

Supporting Information

Cyclofenil Derivatives as Estrogen Receptor Ligands: Syntheses, Biological Evaluation, and SAR Study.

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1. Elemental analysis data for evaluated compounds

Compd.	Formula	Calculated		Found	
		C	H	C	H
2	C ₁₇ H ₁₆ O ₂ ·0.1 H ₂ O	80.35	6.43	80.51	6.48
3	C ₁₈ H ₁₈ O ₂	81.17	6.81	81.25	6.88
4	C ₁₉ H ₂₀ O ₂	81.40	7.19	81.43	7.23
5	C ₂₀ H ₂₂ O ₂	81.60	7.53	81.54	7.61
18	C ₁₉ H ₂₀ O ₃ ·0.2 H ₂ O	76.54	6.83	76.49	6.89
20	C ₁₉ H ₁₉ O ₂ F·0.1 H ₂ O	76.03	6.45	75.88	6.56
21a	C ₂₂ H ₂₄ O ₄ ·0.2 H ₂ O	74.22	6.91	74.26	6.86
21b	C ₂₂ H ₂₄ O ₄	74.98	6.86	79.87	7.02
24a	C ₂₀ H ₂₂ O ₃	77.39	7.14	77.21	7.15
24b	C ₂₁ H ₂₄ O ₃	77.75	7.46	77.73	7.47
26a	C ₂₀ H ₂₁ O ₂ F	76.90	6.78	76.73	6.82
26b	C ₂₁ H ₂₃ O ₂ F·0.1 H ₂ O	76.85	7.12,	76.61	7.19
27	C ₂₂ H ₂₆ O ₃	78.07	7.74	77.82	7.82.
28	C ₂₁ H ₂₄ O ₃ ·0.1 H ₂ O	74.67	7.87	74.63	7.61
29a	C ₂₀ H ₂₂ O ₂ ·0.1 H ₂ O	81.10	7.55	81.16	7.77
29b	C ₂₁ H ₂₄ O ₂	81.78	7.84	81.38	7.85
30a	C ₂₁ H ₂₂ O ₄	74.54	6.55	74.28	6.90
30b	C ₂₄ H ₂₆ O ₆	70.23	6.38	69.97	6.26
32	C ₂₂ H ₂₄ O ₄	74.98	6.86	74.68	6.84
35a	C ₂₀ H ₂₂ O ₃	77.39	7.14	77.57	7.42
35b	C ₂₁ H ₂₄ O ₃	77.75	7.46	77.43	7.69
35c	C ₂₂ H ₂₆ O ₃	78.07	7.74	78.15	7.96

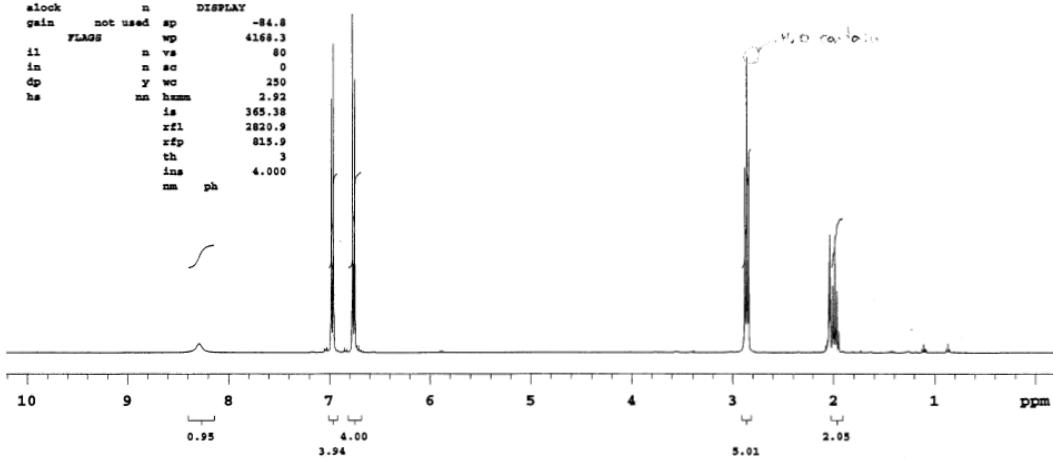
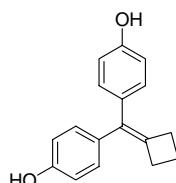
2. ^1H and ^{13}C NMR spectra

