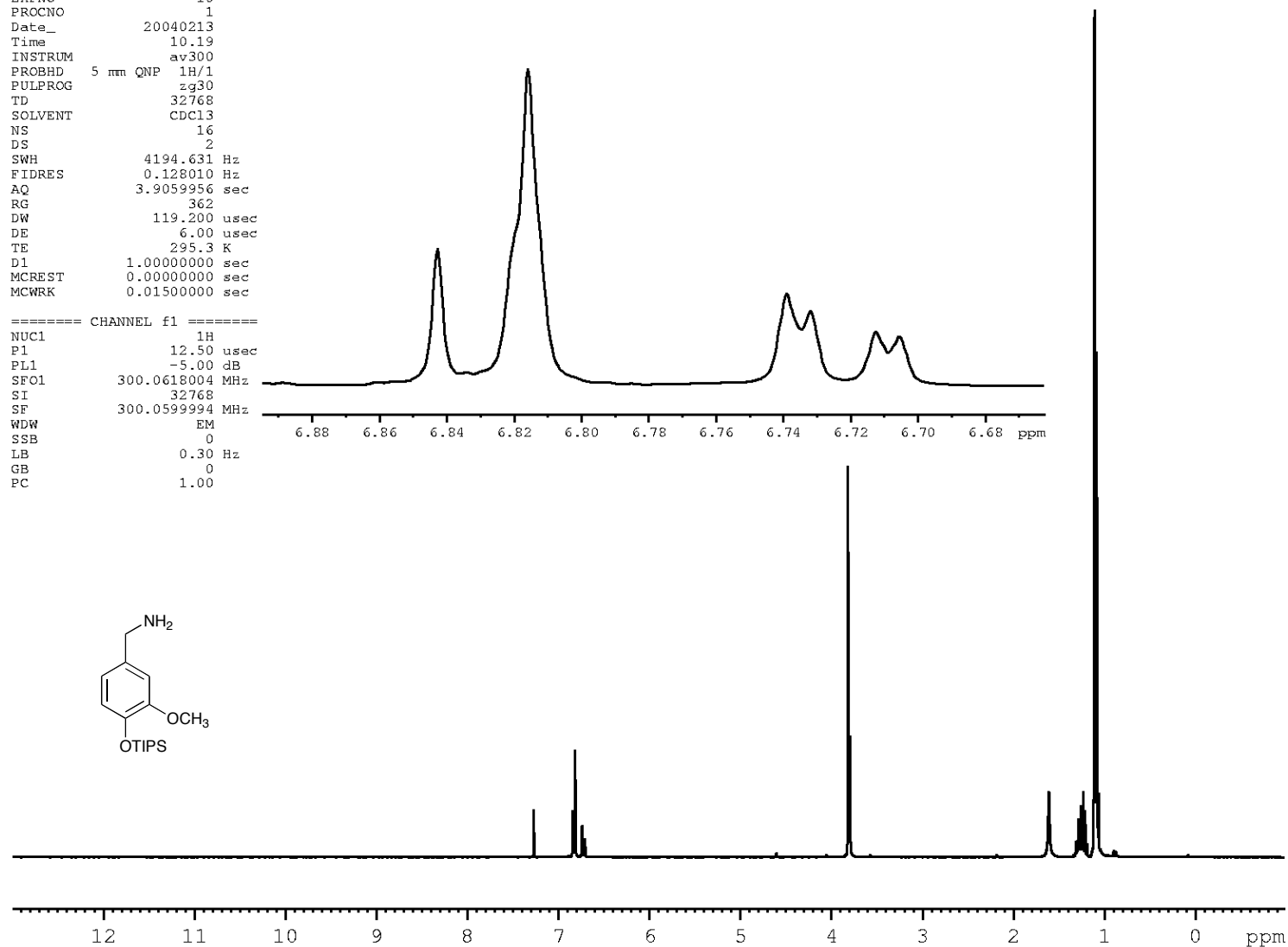
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PL12       27.47 dB
SFO2       300.0615003 MHz
SI         32768
SF         75.4501170 MHz
WDW        EM
SSB        0
LB         2.00 Hz
GB         0
PC         1.40
    
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3-Methoxy-4-(triisopropoxy)benzylamine (13) crude ^1H NMR spectrum

NAME 02132004-4-projects
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 PROCNO 1
 Date_ 20040213
 Time_ 10.19
 INSTRUM av300
 PROBHD 5 mm QNP 1H/1
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 4194.631 Hz
 FIDRES 0.128010 Hz
 AQ 3.9059956 sec
 RG 362
 DW 119.200 usec
 DE 6.00 usec
 TE 295.3 K
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 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

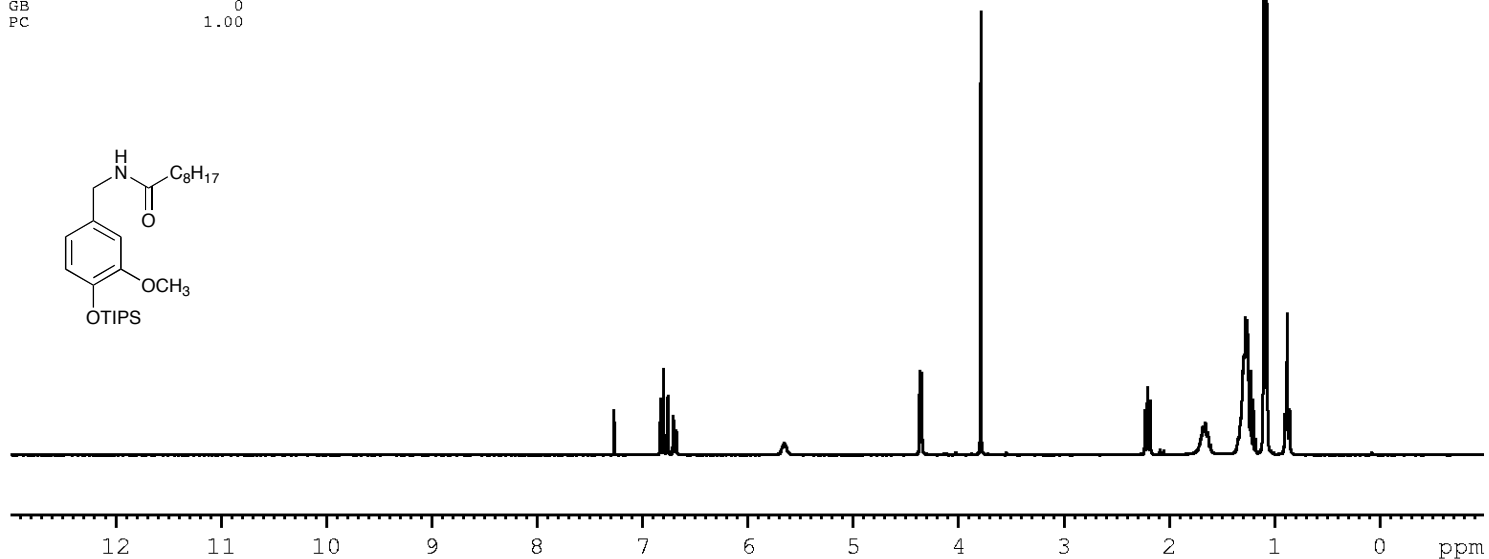
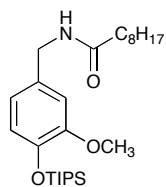
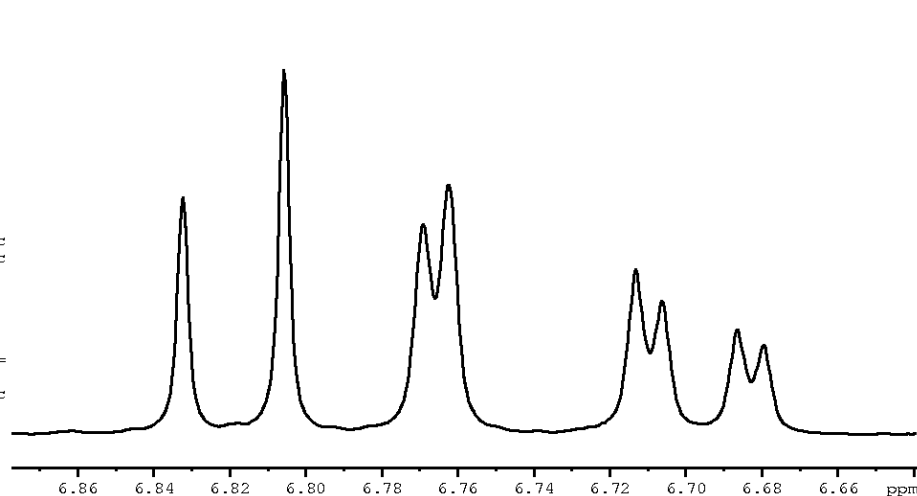
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 SF 300.0599994 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



3-Methoxy-4-(triisopropylsilyloxy)-N-(nonanoyl)benzylamine (14) ^1H NMR spectrum

NAME 04262004-7-projects
 EXPNO 10
 PROCNO 1
 Date_ 20040426
 Time_ 11.24
 INSTRUM av300
 PROBHD 5 mm QNP 1H/1
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 4194.631 Hz
 FIDRES 0.128010 Hz
 AQ 3.9059956 sec
 RG 362
 DW 119.200 usec
 DE 6.00 usec
 TE 294.9 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 8.50 usec
 PL1 8.00 dB
 SFO1 300.0618004 MHz
 SI 32768
 SF 300.0599986 MHz
 WDW EM
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 GB 0
 PC 1.00



3-Methoxy-4-(triisopropylsilyloxy)-N-(nonanoyl)benzylamine (14) ¹³C NMR spectrum

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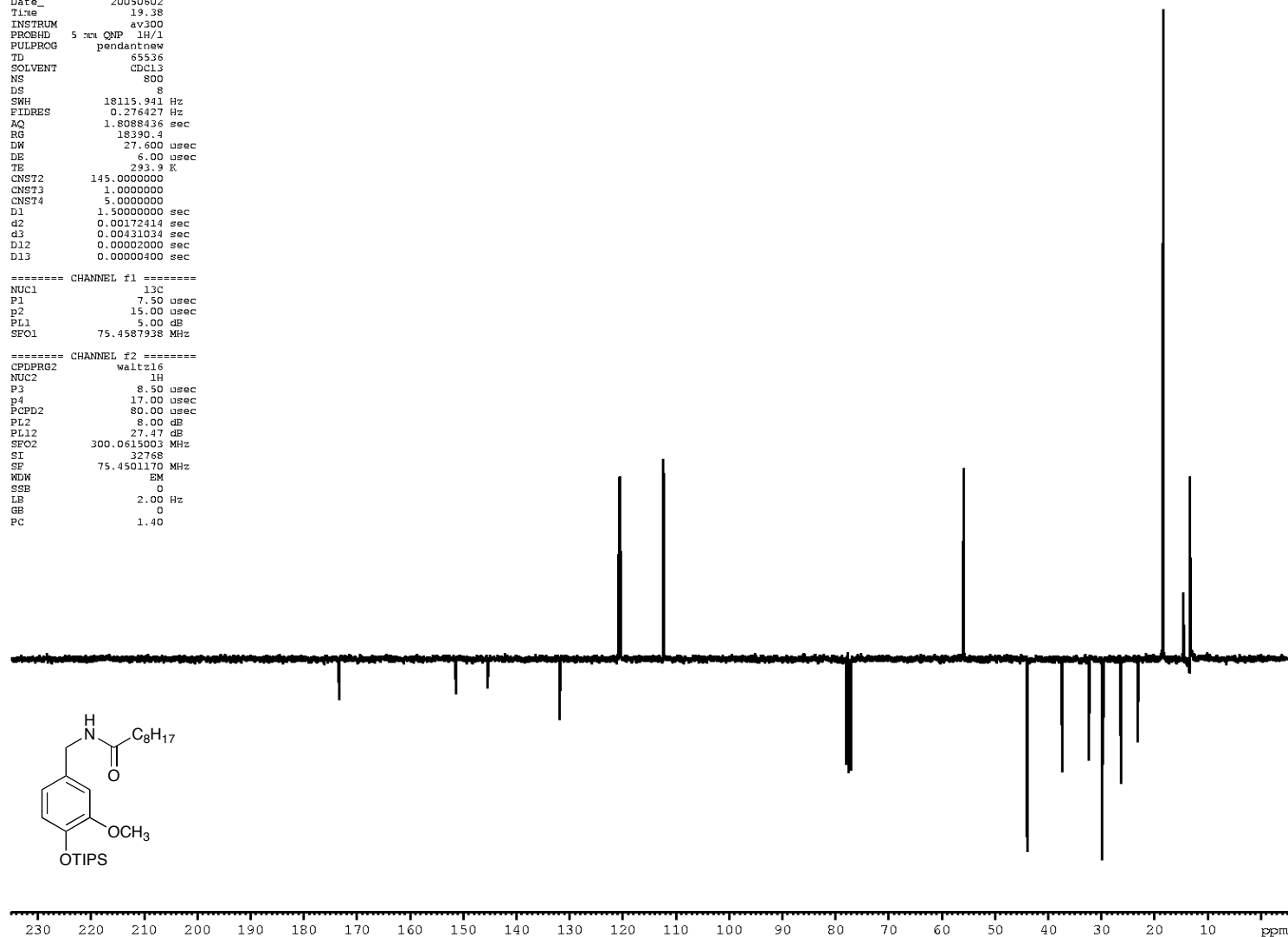
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PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         800
DS         8
SWH        18115.941 Hz
FIDRES     0.276427 Hz
AQ         1.8088436 sec
RG         18390.4
DW         27.600 usec
DE         8.00 usec
TE         293.9 K
CNST2     145.0000000
CNST3     1.0000000
CNST4     5.0000000
D1         1.50000000 sec
d2         0.00172414 sec
d3         0.00431034 sec
d12        0.00002000 sec
d13        0.00000400 sec
  
```

```

===== CHANNEL f1 =====
NUC1       13C
P1         7.50 usec
p2         15.00 usec
PL1        9.00 dB
SFO1       75.4587938 MHz
  
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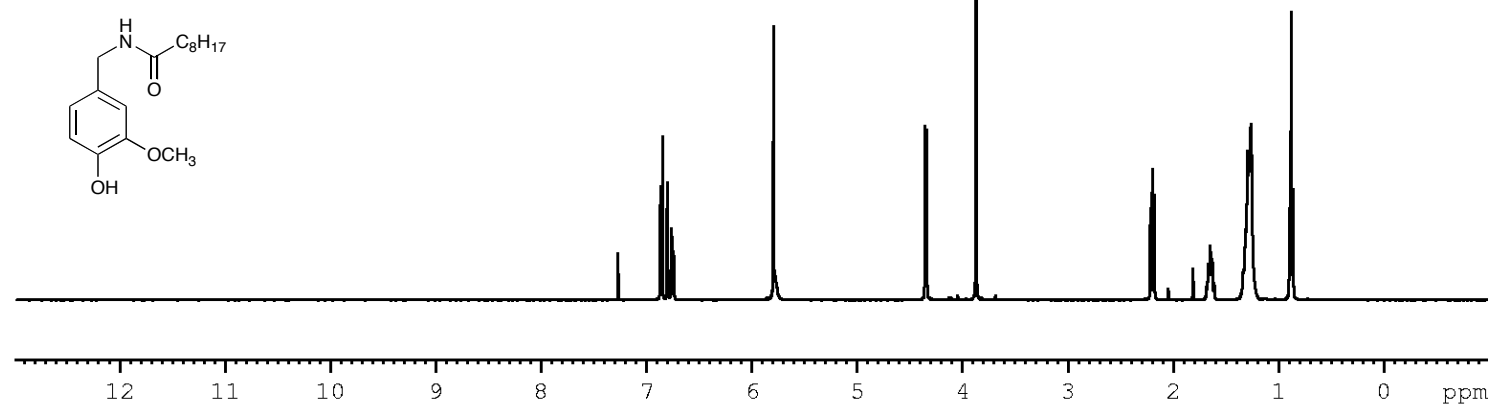
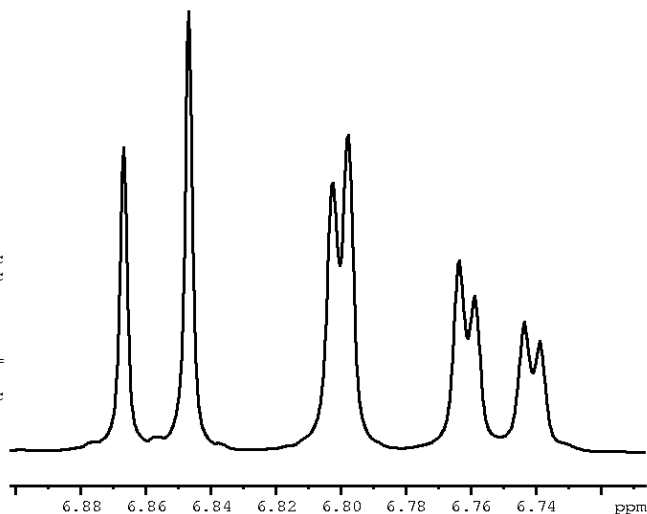
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NUC2       1H
P3         8.50 usec
p4         17.00 usec
PCPD2      80.00 usec
PL2        8.00 dB
PL12       27.47 dB
SFO2       300.0615003 MHz
SI         32768
SF         75.4501170 MHz
WDW         EM
SSB         0
LB         2.00 Hz
GB         0
PC         1.40
  
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4-Hydroxy-3-methoxy-N-(nonanoyl)benzylamine (2) ¹H NMR spectrum

NAME 06112008-15-meganM
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 PROCNO 1
 Date_ 20080611
 Time_ 18.11
 INSTRUM AVII400
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 2
 SWH 5597.015 Hz
 FIDRES 0.085404 Hz
 AQ 5.8545995 sec
 RG 128
 DW 89.333 usec
 DE 6.00 usec
 TE 295.4 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 12.00 usec
 PL1 -2.00 dB
 PL1W 15.04845142 W
 SFO1 400.1324008 MHz
 SI 32768
 SF 400.1300075 MHz
 WDW EM
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 LB 0.30 Hz
 GB 0
 PC 1.00



4-Hydroxy-3-methoxy-N-(nonanoyl)benzylamine (2) ¹³C NMR spectrum

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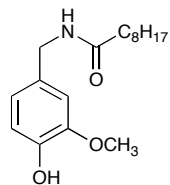
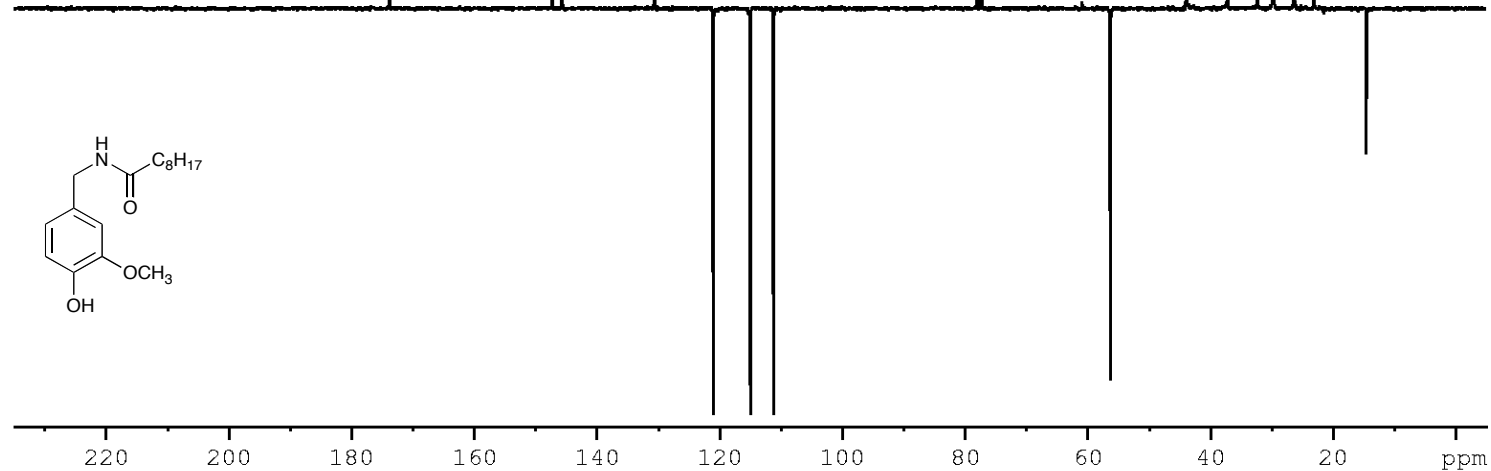
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PULPROG   pendantnew
TD         65536
SOLVENT   CDCl3
NS         800
DS         8
SWH        18115.941 Hz
FIDRES     0.276427 Hz
AQ         1.8088436 sec
RG         18390.4
DW         27.600 usec
DE         6.00 usec
TE         296.0 K
CNST2     145.0000000
CNST3     1.0000000
CNST4     5.0000000
D1         1.50000000 sec
d2         0.00172414 sec
d3         0.00431034 sec
D12        0.00002000 sec
D13        0.00000400 sec
    
```

```

===== CHANNEL f1 =====
NUC1       13C
P1         7.50 usec
p2         15.00 usec
PL1        5.00 dB
SFO1       75.4587938 MHz
    
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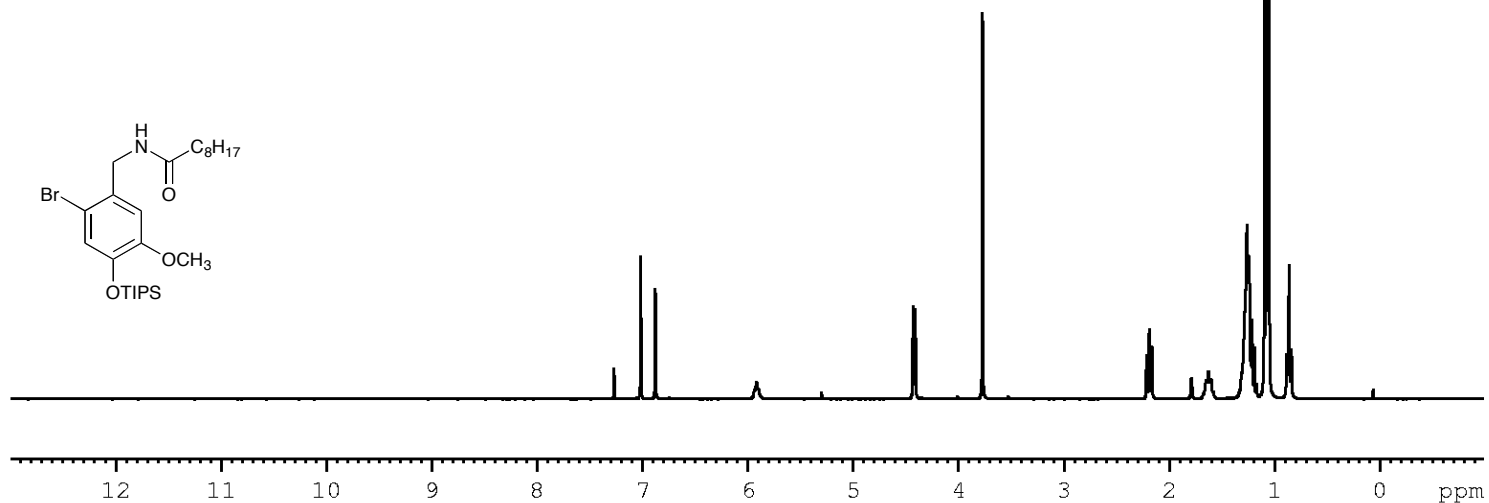
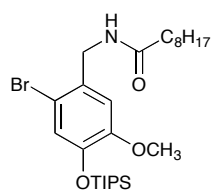
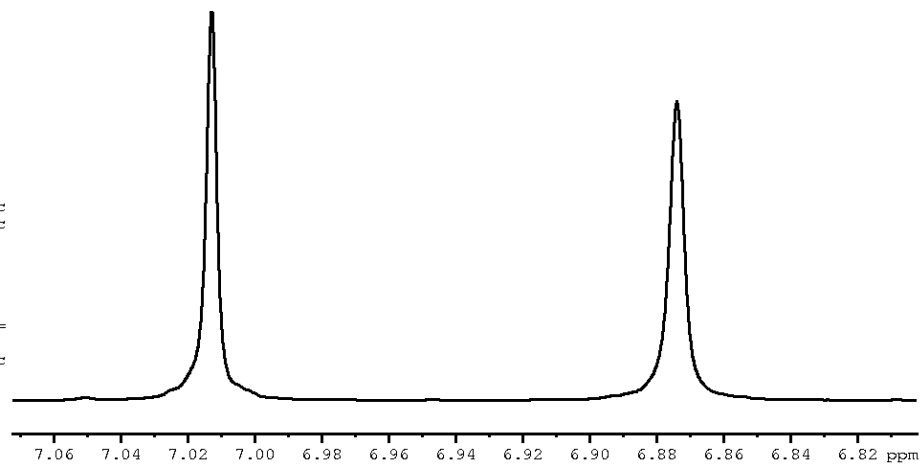
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NUC2       1H
P3         8.50 usec
p4         17.00 usec
PCPD2      80.00 usec
PL2        8.00 dB
PL12       27.47 dB
SFO2       300.0615003 MHz
SI         32768
SF         75.4501170 MHz
WDW        EM
SSB        0
LB         2.00 Hz
GB         0
PC         1.40
    
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2-Bromo-5-methoxy-4-(triisopropylsilyloxy)-N-(nonanoyl)benzylamine (15) ^1H NMR spectrum

NAME 06092005-24-michael
 EXPNO 10
 PROCNO 1
 Date_ 20050610
 Time_ 1.28
 INSTRUM av300
 PROBHD 5 mm QNP 1H/1
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 4194.631 Hz
 FIDRES 0.128010 Hz
 AQ 3.9059956 sec
 RG 128
 DW 119.200 usec
 DE 6.00 usec
 TE 293.8 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 8.50 usec
 PL1 8.00 dB
 SFO1 300.0618004 MHz
 SI 32768
 SF 300.0600007 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



2-Bromo-5-methoxy-4-(triisopropylsilyloxy)-N-(nonanoyl)benzylamine (15) ^{13}C NMR spectrum

```

NAME      06092005-24-michael
EXPNO     11
PROCNO    1
Date_     20050610
Time      2.14
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PROBHD    5 mm QNP 1H/1
PULPROG   pendantnew
TD        65536
SOLVENT   CDCl3
NS        800
DS        8
SWH       18115.941 Hz
FIDRES    0.276427 Hz
AQ        1.8088436 sec
RG        18390.4
DW        27.600 usec
DE        6.00 usec
TE        294.0 K
CNST2     145.0000000
CNST3     1.0000000
CNST4     5.0000000
D1        1.50000000 sec
d2        0.00172414 sec
d3        0.00431034 sec
D12       0.00002000 sec
D13       0.00004000 sec

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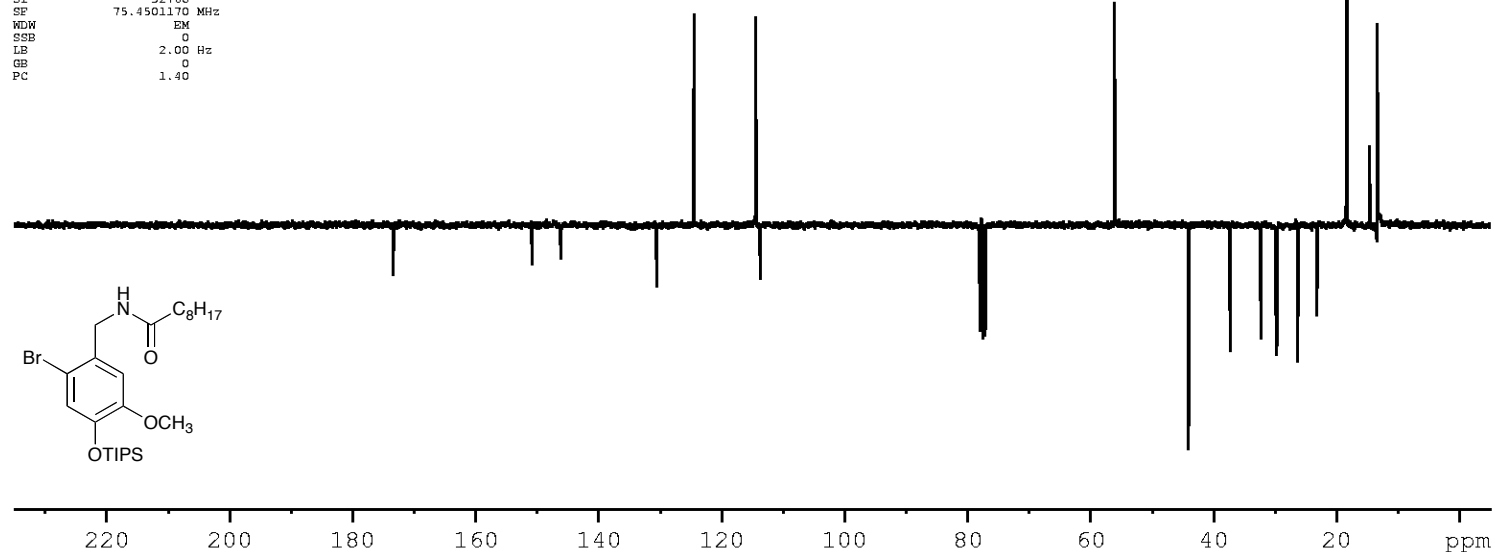
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NUC1      13C
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p2        15.00 usec
PL1       5.00 dB
SFO1      75.4587938 MHz