Bis(4-hydroxyphenyl)methylenecyclobutane (2). ^1H NMR (400 MHz, acetone- d_6)

Bis(4-hydroxyphenyl)methylenecyclobutane (2)

exp1 std1h

```
SAMPLE          DEC. & VT
date  Feb 24 2004 dn      M1
solvent Acetone dof      0
file   exp dm      nnn
ACQUISITION dm      a
sfrq  399.953 dmf     200
tn    M1 dprw    20
at    4.096 PROCESSING
np    65536 lb      0.30
sw    8000.0 wfile
fb    4400 proc     ft
bs    2 fn      not used
tpwr  63 math     f
pw    9.6
d1    0 werr
tof   -425.7 wexp
nt    32 wbs
ct    32 wnt
clock  n DISPLAY
gain  not used sp      -84.8
      wp      4168.3
FLAGS
il    n vs      80
in    n sc      0
dp    y wc      250
hs    nn hzmm   2.92
      is      365.38
      r1f1    2820.9
      r1f2    815.9
      th      3
      ins     4.000
      nm     ph
```

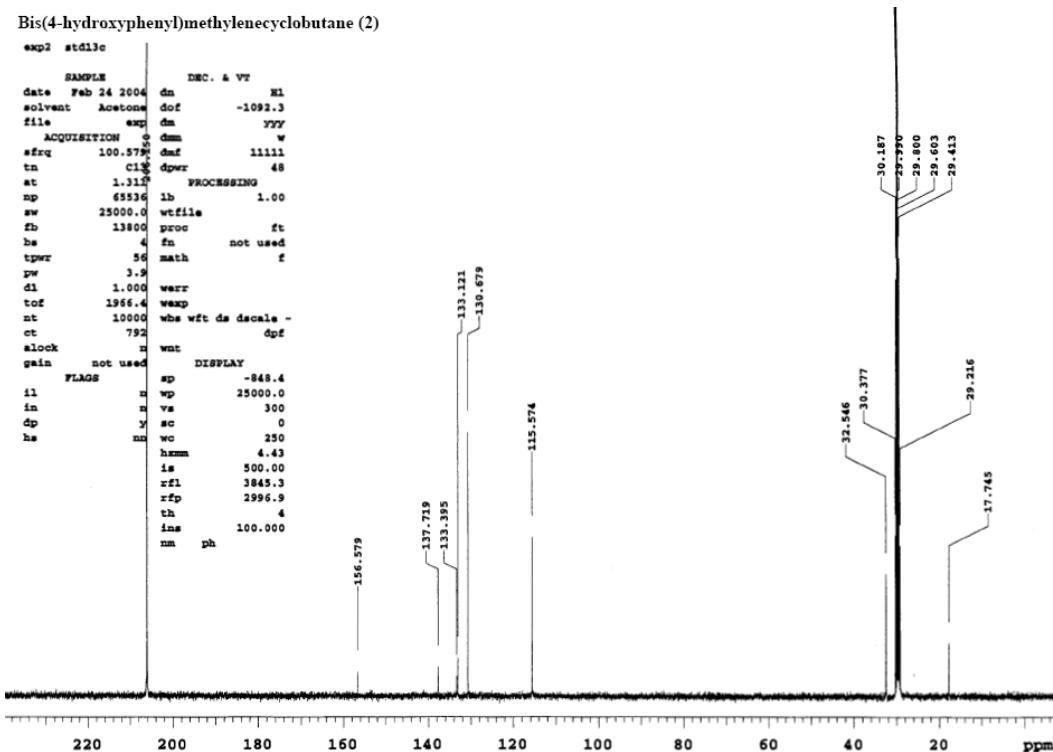


^{13}C NMR (100 MHz, acetone- d_6)