```

```

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
P3        8.50 usec
p4        17.00 usec
PCPD2     80.00 usec
PL2       8.00 dB
PL12      27.47 dB
SFO2      300.0615003 MHz
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LB        2.00 Hz
GB        0
PC        1.40

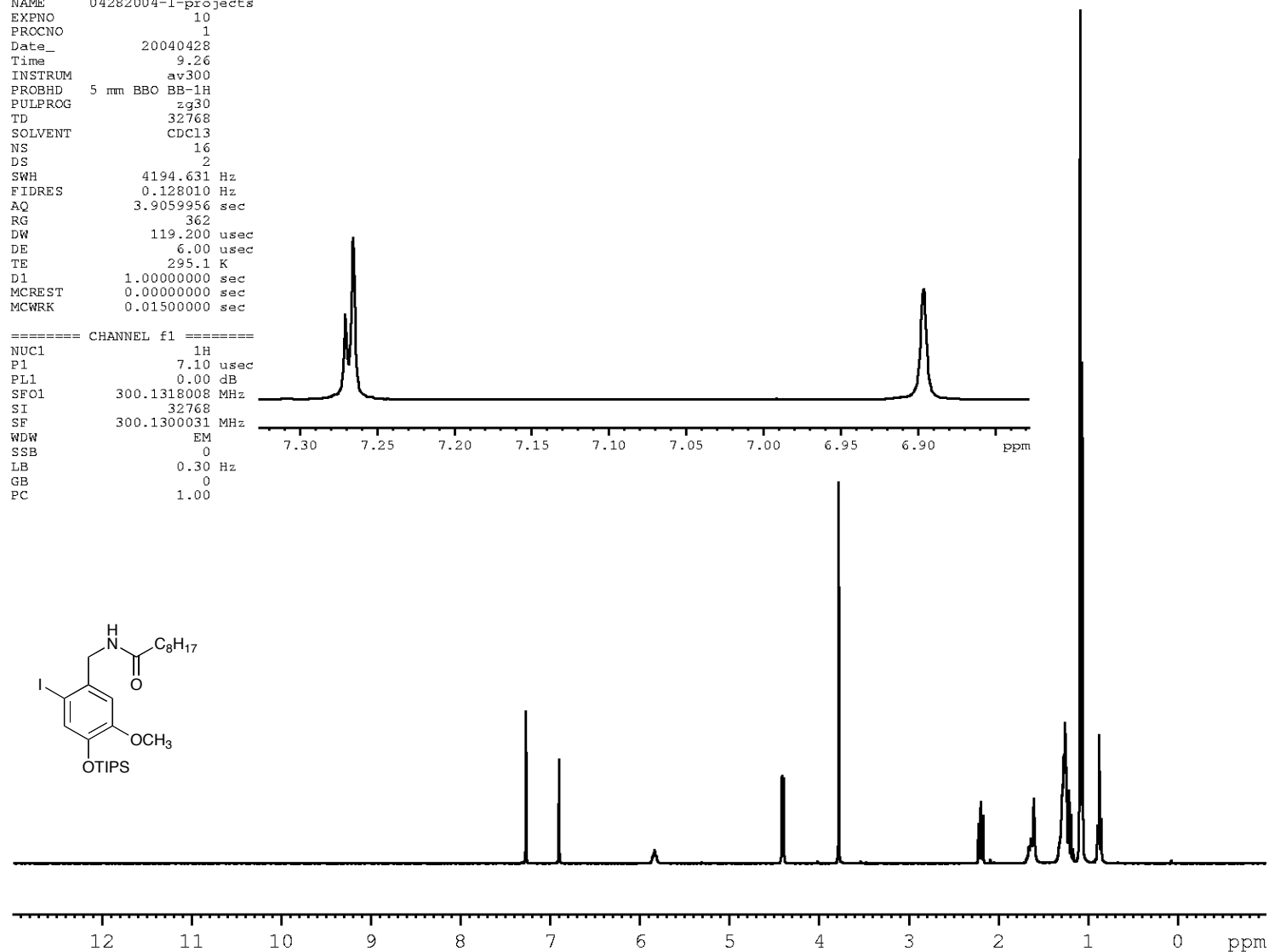
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6-Iodo-3-methoxy-4-(triisopropylsilyloxy)-N-(nonanoyl)benzylamine (16) ^1H NMR spectrum

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 Date_ 20040428
 Time_ 9.26
 INSTRUM av300
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 4194.631 Hz
 FIDRES 0.128010 Hz
 AQ 3.9059956 sec
 RG 362
 DW 119.200 usec
 DE 6.00 usec
 TE 295.1 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 7.10 usec
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 LB 0.30 Hz
 GB 0
 PC 1.00



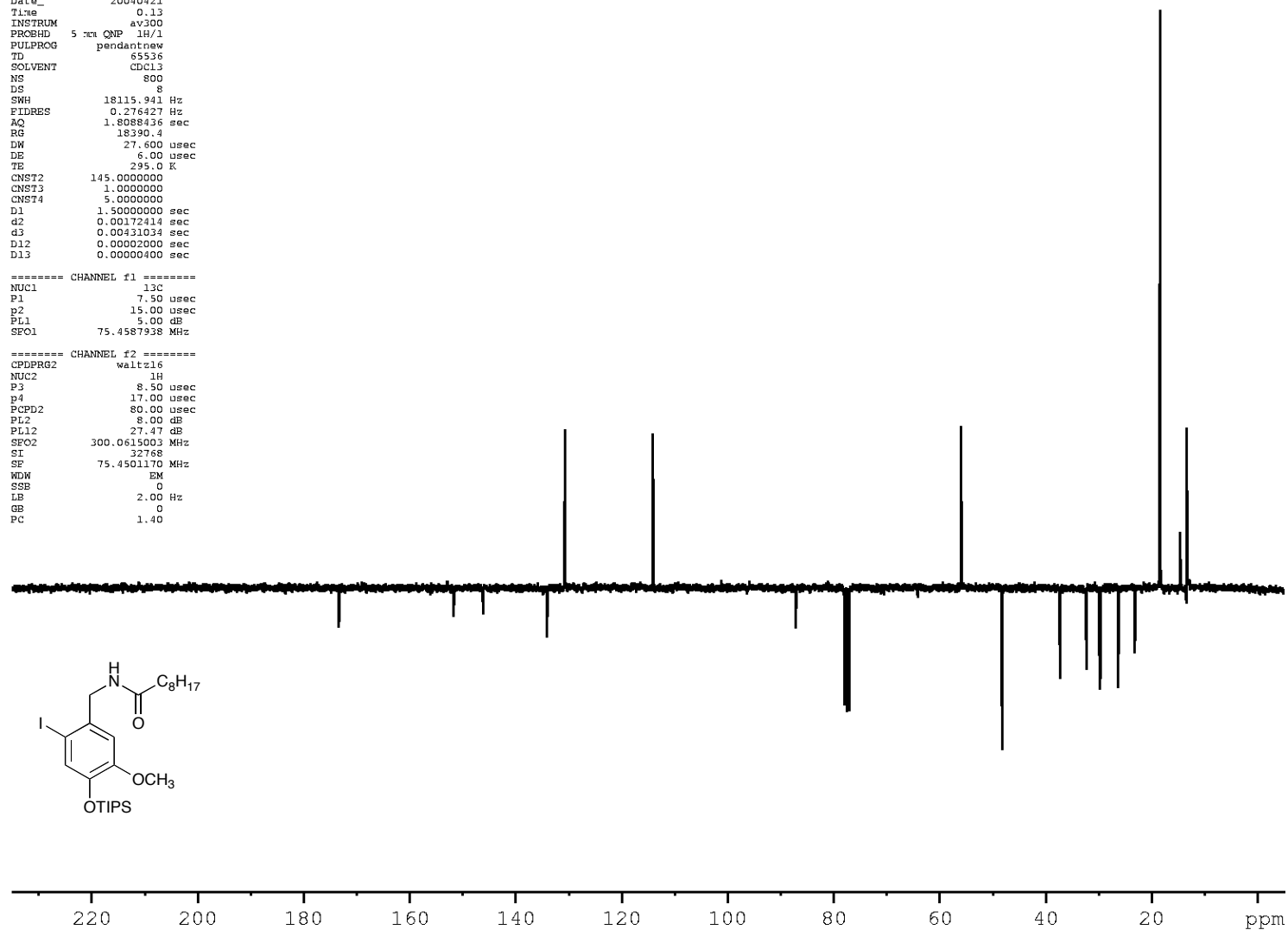
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```

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PULPROG   zgpg30
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SOLVENT   CDCl3
NS         800
DS         8
SWH        18115.941 Hz
FIDRES     0.276427 Hz
AQ         1.8088436 sec
RG         18390.4
DW         27.600 usec
DE         6.00 usec
TE         295.0 K
CNST2     145.0000000
CNST3     1.0000000
CNST4     5.0000000
D1         1.50000000 sec
d2         0.00172414 sec
d3         0.00431034 sec
D12        0.00002000 sec
D13        0.00000400 sec

===== CHANNEL f1 =====
NUC1       13C
P1         7.50 usec
p2         15.00 usec
PL1        5.00 dB
SFO1       75.4587938 MHz

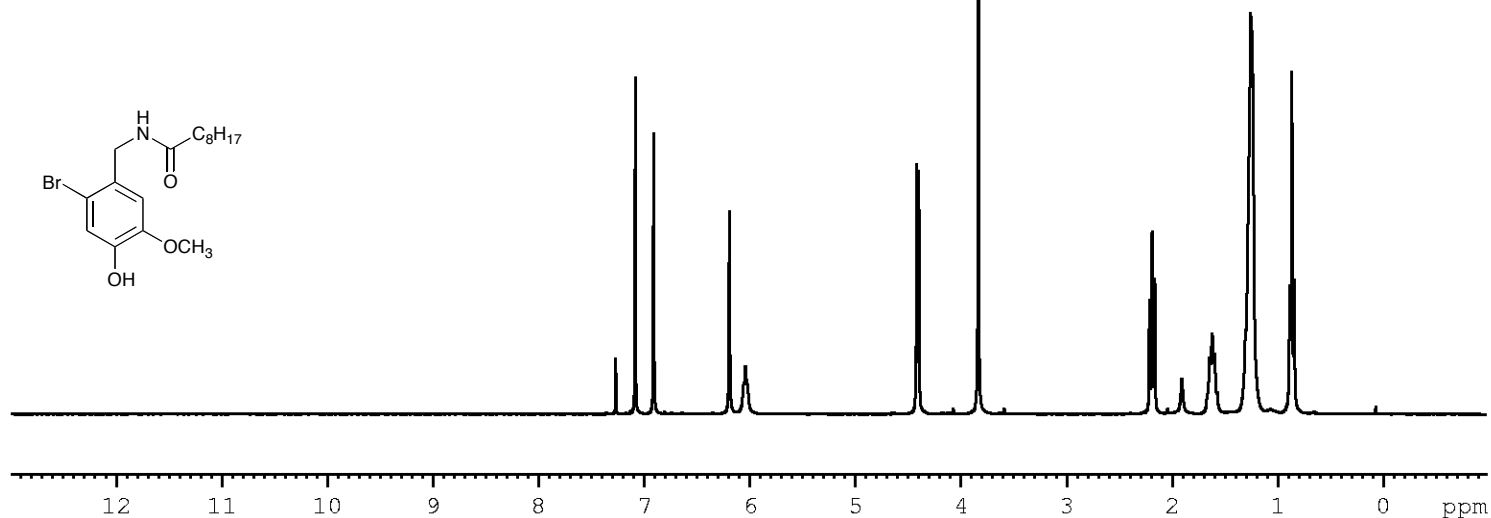
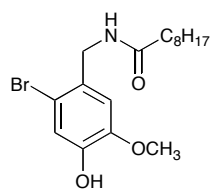
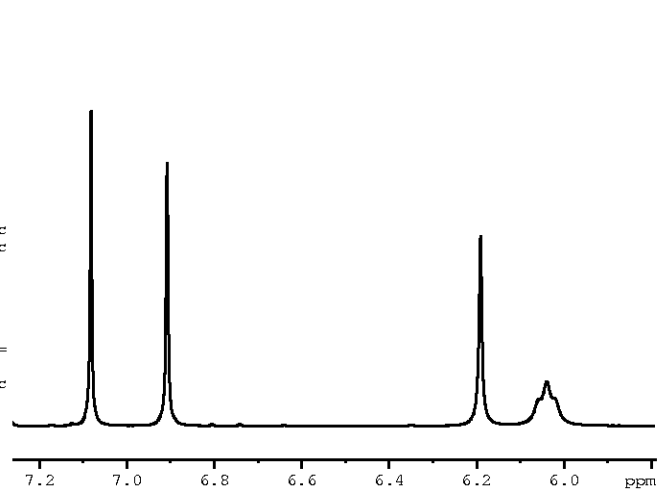
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NUC2       1H
P3         8.50 usec
p4         17.00 usec
PCPD2      80.00 usec
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PL12       27.47 dB
SFO2       300.0615003 MHz
SI         32768
SF         75.4501170 MHz
WDW        EM
SSB        0
LB         2.00 Hz
GB         0
PC         1.40
    
```



6-Bromo-4-hydroxy-3-methoxy-N-(nonanoyl)benzylamine (17) ^1H NMR spectrum

NAME 06102005-27-michael
 EXPNO 10
 PROCNO 1
 Date_ 20050610
 Time 14.02
 INSTRUM av300
 PROBHD 5 mm QNP 1H/1
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 4194.631 Hz
 FIDRES 0.128010 Hz
 AQ 3.9059956 sec
 RG 161.3
 DW 119.200 usec
 DE 6.00 usec
 TE 293.7 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 8.50 usec
 PL1 8.00 dB
 SFO1 300.0618004 MHz
 SI 32768
 SF 300.0600003 MHz
 WDW EM
 SSB 0
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 PC 1.00



6-Bromo-4-hydroxy-3-methoxy-N-(nonanoyl)benzylamine (17) ^{13}C NMR spectrum

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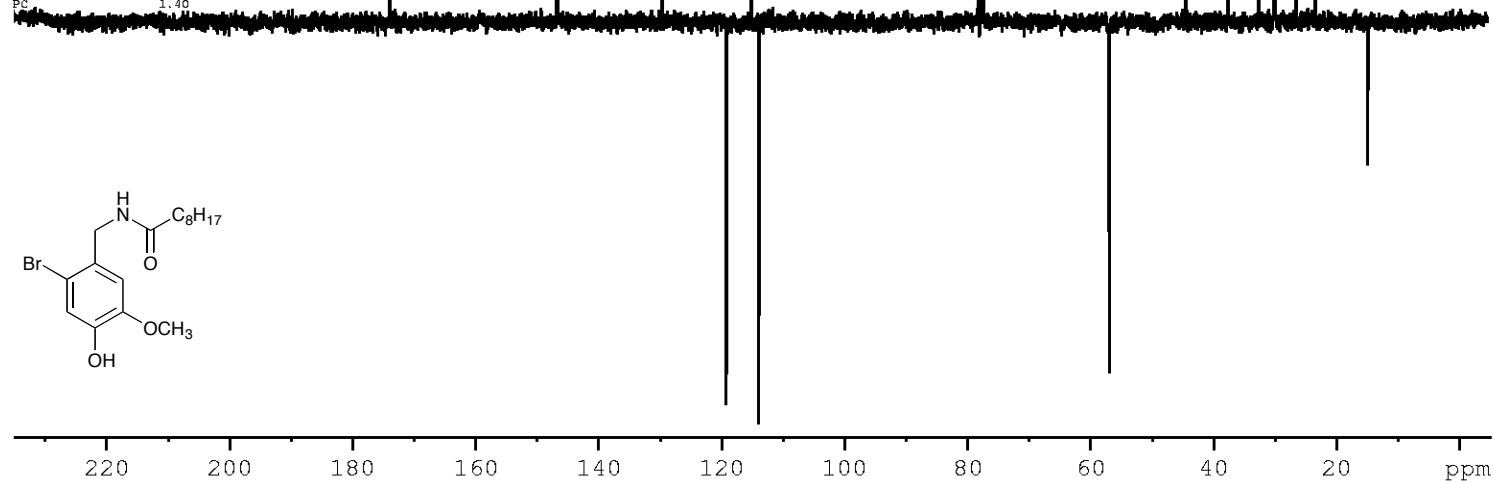
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TD         65536
SOLVENT   CDCl3
NS         800
DS         8
SWH        18115.941 Hz
FIDRES     0.276427 Hz
AQ         1.8088436 sec
RG         18390.4
DW         27.600 usec
DE         6.00 usec
TE         295.3 K
CNST2     145.0000000
CNST3     1.0000000
CNST4     5.0000000
D1         1.50000000 sec
d2         0.00172414 sec
d3         0.00431034 sec
D12        0.00002000 sec
D13        0.00000400 sec
    
```

```

===== CHANNEL f1 =====
NUC1       13C
P1         7.50 usec
P2         15.00 usec
PL1        5.00 dB
SFO1       75.4587938 MHz
    
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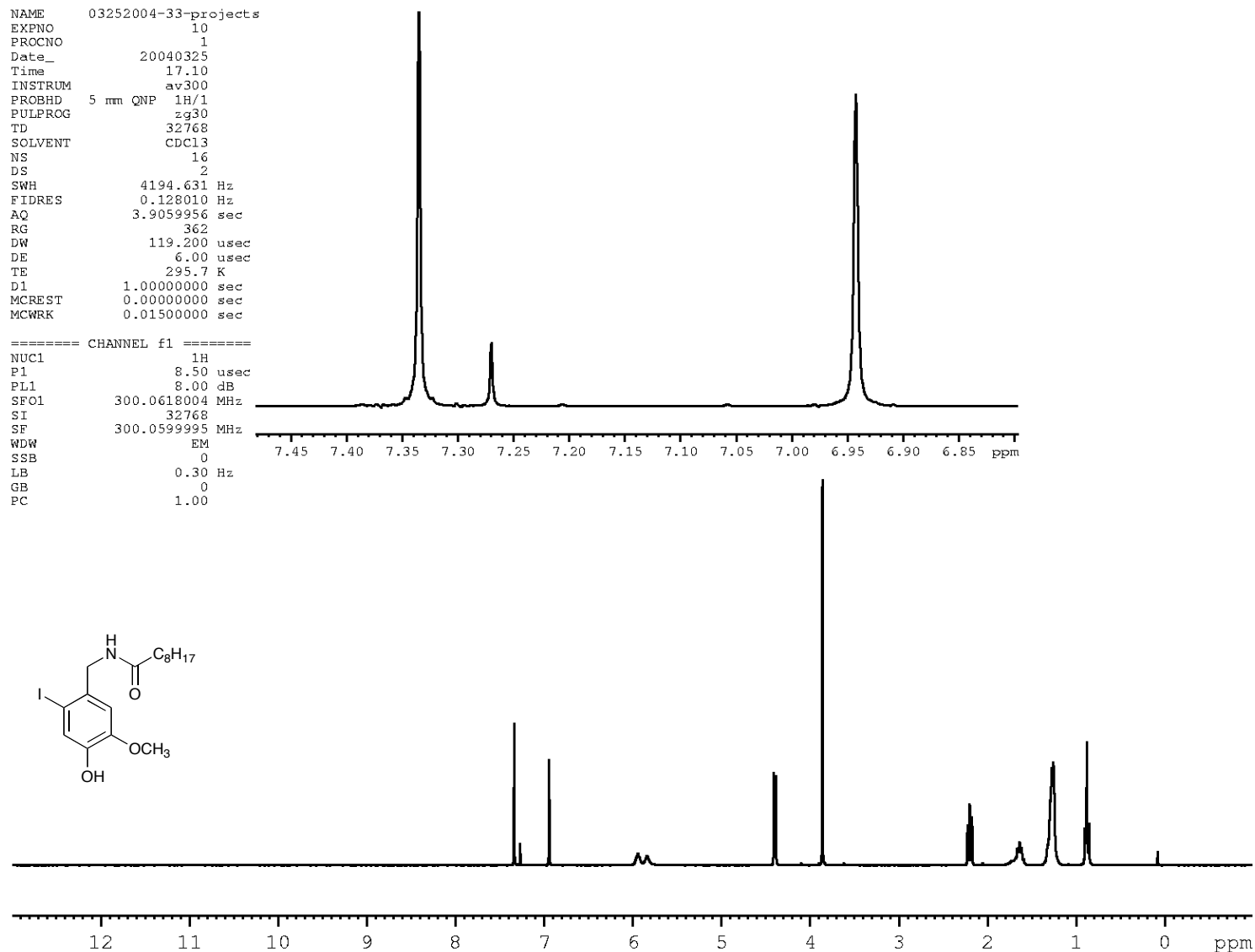
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CPDPRG2    waltz16
NUC2       1H
P3         8.50 usec
P4         17.00 usec
PCPD2      80.00 usec
PL2        8.00 dB
PL12       27.47 dB
SFO2       300.0615003 MHz
SI         32768
SF         75.4501004 MHz
WDW         EM
SSB         0
LB         2.00 Hz
GB         0
PC         1.40
    
```



4-Hydroxy-6-iodo-3-methoxy-N-(nonanoyl)benzylamine (18) ¹H NMR spectrum

NAME 03252004-33-projects
 EXPNO 10
 PROCNO 1
 Date_ 20040325
 Time 17.10
 INSTRUM av300
 PROBHD 5 mm QNP 1H/1
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 4194.631 Hz
 FIDRES 0.128010 Hz
 AQ 3.9059956 sec
 RG 362
 DW 119.200 usec
 DE 6.00 usec
 TE 295.7 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 8.50 usec
 PL1 8.00 dB
 SFO1 300.0618004 MHz
 SI 32768
 SF 300.0599995 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



4-Hydroxy-6-iodo-3-methoxy-N-(nonanoyl)benzylamine (18) ¹³C NMR spectrum

```

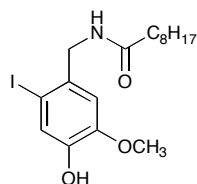
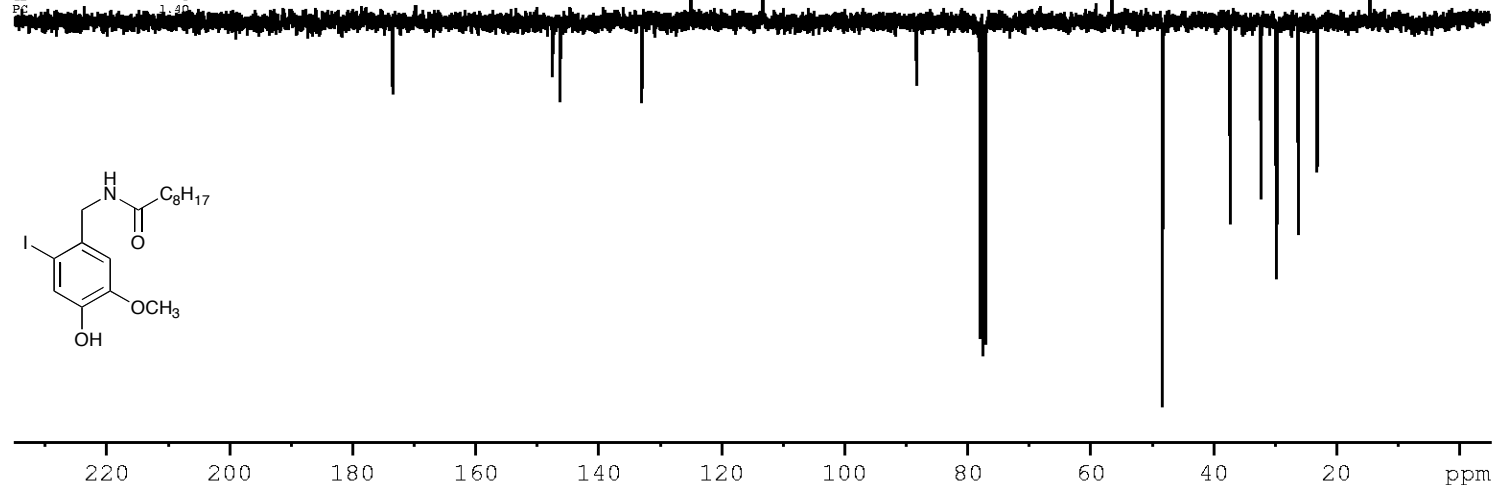
NAME      03252004-33-projects
EXPNO     11
PROCNO    1
Date_     20040325
Time      17.56
INSTRUM   av300
PROBHD    5 mm QNP 1H/1
PULPROG   zgpg30
TD        65536
SOLVENT   CDCl3
NS         800
DS         8
SWH        18115.941 Hz
FIDRES     0.276427 Hz
AQ         1.8088436 sec
RG         18390.4
DW         27.600 usec
DE         6.00 usec
TE         296.1 K
CNST2     145.0000000
CNST3     1.0000000
CNST4     5.0000000
D1         1.50000000 sec
d2         0.00172414 sec
d3         0.00431034 sec
D12        0.00002000 sec
D13        0.00000400 sec
    