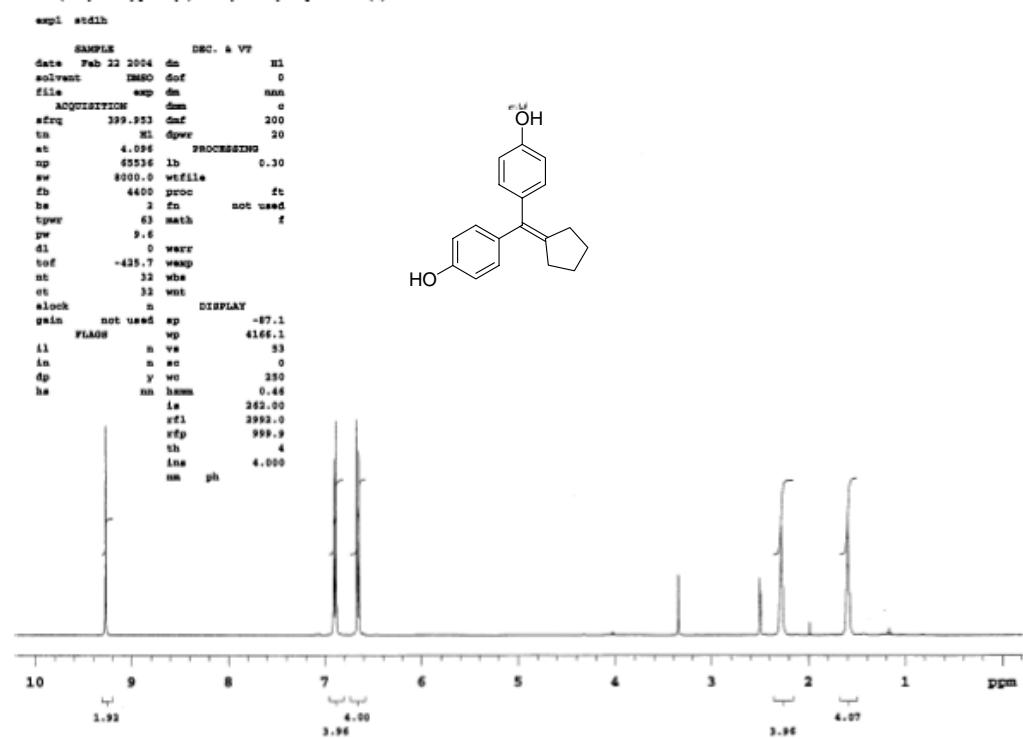
Bis(4-hydroxyphenyl)methylenecyclobutane (2)