```

```

===== CHANNEL f1 =====
NUC1       13C
P1         7.50 usec
p2         15.00 usec
PL1        5.00 dB
SFO1       75.4587938 MHz
    
```

```

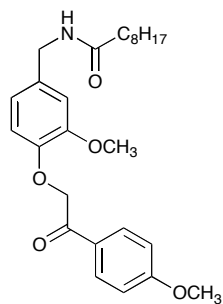
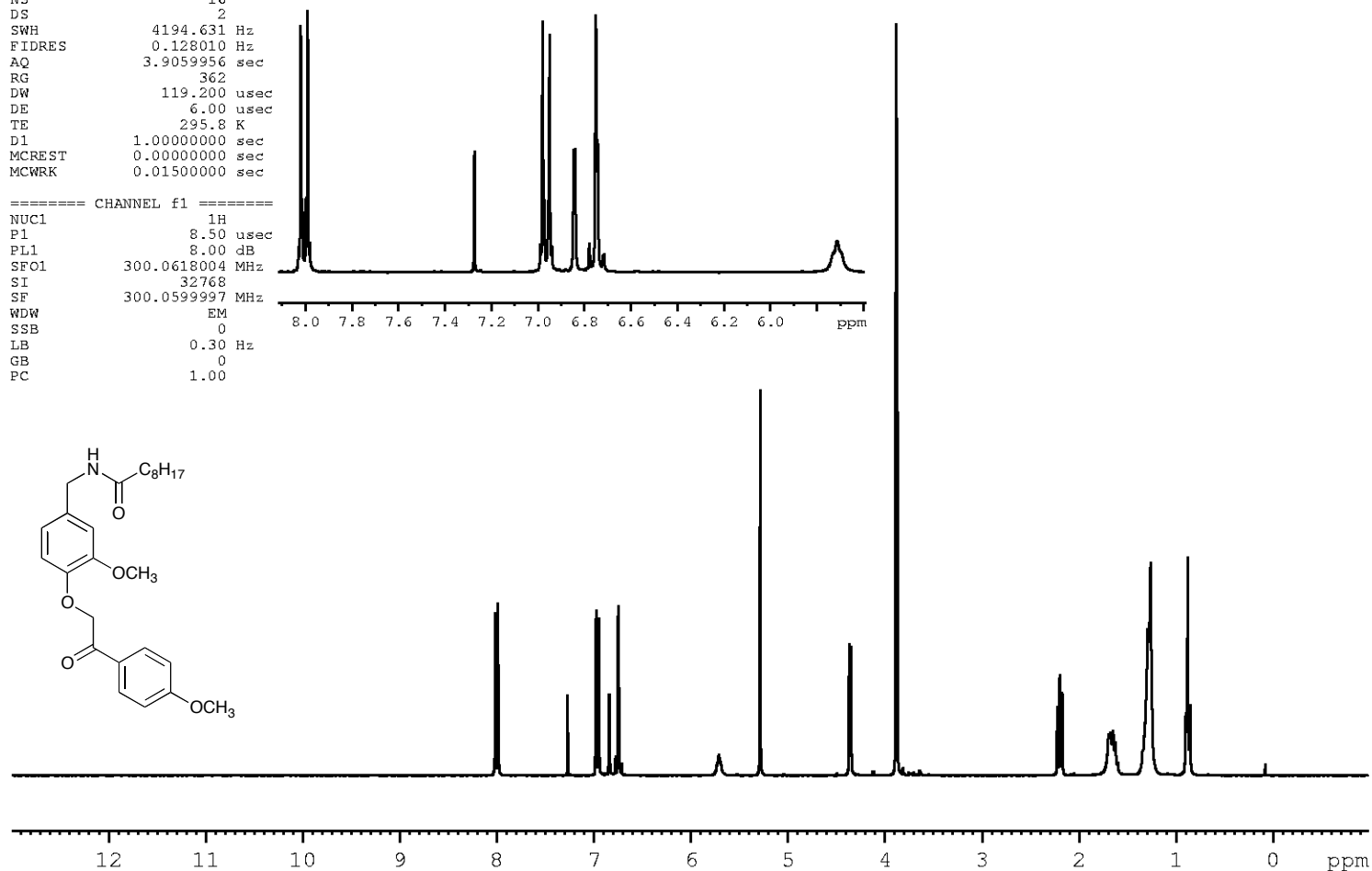
===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
P3         8.50 usec
p4         17.00 usec
PCPD2      80.00 usec
PL2        8.00 dB
PL12       27.47 dB
SFO2       300.0615003 MHz
SI         32768
SF         75.4501170 MHz
WDW        EM
SSB        0
LB         2.00 Hz
GB         0
PC         1.40
    
```



3-Methoxy-4-(4'-methoxyacetophenone-2-oxyl)-N-nonanoylbenzylamine (3) ¹H NMR spectrum

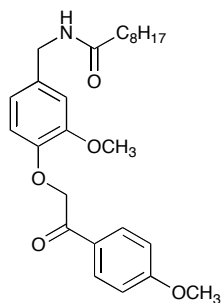
NAME 04152004-6-projects
 EXPNO 10
 PROCNO 1
 Date_ 20040415
 Time 9.55
 INSTRUM av300
 PROBHD 5 mm QNP 1H/1
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 4194.631 Hz
 FIDRES 0.128010 Hz
 AQ 3.9059956 sec
 RG 362
 DW 119.200 usec
 DE 6.00 usec
 TE 295.8 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 8.50 usec
 PL1 8.00 dB
 SFO1 300.0618004 MHz
 SI 32768
 SF 300.0599997 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



3-Methoxy-4-(4'-methoxyacetophenone-2-oxyl)-N-nonanoylbenzylamine (3) ¹³C NMR spectrum

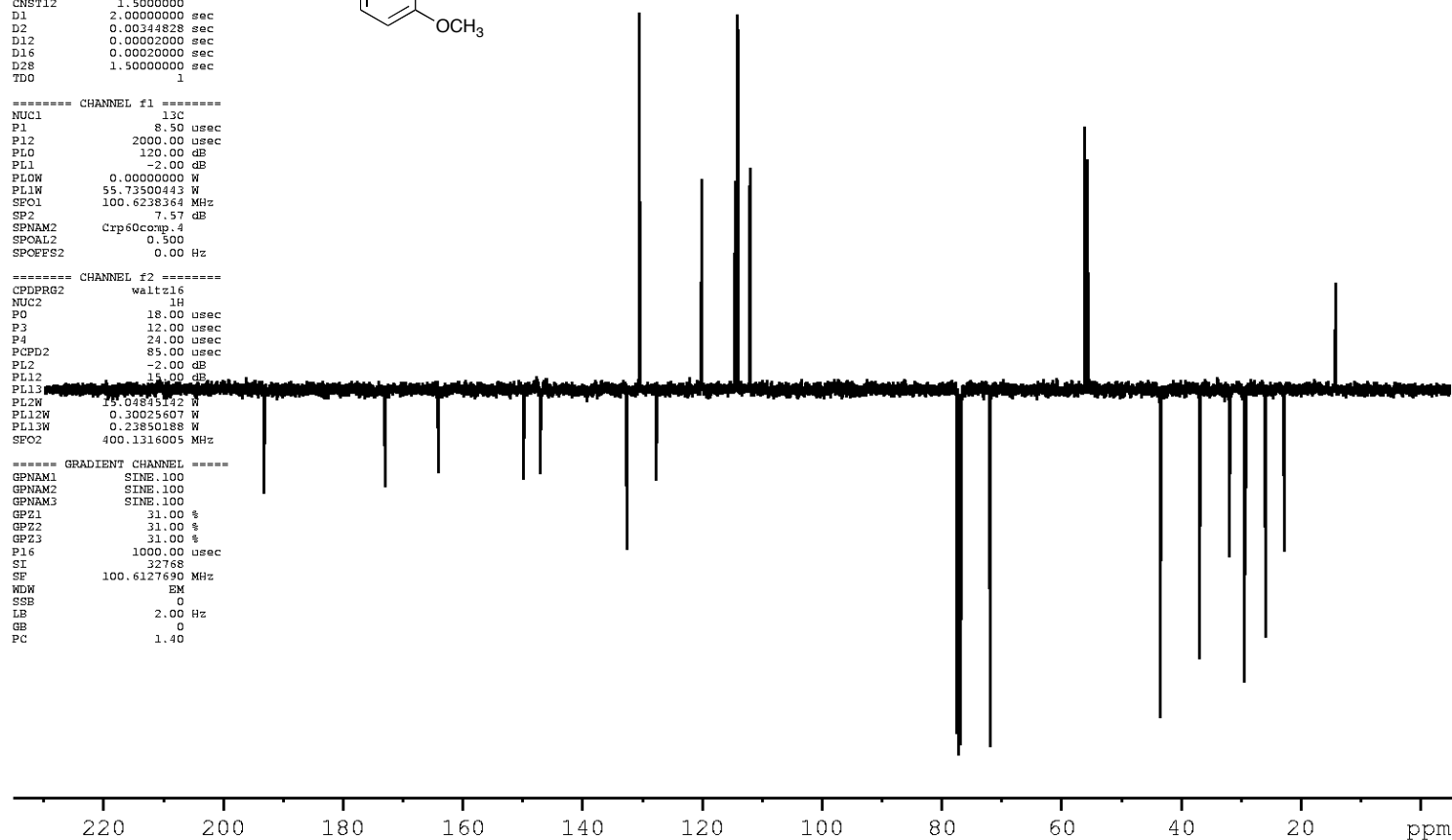
NAME 06112008-16-neganM
 EXPNO 11
 PROCNO 1
 Date_ 20080611
 Time_ 21.03
 INSTRUM AVII400
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 800
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 2050
 DW 20.800 usec
 DE 6.00 usec
 TE 296.4 K
 CNST2 145.0000000
 CNST12 1.5000000
 D1 2.00000000 sec
 D2 0.00344828 sec
 D12 0.00020000 sec
 D16 0.00020000 sec
 D28 1.50000000 sec
 TDO 1



===== CHANNEL f1 =====
 NUC1 13C
 P1 8.50 usec
 P12 2000.00 usec
 PL0 120.00 dB
 PL1 -2.00 dB
 PLOW 0.00000000 W
 PL1W 55.73500443 W
 SFO1 100.6238364 MHz
 SP2 7.57 dB
 SPMAM2 Crp60comp.4
 SPOAL2 0.500
 SPOEFS2 0.00 Hz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 P0 18.00 usec
 P3 12.00 usec
 P4 24.00 usec
 PCPD2 85.00 usec
 PL2 -2.00 dB
 PL12 15.00 dB
 PL13 15.00 dB
 PL2W 15.04845142 W
 PL12W 0.30025607 W
 PL13W 0.23850188 W
 SFO2 400.1316005 MHz

===== GRADIENT CHANNEL =====
 GPNAM1 SINE.100
 GPNAM2 SINE.100
 GPNAM3 SINE.100
 GPZ1 31.00 %
 GPZ2 31.00 %
 GPZ3 31.00 %
 P16 1000.00 usec
 SI 32768
 SF 100.6127690 MHz
 WDW EM
 SSB 0
 LB 2.00 Hz
 GB 0
 PC 1.40



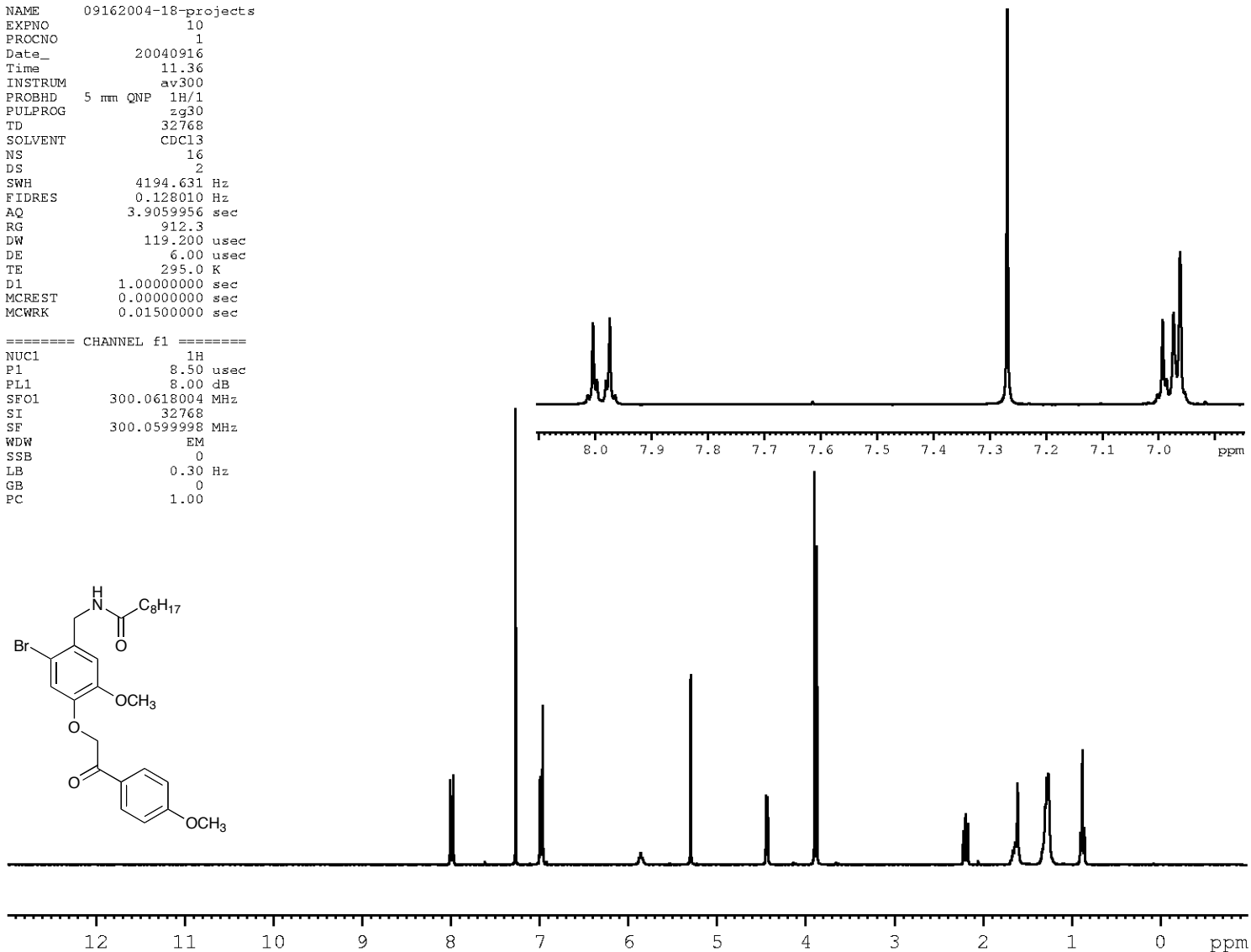
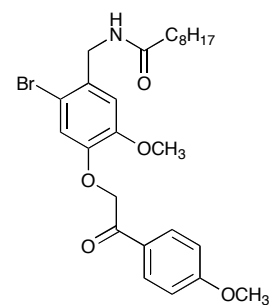
6-Bromo-3-methoxy-4-(4'-methoxyacetophenone-2-oxo)-N-nonanoylbenzylamine (4) ^1H NMR spectrum

```

NAME      09162004-18-projects
EXPNO      10
PROCNO      1
Date_      20040916
Time       11.36
INSTRUM    av300
PROBHD     5 mm QNP 1H/1
PULPROG    zg30
TD         32768
SOLVENT    CDCl3
NS         16
DS         2
SWH        4194.631 Hz
FIDRES     0.128010 Hz
AQ         3.9059956 sec
RG         912.3
DW         119.200 usec
DE         6.00 usec
TE         295.0 K
D1         1.000000000 sec
MCREST     0.00000000 sec
MCWRK      0.01500000 sec
    
```

```

===== CHANNEL f1 =====
NUC1       1H
P1         8.50 usec
PL1        8.00 dB
SFO1       300.0618004 MHz
SI         32768
SF         300.0599998 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
    
```



6-Bromo-3-methoxy-4-(4'-methoxyacetophenone-2-oxy)-N-nonanoylbenzylamine (4) ¹³C NMR spectrum