exp2 std1c

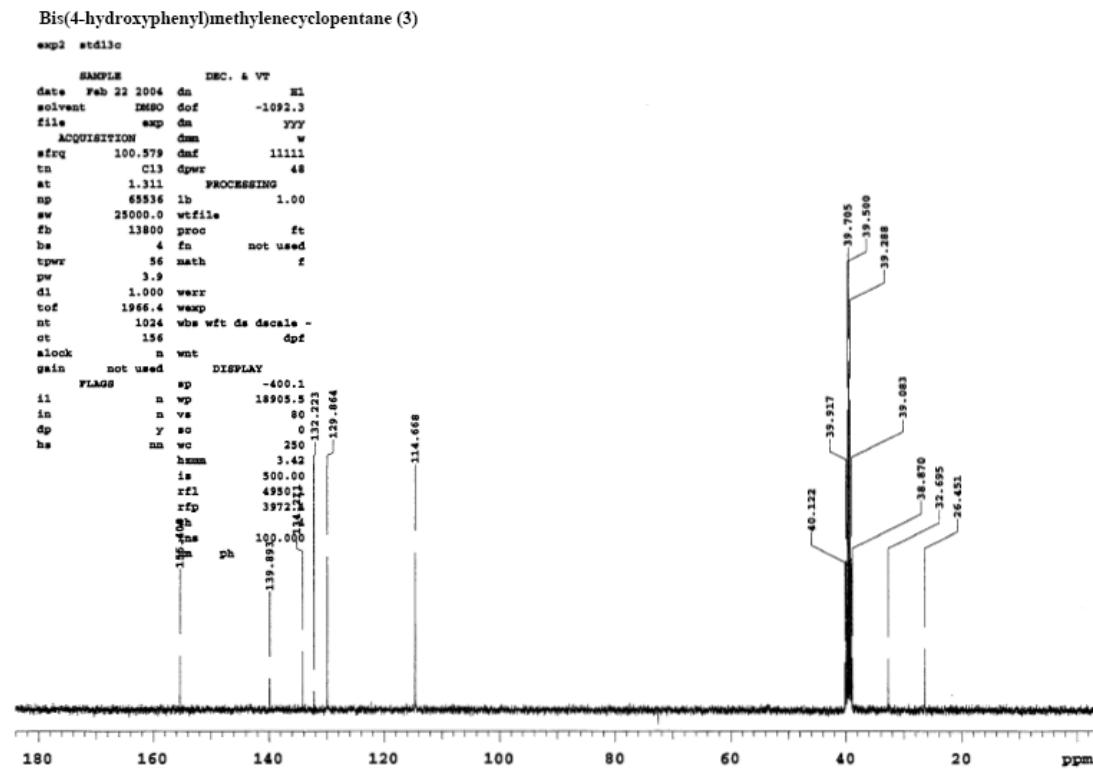
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solvent Acetone dof     -1091.3
file   exp dm      VVY
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sfrq  100.579 dmf     11111
tn    C13 dprw    48
at    1.315 PROCESSING
np    65536 lb      1.00
sw    25000.0 wfile
fb    13800 proc     ft
bs    4 fn      not used
tpwr  56 math     f
pw    3.9
d1    1.000 werr
tof   1966.4 wexp
nt    10000 wbs wft da dscale -
ct    792 dpf
clock  n wnt
gain  not used DISPLAY
FLAGS
il    n wp      25000.0
in    n vs      300
dp    y sc      0
hs    nn wc      250
      hmz     4.43
      is      500.00
      r1f1    3845.3
      r1f2    2996.9
      th      4
      ins    100.000
      nm     ph
```



Bis(4-hydroxyphenyl)methylenecyclopentane (3). ^1H NMR (400 MHz, DMSO- d_6)
 Bis(4-hydroxyphenyl)methylenecyclopentane (3)



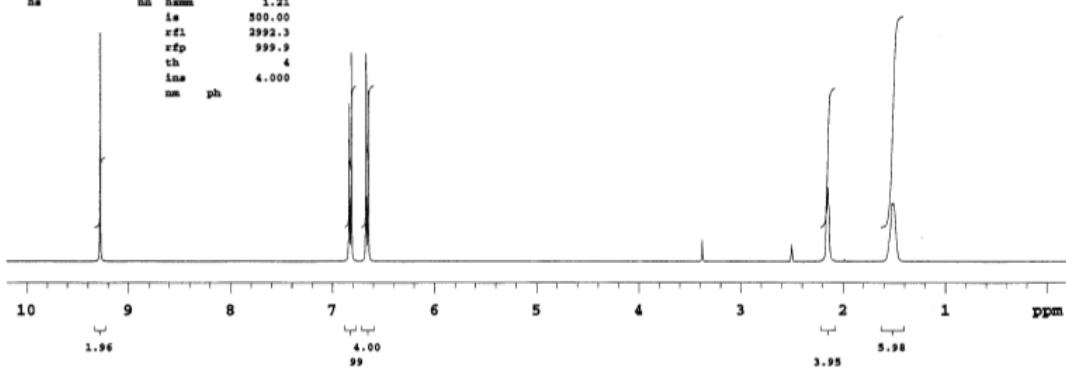
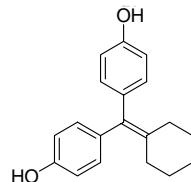
^{13}C NMR (100 MHz, DMSO- d_6)



Bis(4-hydroxyphenyl)methylenecyclohexane (4). ^1H NMR (400 MHz, DMSO- d_6)

Bis(4-hydroxyphenyl)methylenecyclohexane (4)

```
sample stdlh
SAMPLE DEC. & VT
date Feb 22 2004 dn H1
solvent DMSO dof 0
file exp dm nnn
ACQUISITION dnm c
sfrq 399.953 dmf 200
tn K1 dpwr 20
at 4.096 PROCESSING
np 65536 lb 0.30
sw 8000.0 wtfille
fb 4400 proc ft
bs 2 fn not used
tppw 63 math f
pw 9.6
di 0 warr
tof -425.7 wexp
nt 32 vbs
ct 32 wnt
alock n DISPLAY
gain not used sp -87.1
PLAQS wp 4166.1
il n vs 33
in n sc 0
dp y wo 280
hs nn hsmm 1.21
is 500.00
rf1 2992.3
rfp 999.9
th 4
ins 4.000
nm ph
```

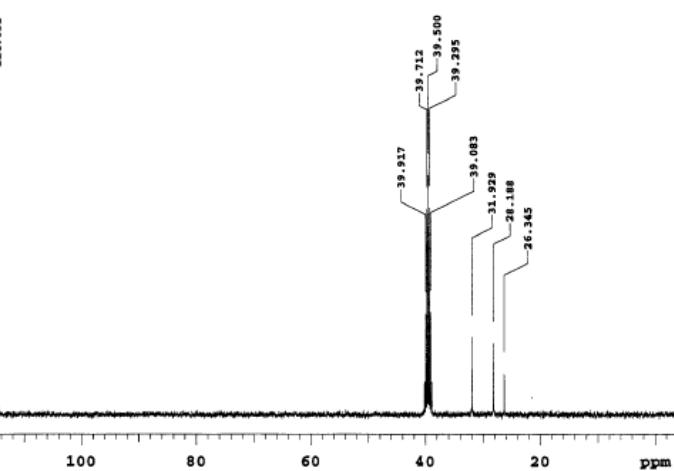


^{13}C NMR (100 MHz, DMSO- d_6)

Bis(4-hydroxyphenyl)methylenecyclohexane (4)

exp2 std13c