```

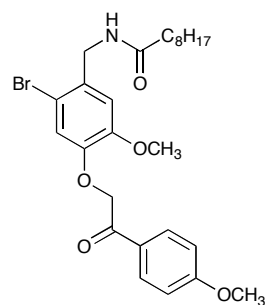
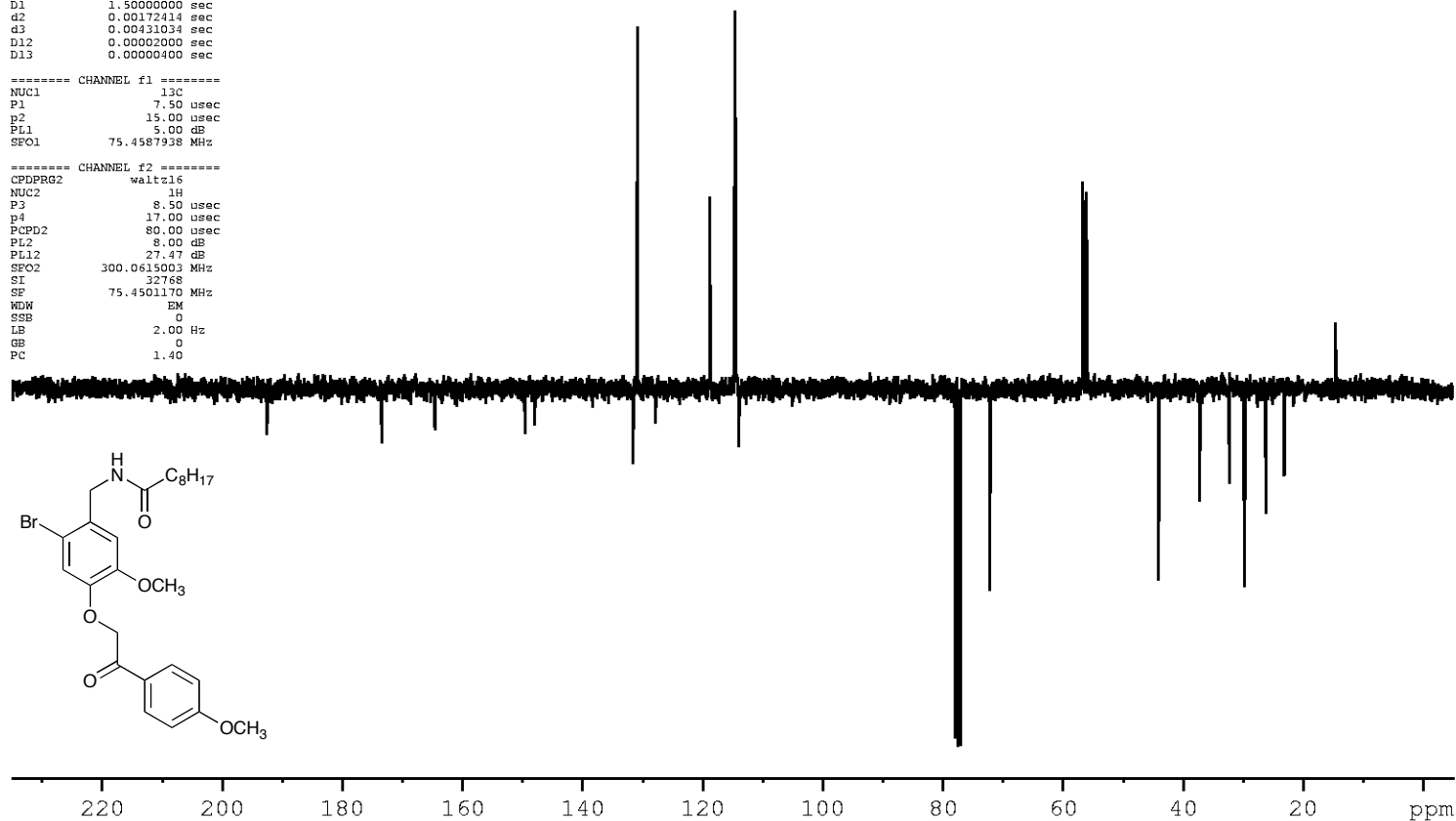
NAME      09202004-29-projects
EXPNO     10
PROCNO     1
Date_     20040920
Time      18.45
INSTRUM    av300
PROBHD     5 mm QNP 1H/1
PULPROG    pendantnew
TD         65536
SOLVENT    CDCl3
NS         800
DS         8
SWH         18115.941 Hz
FIDRES     0.276427 Hz
AQ         1.8088436 sec
RG         18390.4
DW         27.600 usec
DE         6.00 usec
TE         295.4 K
CNST2      145.0000000
CNST3      1.0000000
CNST4      5.0000000
D1         1.5000000 sec
d2         0.00172414 sec
d3         0.00431034 sec
D12        0.00002000 sec
D13        0.00000400 sec
    
```

```

===== CHANNEL f1 =====
NUC1       13C
P1         7.50 usec
P2         15.00 usec
PL1        5.00 dB
SFO1       75.4587938 MHz
    
```

```

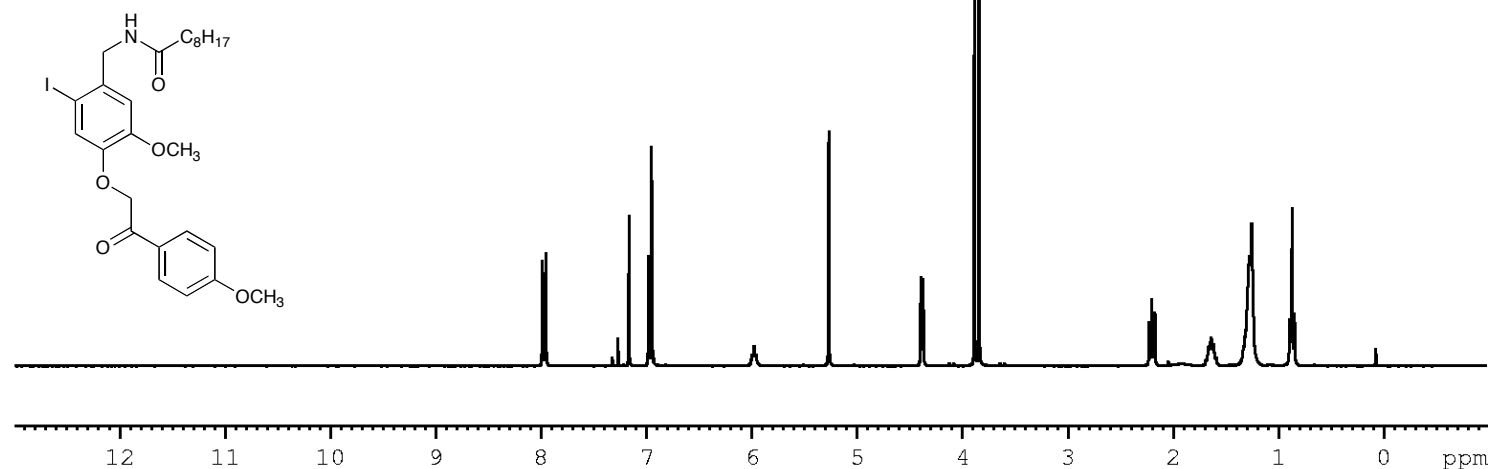
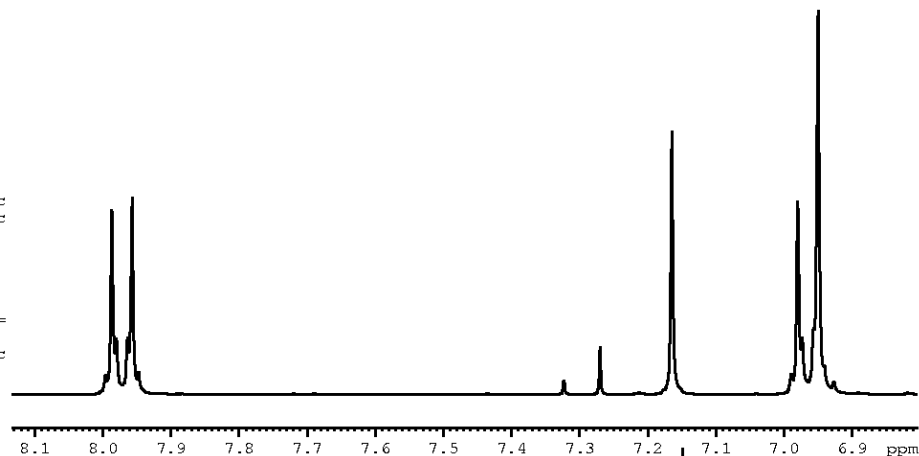
===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
P3         8.50 usec
P4         17.00 usec
PCPD2      80.00 usec
PL2        8.00 dB
PL12       27.47 dB
SFO2       300.0615003 MHz
SI         32768
SF         75.4501170 MHz
WDW        EM
SSB        0
LB         2.00 Hz
GB         0
PC         1.40
    
```



6-Iodo-3-methoxy-4-(4'-methoxyacetophenone-2-oxy)-N-nonanoylbenzylamine (5) ^1H NMR spectrum

NAME 04202004-47-projects
 EXPNO 10
 PROCNO 1
 Date_ 20040421
 Time 0.19
 INSTRUM av300
 PROBHD 5 mm QNP 1H/1
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 4194.631 Hz
 FIDRES 0.128010 Hz
 AQ 3.9059956 sec
 RG 228.1
 DW 119.200 usec
 DE 6.00 usec
 TE 294.8 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 8.50 usec
 PL1 8.00 dB
 SFO1 300.0618004 MHz
 SI 32768
 SF 300.0599995 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



6-Iodo-3-methoxy-4-(4'-methoxyacetophenone-2-oxy)-N-nonanoylbenzylamine (5) ^{13}C NMR spectrum

```

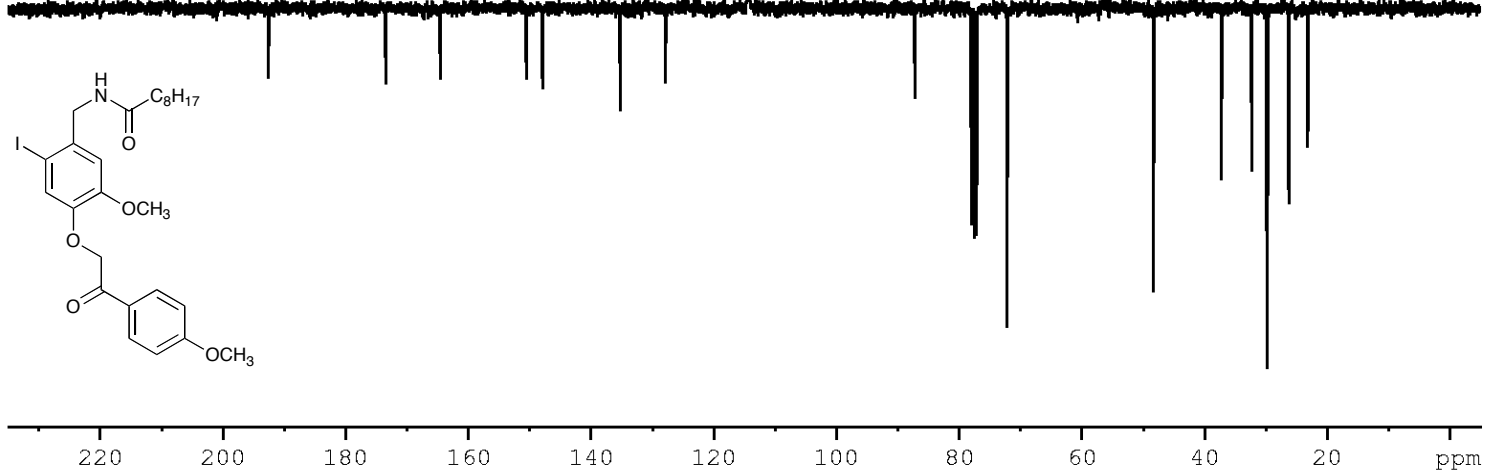
NAME      04202004-47-projects
EXPNO     11
PROCNO    1
Date_     20040421
Time      1.05
INSTRUM   av300
PROBHD    5 mm QNP 1H/1
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         800
DS         8
SWH        18115.941 Hz
FIDRES     0.276427 Hz
AQ         1.8088436 sec
RG         18390.4
DW         27.600 usec
DE         6.00 usec
TE         295.2 K
CNST2     145.0000000
CNST3     1.0000000
CNST4     5.0000000
D1         1.50000000 sec
d2         0.00172414 sec
d3         0.00431034 sec
D12        0.00002000 sec
D13        0.0000400 sec
    
```

```

===== CHANNEL f1 =====
NUC1       13C
P1         7.50 usec
p2         15.00 usec
PL1        5.00 dB
SFO1       75.4587938 MHz
    
```

```

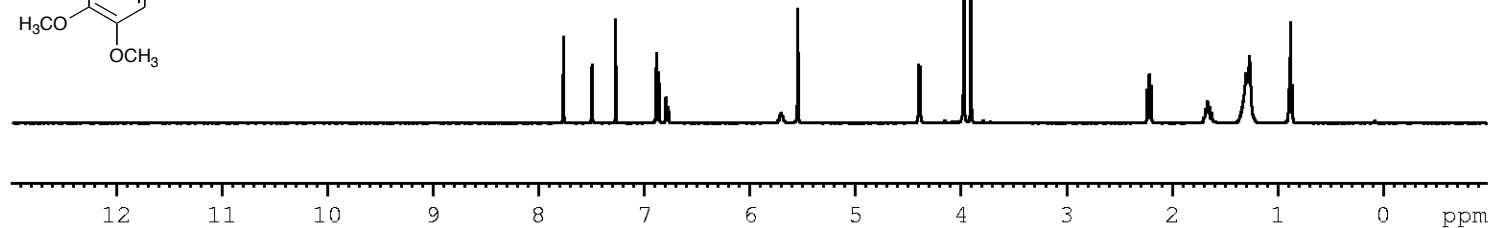
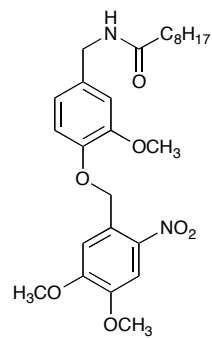
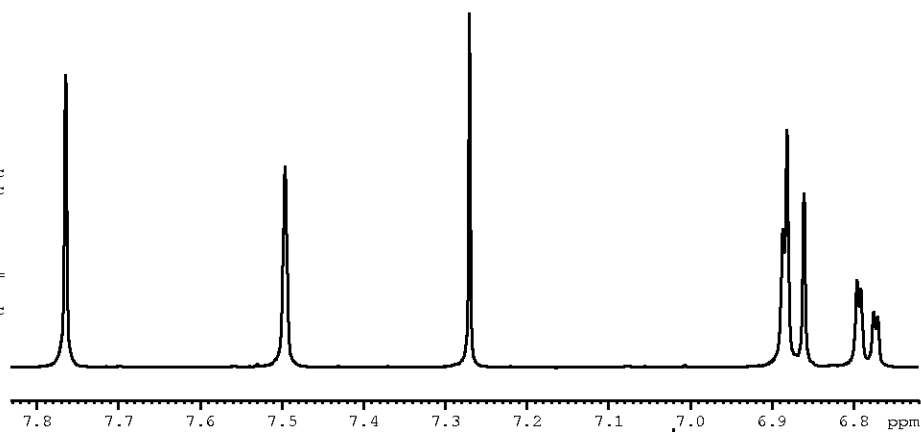
===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
P3         8.50 usec
p4         17.00 usec
PCPD2      80.00 usec
PL2        8.00 dB
PL12       27.47 dB
SFO2       300.0615003 MHz
SI         32768
SF         75.4501170 MHz
WDW        EM
SSB        0
LB         2.00 Hz
GB         0
PC         1.40
    
```



3-Methoxy-4-(4,5-dimethoxy-2-nitrobenzyl)-N-(nonanoyl)benzylamine (6) ^1H NMR spectrum

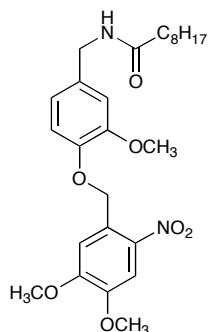
NAME 04292008-22-meganM
 EXPNO 10
 PROCNO 1
 Date_ 20080429
 Time_ 12.31
 INSTRUM AVII400
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 2
 SWH 5597.015 Hz
 FIDRES 0.085404 Hz
 AQ 5.8545995 sec
 RG 228
 DW 89.333 usec
 DE 6.00 usec
 TE 294.5 K
 D1 1.00000000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 12.00 usec
 PL1 -2.00 dB
 PL1W 15.04845142 W
 SFO1 400.1324008 MHz
 SI 32768
 SF 400.1300072 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



3-Methoxy-4-(4,5-dimethoxy-2-nitrobenzyl)-N-(nonanoyl)benzylamine (6) ¹³C NMR spectrum

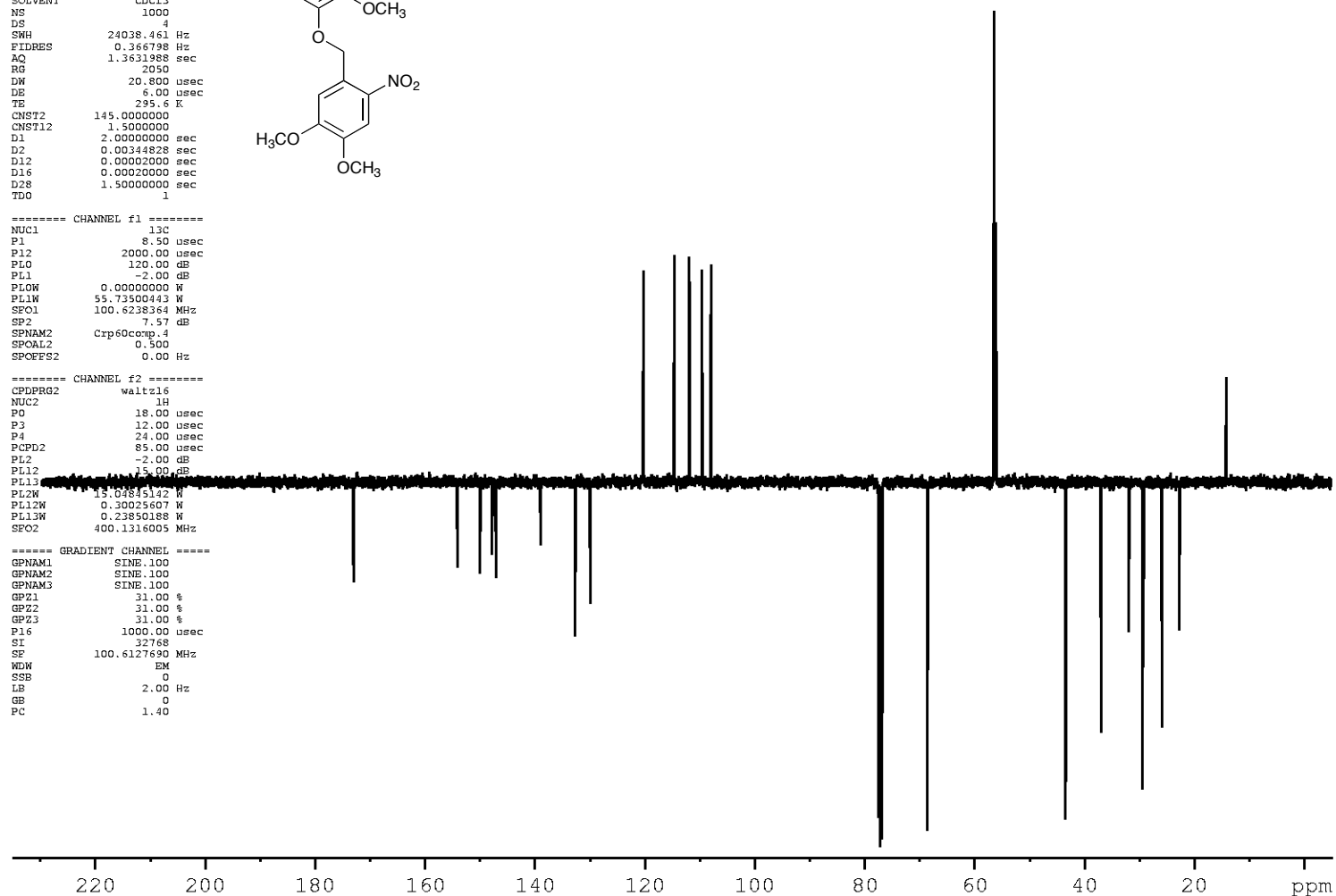
NAME 05012008-9-meganM
EXPNO 10
PROCNO 1
Date_ 20080502
Time_ 0.14
INSTRUM AVII400
PROBHD 5 mm PABBO BB-
PULPROG deptgpgpgp.2
TD 65536
SOLVENT CDCl3
NS 1000
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.00 usec
TE 295.6 K
CNST2 145.0000000
CNST12 1.5000000
D1 2.0000000 sec
D2 0.00344828 sec
D12 0.00002000 sec
D16 0.00020000 sec
D28 1.50000000 sec
TDO 1



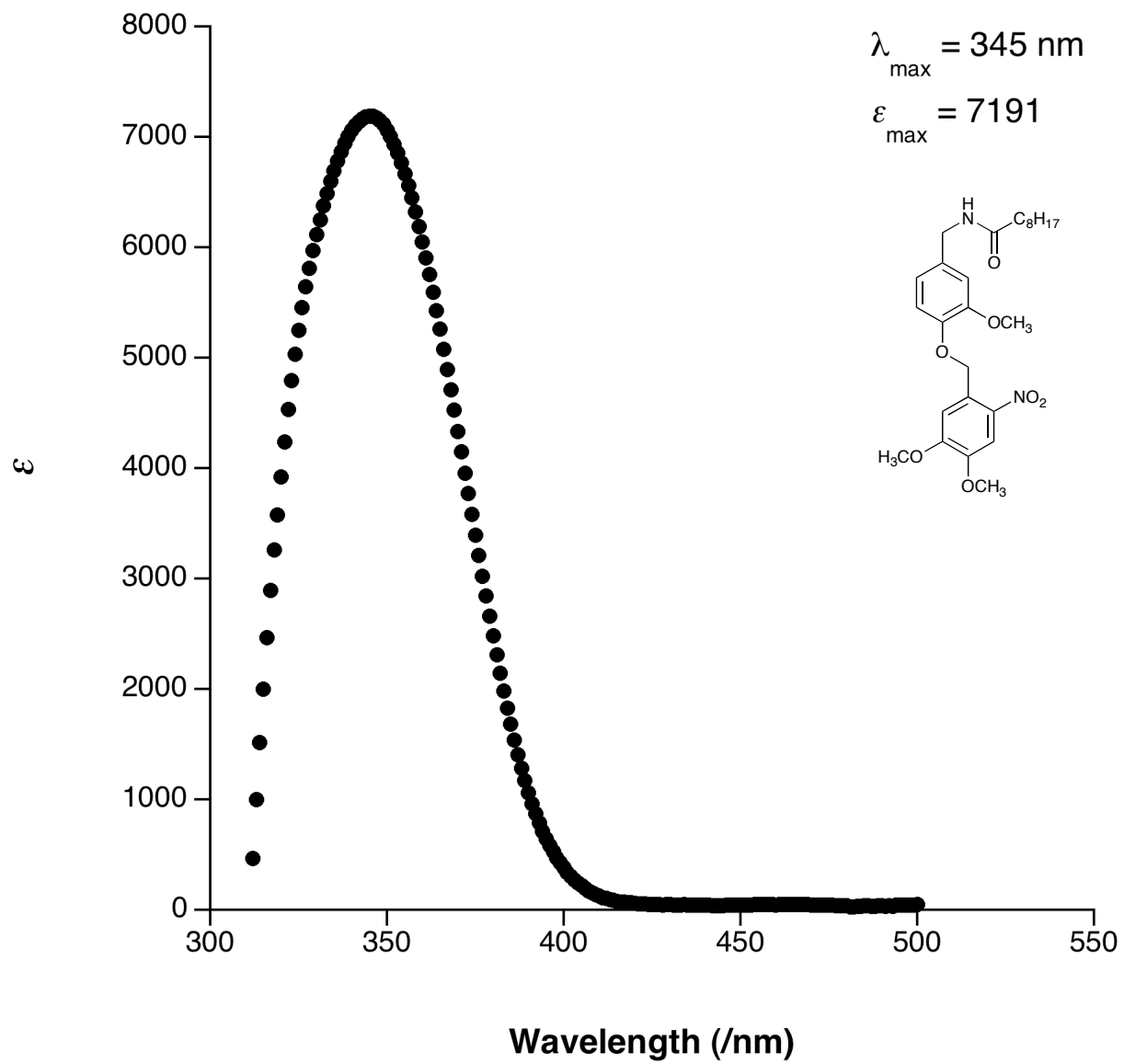
===== CHANNEL f1 =====
NUC1 13C
P1 8.50 usec
P12 2000.00 usec
PLO 120.00 dB
PL1 -2.00 dB
PLOW 0.00000000 W
PL1W 55.73500443 W
SFO1 100.6238364 MHz
SP2 7.57 dB
SPNAM2 Crp60comp.4
SFOAL2 0.500
SPOFFS2 0.00 Hz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
P0 18.00 usec
P3 12.00 usec
P4 24.00 usec
PCPD2 85.00 usec
PL2 -2.00 dB
PL12 15.00 dB
PL13
PL2W 15.04845142 W
PL12W 0.10025607 W
PL13W 0.23850188 W
SFO2 400.1316005 MHz

===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPNAM3 SINE.100
GPZ1 31.00 %
GPZ2 31.00 %
GPZ3 31.00 %
P16 1000.00 usec
ST 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40



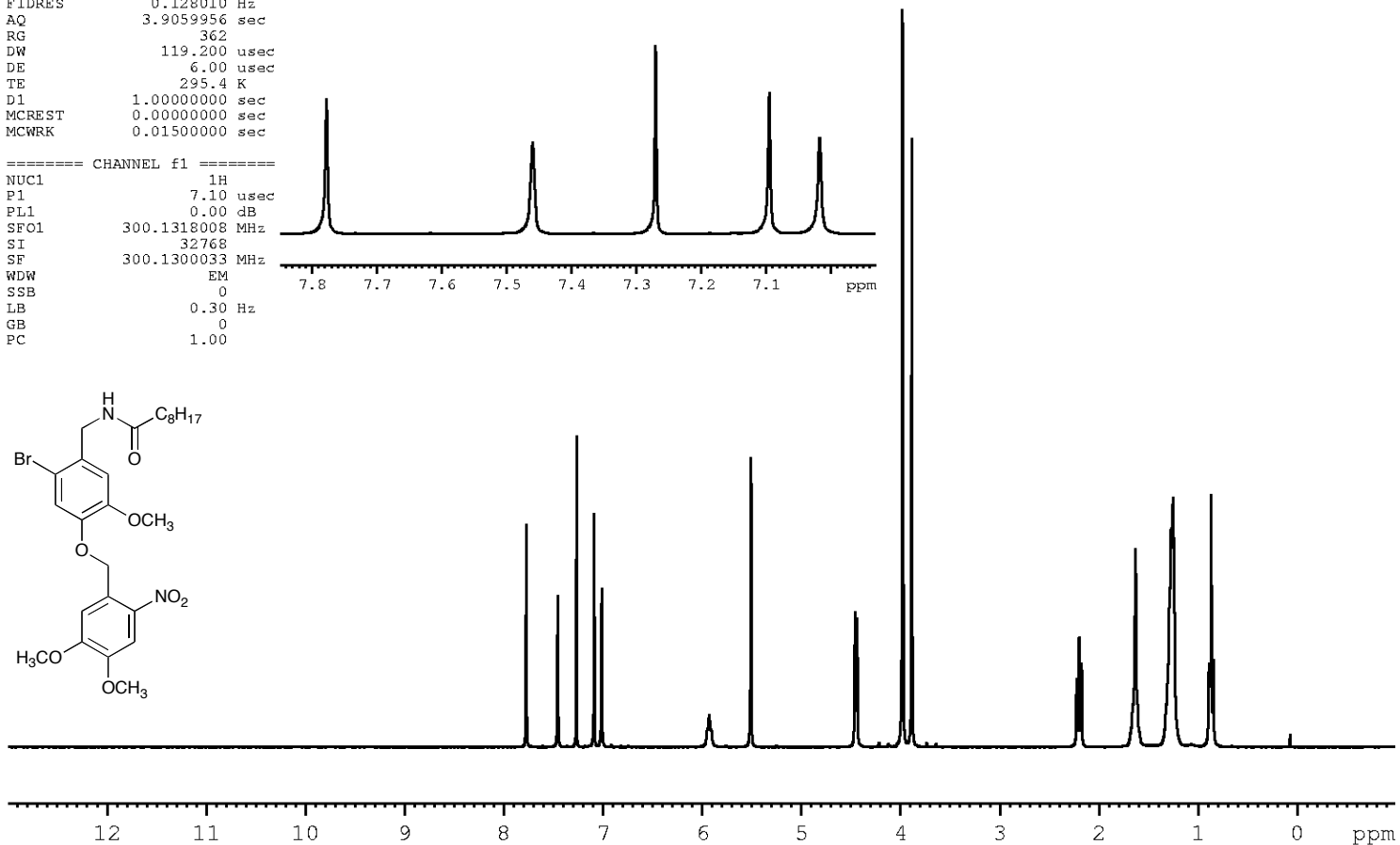
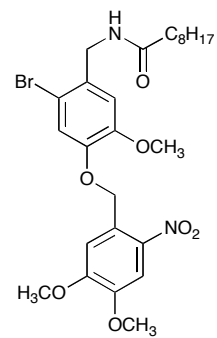
3-Methoxy-4-(4,5-dimethoxy-2-nitrobenzyl)-N-(nonanoyl)benzylamine (6) UV/Vis spectrum



6-Bromo-3-methoxy-4-(4,5-dimethoxy-2-nitrobenzyl)-N-(nonanoyl)benzylamine (7) ^1H NMR spectrum

NAME 01152006-27-michael
 EXPNO 10
 PROCNO 1
 Date_ 20060115
 Time_ 15.13
 INSTRUM av300
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 4194.631 Hz
 FIDRES 0.128010 Hz
 AQ 3.9059956 sec
 RG 362
 DW 119.200 usec
 DE 6.00 usec
 TE 295.4 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 7.10 usec
 PL1 0.00 dB
 SFO1 300.1318008 MHz
 SI 32768
 SF 300.1300033 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



6-Bromo-3-methoxy-4-(4,5-dimethoxy-2-nitrobenzyl)-N-(nonanoyl)benzylamine (7) ^{13}C NMR spectrum

```

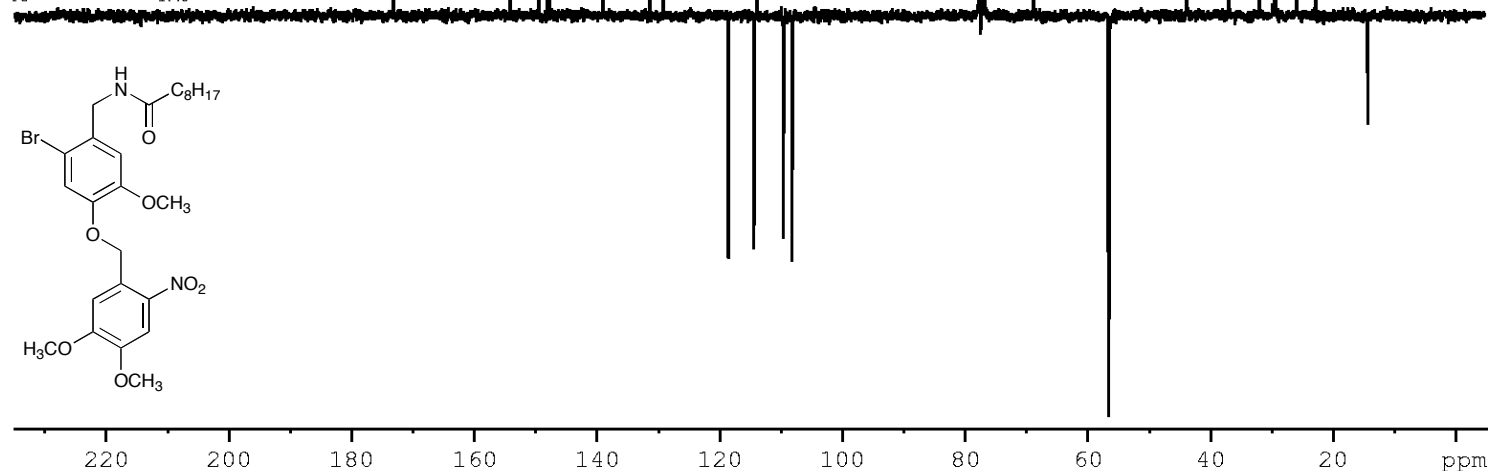
NAME      01152006-27-michael
EXPNO     11
PROCNO    1
Date_     20060115
Time      20.47
INSTRUM   av300
PROBHD    5 mm BBO BB-1H
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         6000
DS         8
SWH        18115.941 Hz
FIDRES     0.276427 Hz
AQ         1.8088436 sec
RG         18390.4
DW         27.600 usec
DE         6.00 usec
TE         296.1 K
CNST2     145.0000000
CNST3     1.0000000
CNST4     5.0000000
D1         1.50000000 sec
d2         0.00172414 sec
d3         0.00431034 sec
D12        0.00002000 sec
D13        0.00000400 sec
    
```

```

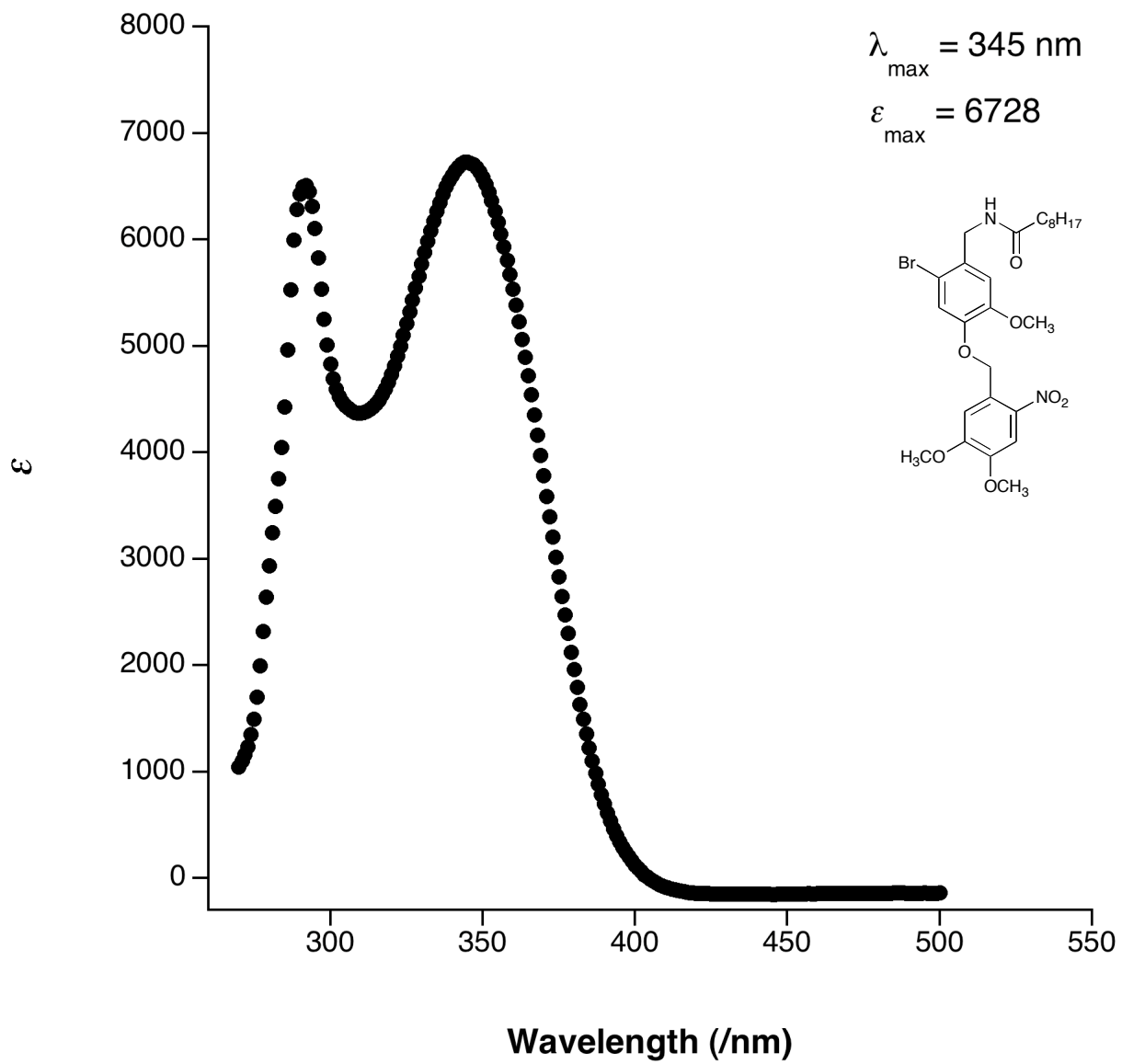
===== CHANNEL f1 =====
NUC1       13C
P1         7.40 usec
p2         14.80 usec
PL1        -3.00 dB
SFO1       75.4764273 MHz
    
```

```

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2        1H
P3          6.80 usec
p4          13.60 usec
PCPD2       80.00 usec
PL2         0.00 dB
PL12        22.00 dB
SFO2       300.1315007 MHz
SI          32768
SF         75.4677490 MHz
WDW         EM
SSB         0
LB          2.00 Hz
GB          0
PC          1.40
    
```



6-Bromo-3-methoxy-4-(4,5-dimethoxy-2-nitrobenzyl)-N-(nonanoyl)benzylamine (7) UV/Vis spectrum



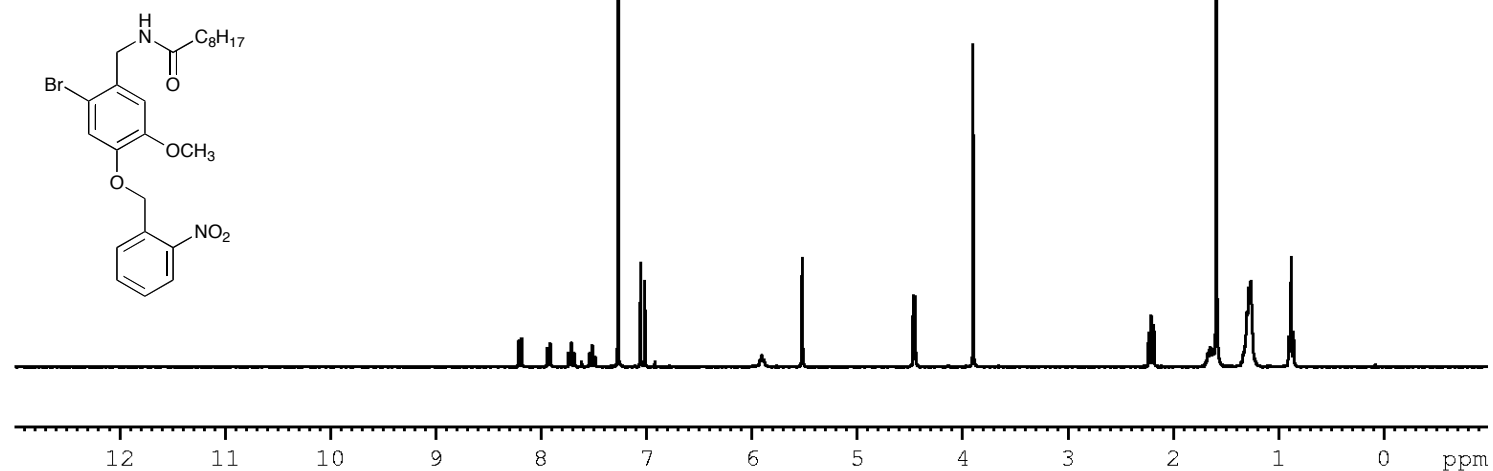
6-Bromo-3-methoxy-4-(2-nitrobenzyl)-N-(nonanoyl)benzylamine (8) ^1H NMR spectrum

```

NAME      01122006-4-michael
EXPNO     10
PROCNO     1
Date_     20060112
Time      17.39
INSTRUM    av300
PROBHD     5 mm QNP 1H/1
PULPROG    zg30
TD         32768
SOLVENT    CDCl3
NS         16
DS         2
SWH        4194.631 Hz
FIDRES     0.128010 Hz
AQ         3.9059956 sec
RG         1024
DW         119.200 usec
DE         6.00 usec
TE         294.4 K
D1         1.00000000 sec
MCREST     0.00000000 sec
MCWRK      0.01500000 sec
    
```

```

===== CHANNEL f1 =====
NUC1       1H
P1         8.50 usec
PL1        8.00 dB
SFO1       300.0618004 MHz
SI         32768
SF         300.0599993 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
    
```



6-Bromo-3-methoxy-4-(2-nitrobenzyl)-N-(nonanoyl)benzylamine (8) ^{13}C NMR spectrum

```

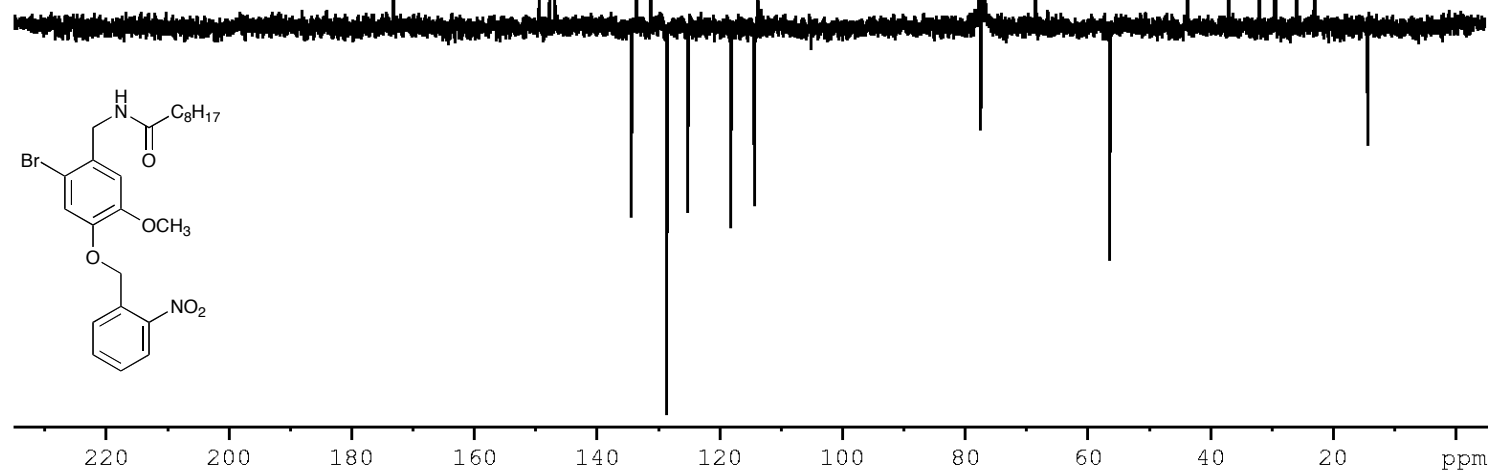
NAME      09252005-17-michael
EXPNO     10
PROCNO    1
Date_     20050925
Time      23.56
INSTRUM   av300
PROBHD    5 mm BBO BB-1H
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         2400
DS         8
SWH        18115.941 Hz
FIDRES     0.276427 Hz
AQ         1.8088436 sec
RG         18390.4
DW         27.600 usec
DE         6.00 usec
TE         294.8 K
CNST2     145.0000000
CNST3     1.0000000
CNST4     5.0000000
D1         5.00000000 sec
d2         0.00172414 sec
d3         0.00431034 sec
D12        0.00002000 sec
D13        0.00000400 sec
    
```

```

===== CHANNEL f1 =====
NUC1       13C
P1         7.40 usec
p2         14.80 usec
PL1        -3.00 dB
SFO1       75.4764273 MHz
    
```

```

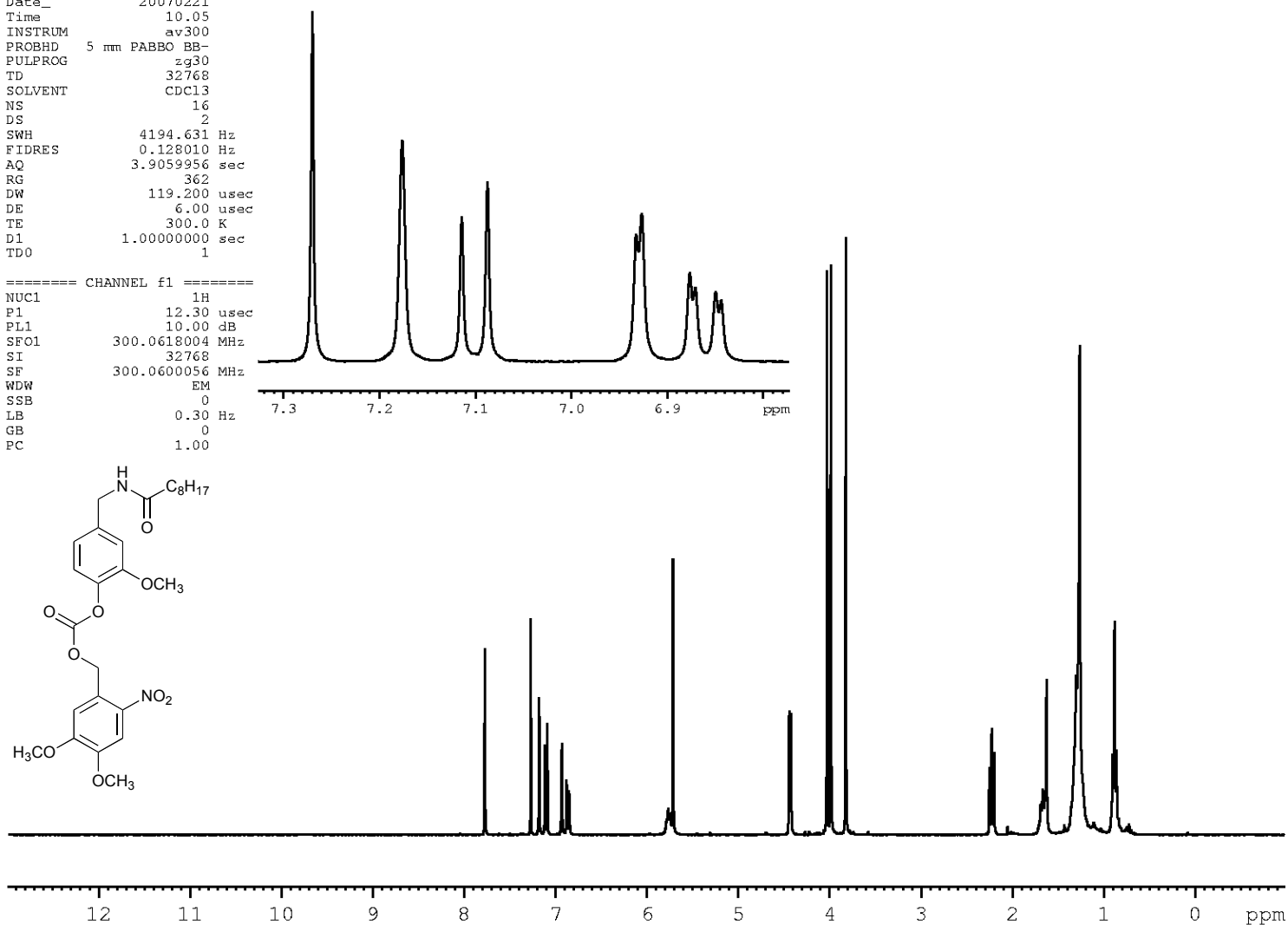
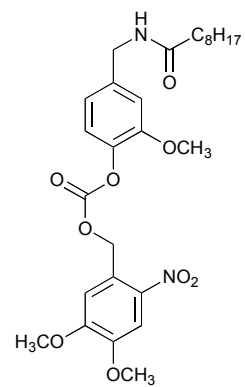
===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
P3         6.80 usec
p4         13.60 usec
PCPD2      80.00 usec
PL2        0.00 dB
PL12       22.00 dB
SFO2       300.1315007 MHz
SI         32768
SF         75.4677490 MHz
WDW        EM
SSB        0
LB         2.00 Hz
GB         0
PC         1.40
    
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4,5-Dimethoxy-2-nitrobenzyl 2'-methoxy-4'-(nonanamidomethyl)phenyl carbonate (9) ^1H NMR spectrum

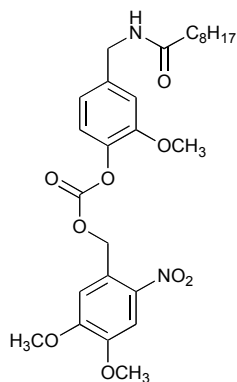
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 PROCNO 1
 Date_ 20070221
 Time_ 10.05
 INSTRUM av300
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 4194.631 Hz
 FIDRES 0.128010 Hz
 AQ 3.9059956 sec
 RG 362
 DW 119.200 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 12.30 usec
 PL1 10.00 dB
 SFO1 300.0618004 MHz
 SI 32768
 SF 300.0600056 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



4,5-Dimethoxy-2-nitrobenzyl 2'-methoxy-4'-(nonanamidomethyl)phenyl carbonate (9) ¹³C NMR spectrum

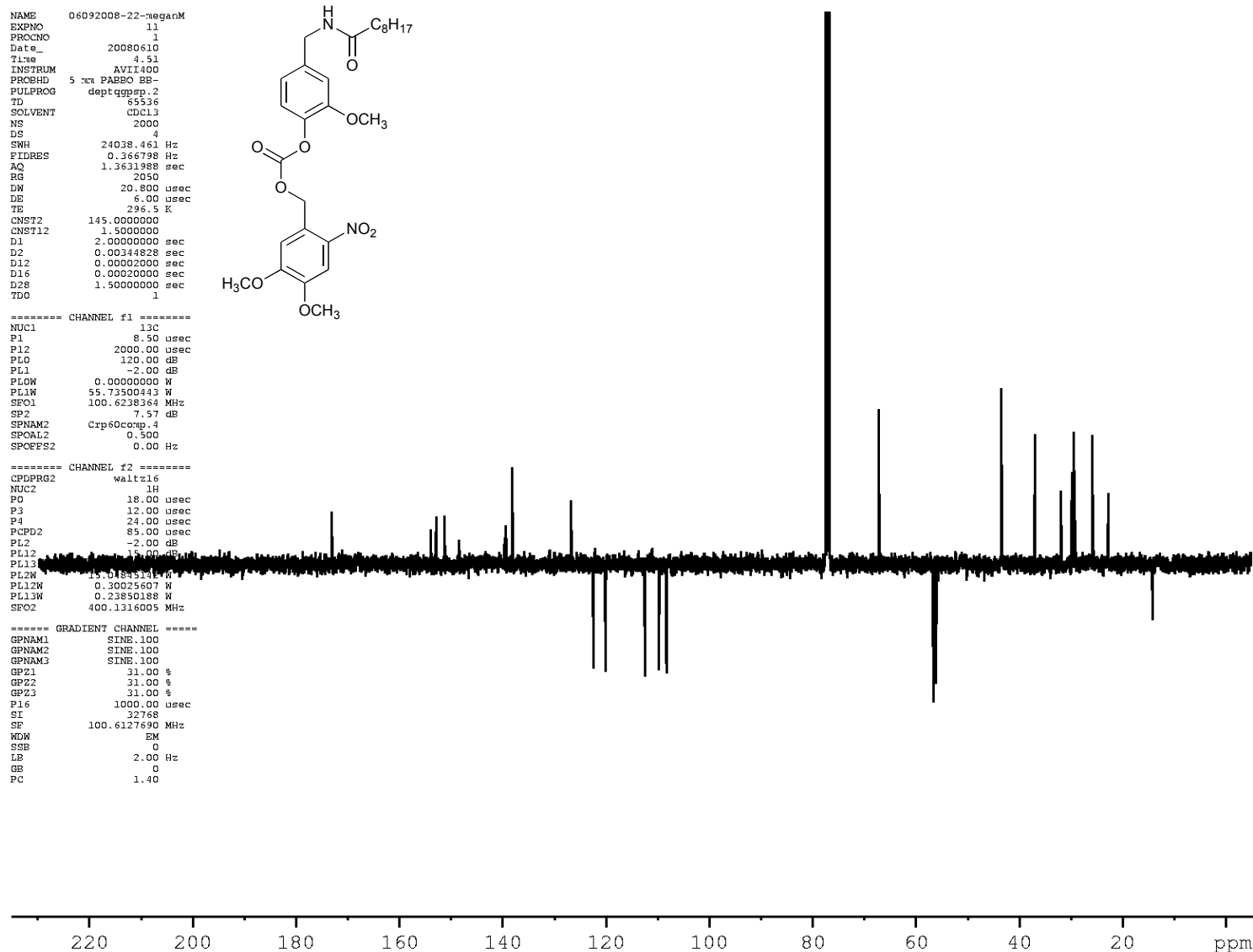
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EXPNO 11
PROCNO 1
Date_ 20080610
Time 4.51
INSTRUM AVII400
PROBHD 5 mm PABBO BB-
PULPROG deptgpgpgp.2
TD 65536
SOLVENT CDCl3
NS 2000
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.00 usec
TE 296.5 K
CNST2 145.0000000
CNST12 1.5000000
D1 2.0000000 sec
D2 0.00344828 sec
D12 0.00002000 sec
D16 0.00020000 sec
D28 1.50000000 sec
TDO 1



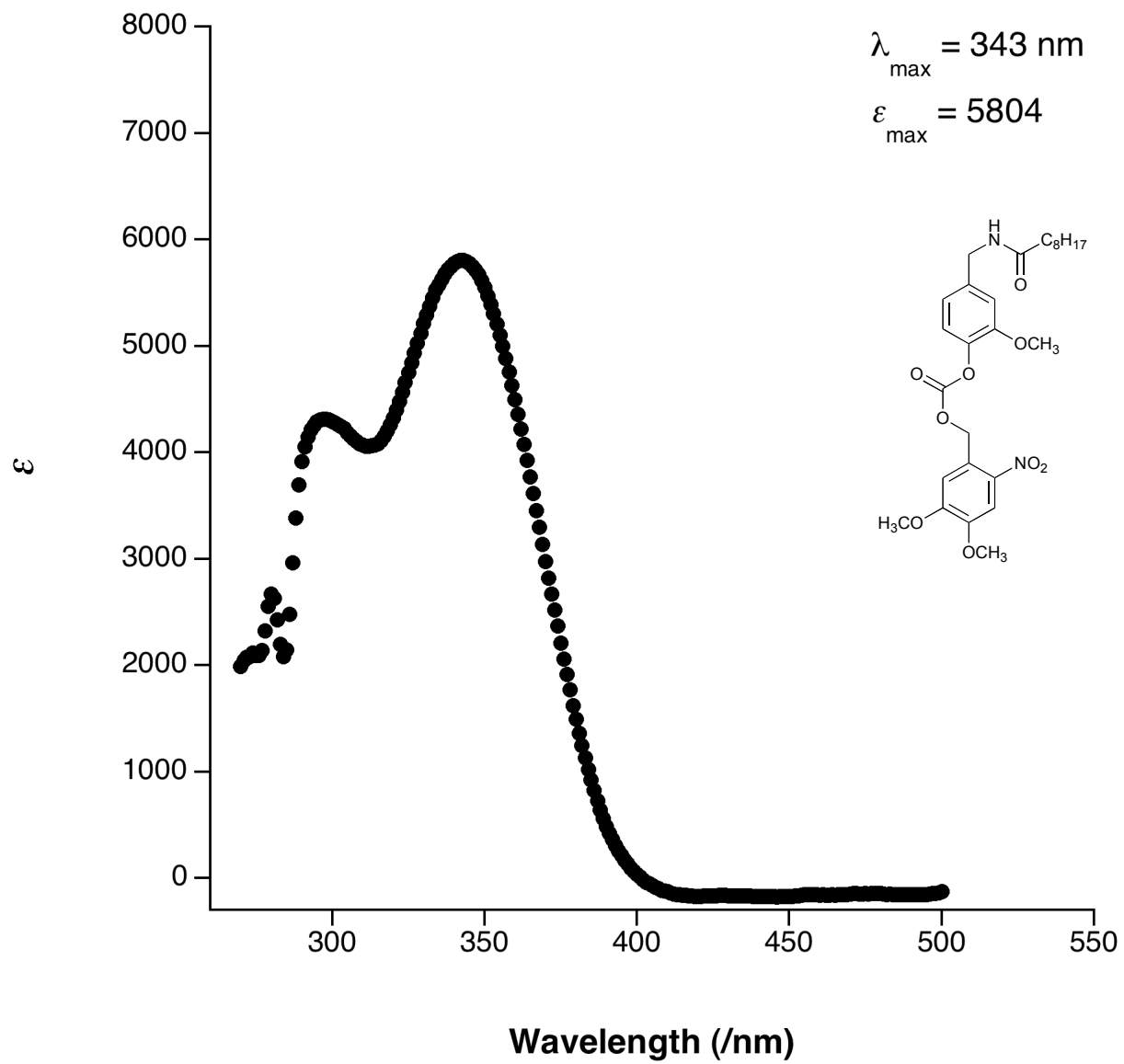
===== CHANNEL f1 =====
NUC1 13C
P1 8.50 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -2.00 dB
PL0W 0.00000000 W
PL1W 55.73500443 W
SFO1 100.6238364 MHz
SP2 7.57 dB
SPNAM2 Crp60comp.4
SFOAL2 0.500
SFOFFS2 0.00 Hz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
P0 18.00 usec
P3 12.00 usec
P4 24.00 usec
PCPD2 85.00 usec
PL2 -2.00 dB
PL12 15.00 dB
PL13 15.00 dB
PL2W 15.04851414 W
PL12W 0.30025607 W
PL13W 0.23850188 W
SFO2 400.1316005 MHz

===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPNAM3 SINE.100
GPZ1 31.00 %
GPZ2 31.00 %
GPZ3 31.00 %
P16 1000.00 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40



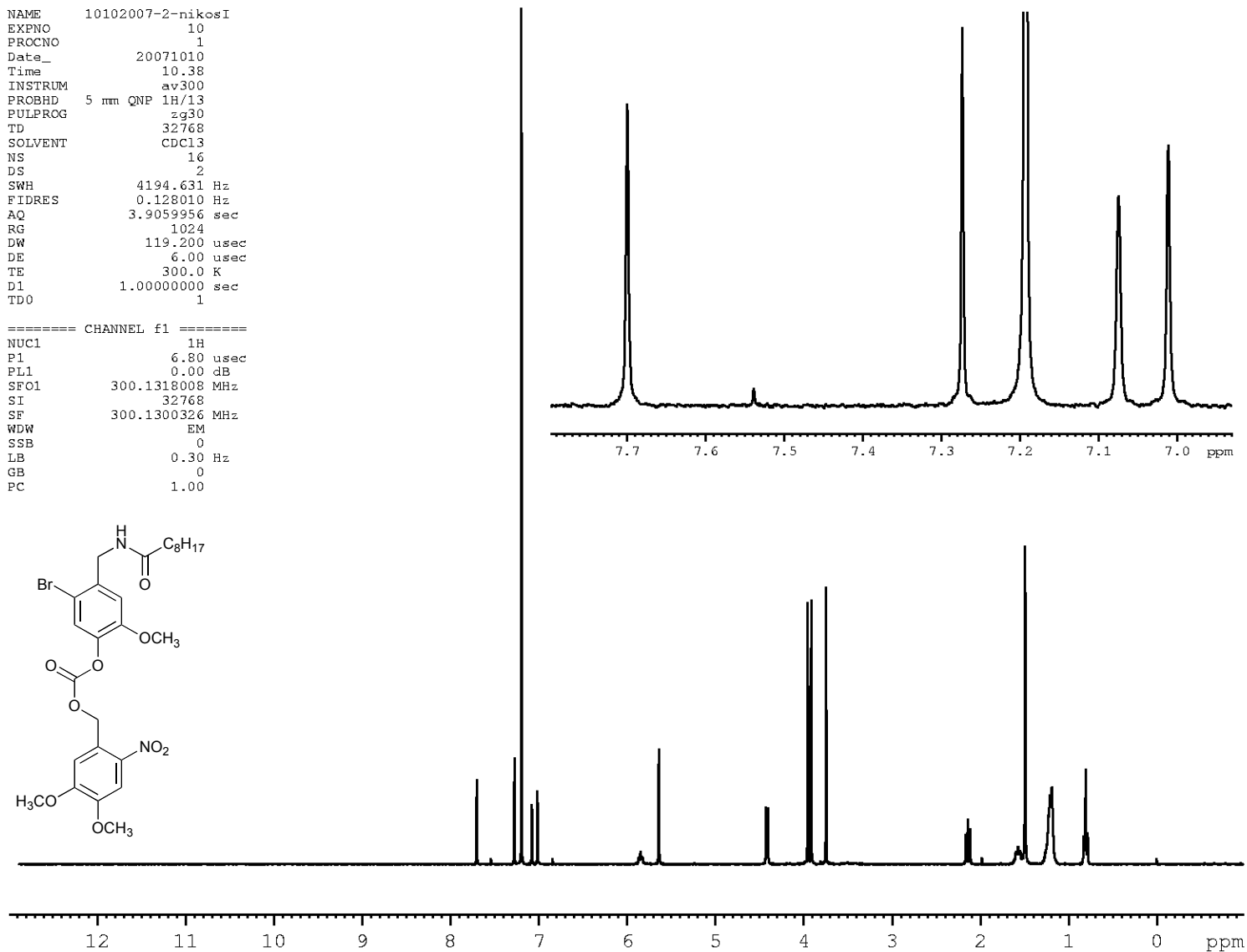
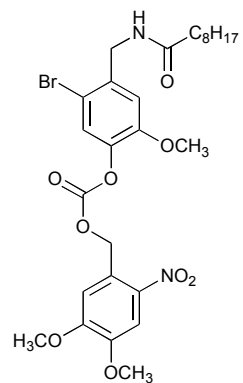
4,5-Dimethoxy-2-nitrobenzyl 2'-methoxy-4'-(nonanamidomethyl)phenyl carbonate (9) UV/Vis spectrum



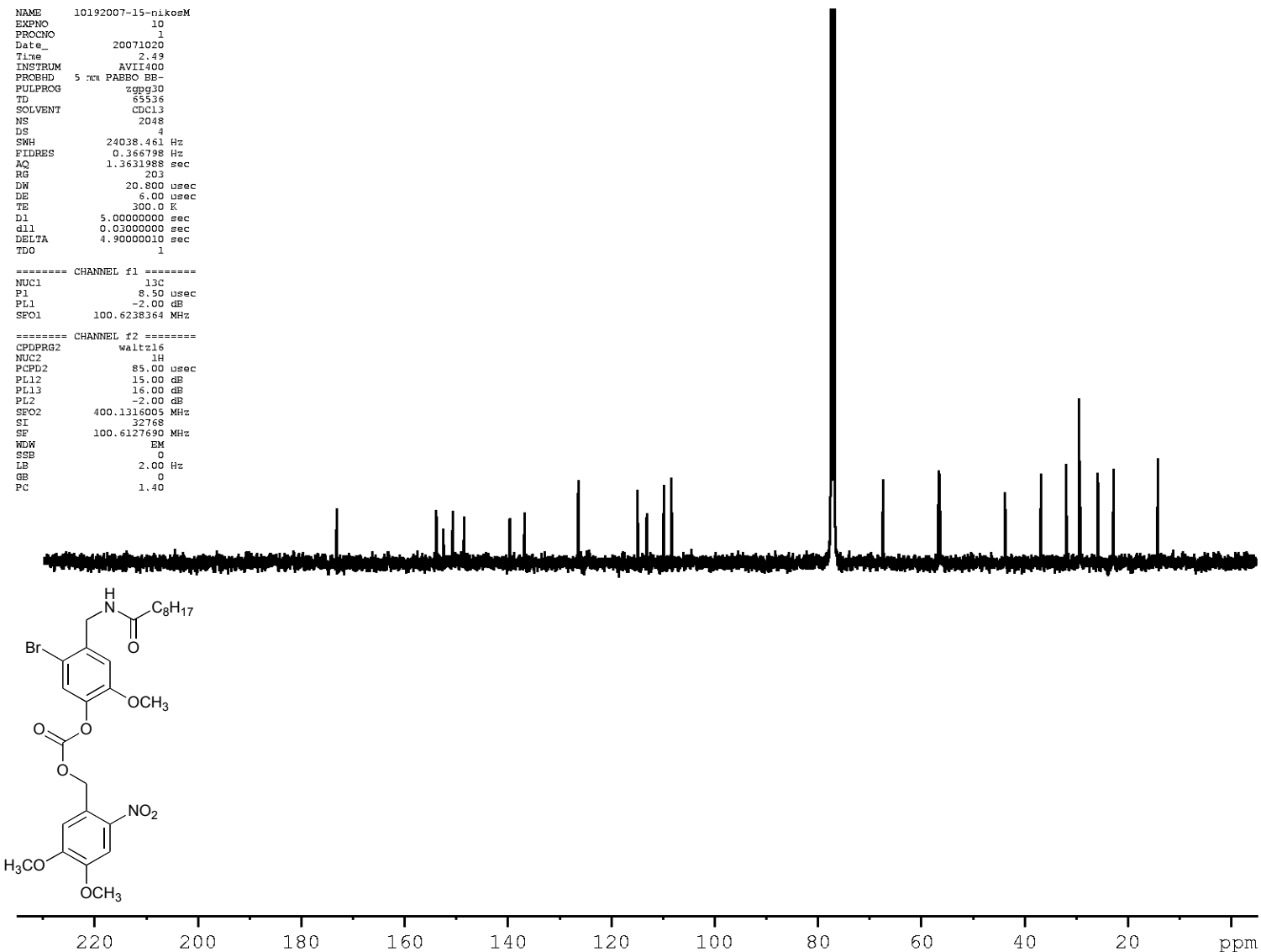
4,5-Dimethoxy-2-nitrobenzyl 5'-bromo-2'-methoxy-4'-(nonanamidomethyl)phenyl carbonate (10) ^1H NMR spectrum

NAME 10102007-2-nikosI
 EXPNO 10
 PROCNO 1
 Date_ 20071010
 Time_ 10.38
 INSTRUM av300
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 4194.631 Hz
 FIDRES 0.128010 Hz
 AQ 3.9059956 sec
 RG 1024
 DW 119.200 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 TDO 1

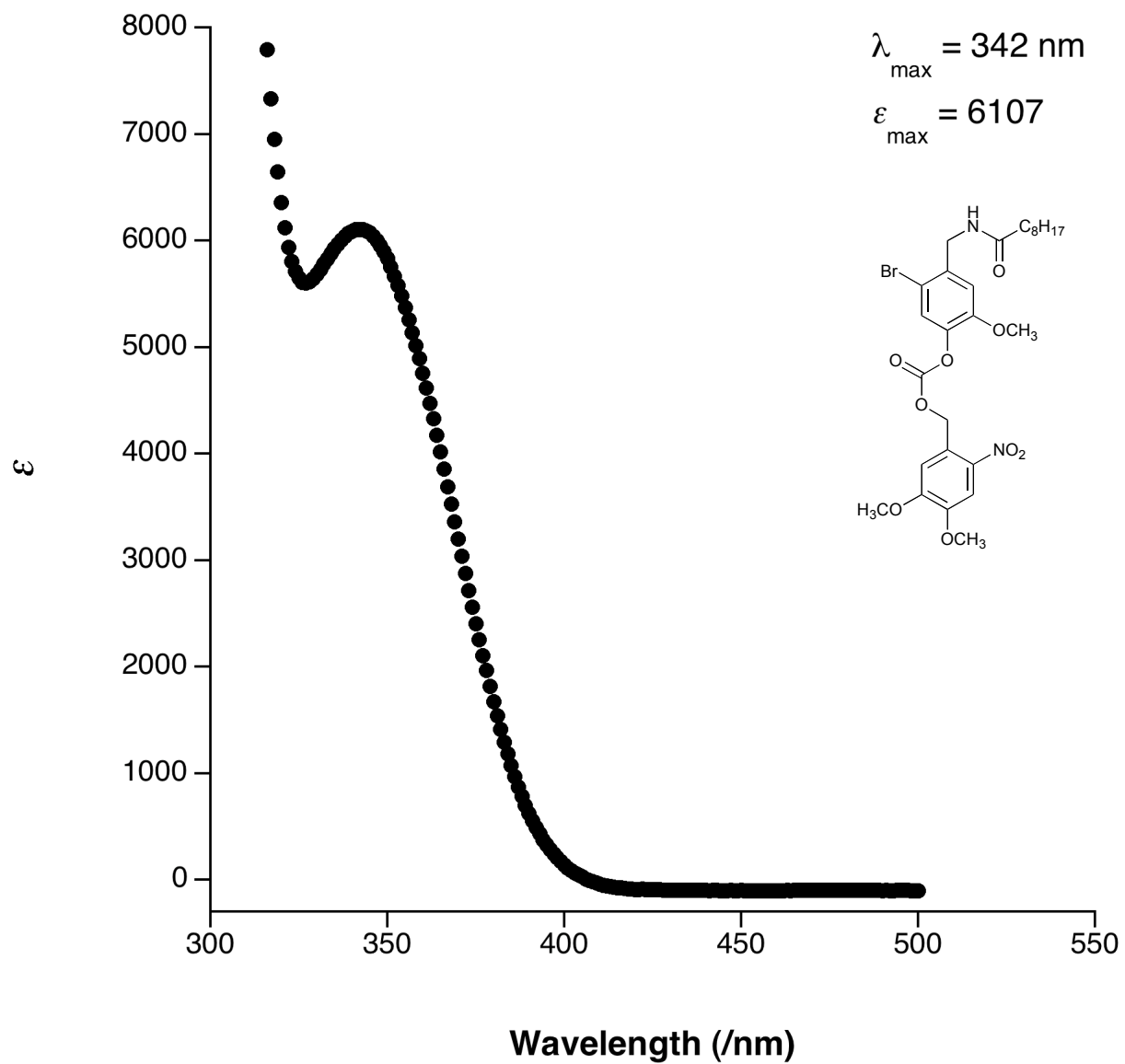
===== CHANNEL f1 =====
 NUC1 1H
 P1 6.80 usec
 PL1 0.00 dB
 SFO1 300.1318008 MHz
 SI 32768
 SF 300.1300326 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



4,5-Dimethoxy-2-nitrobenzyl 5'-bromo-2'-methoxy-4'-(nonanamidomethyl)phenyl carbonate (10) ¹³C NMR spectrum



4,5-Dimethoxy-2-nitrobenzyl 5'-bromo-2'-methoxy-4'-(nonanamidomethyl)phenyl carbonate (10) UV/Vis spectrum



References

1. W. L. F. Armarego and C. L. L. Chai, *Purification of laboratory chemicals*, Butterworth Heinemann, 2003.