```
SAMPLE DEC. & VT
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solvent DMSO dof -1092.3
file exp dm ypy
ACQUISITION dnm w
sfrq 100.579 dmf 11111
tn C13 dpwr 48
at 1.311 PROCESSING 48
np 65536 lb 1.00
sw 25000.0 wtfille
fb 13800 proc ft
bs 4 fn not used
tppw 56 math f
pw 3.9
di 1.000 warr
tof 1966.4 wexp
nt 1024 wbs wft ds dscale 113.11 110.410
ct 128 dfpt
alock n wnt
gain not used DISPLAY
PLAQS sp -400.1
il n wp 18905.5
in n vs 60
dp y sc 0
hs nn wo 250
hsmm 5.63
is 500.00
rf1 498.824
rfp 395.824
th 1.0
ins 100.000
nm ph
```

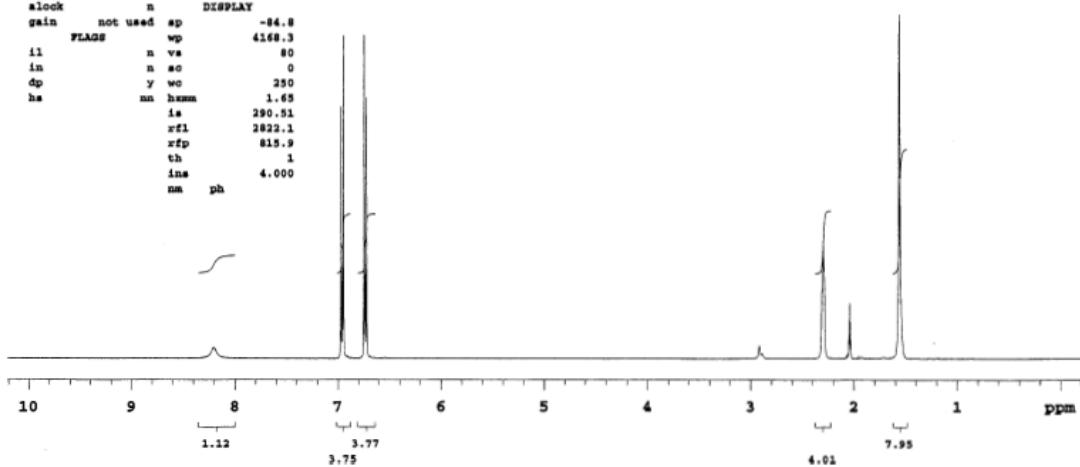
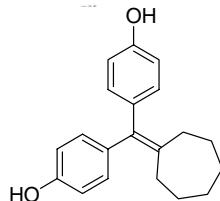


Bis(4-hydroxyphenyl)methylenecycloheptane (5**).** ^1H NMR (400 MHz, acetone- d_6)

Bis(4-hydroxyphenyl)methylenecycloheptane (**5**)

exp1 std13b

```
SAMPLE      DSC. & VT
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solvent   Acetone dof      0
file      exp dm      mnn
ACQUISITION dm      c
sfrq     399.953 dmf    200
tn       H1 dpwv    20
at       4.096 PROCESSING
np       65536 lb      0.30
sw       8000.0 wtf
fb       4400 proc    ft
hs       2 fn      not used
tpwr    63 math    f
pw       9.6
dl       0 warr
tof     -423.7 wexp
nt       32 wha
ct       32 wnt
clock   n
gain    not used sp      -84.8
        wp      4168.3
FLAGS
il       n vs      80
in       n sc      0
dp       y wo      250
ha       nn hsmn    1.65
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        rfp     815.9
        th      1
        ins     4.000
        nm     ph
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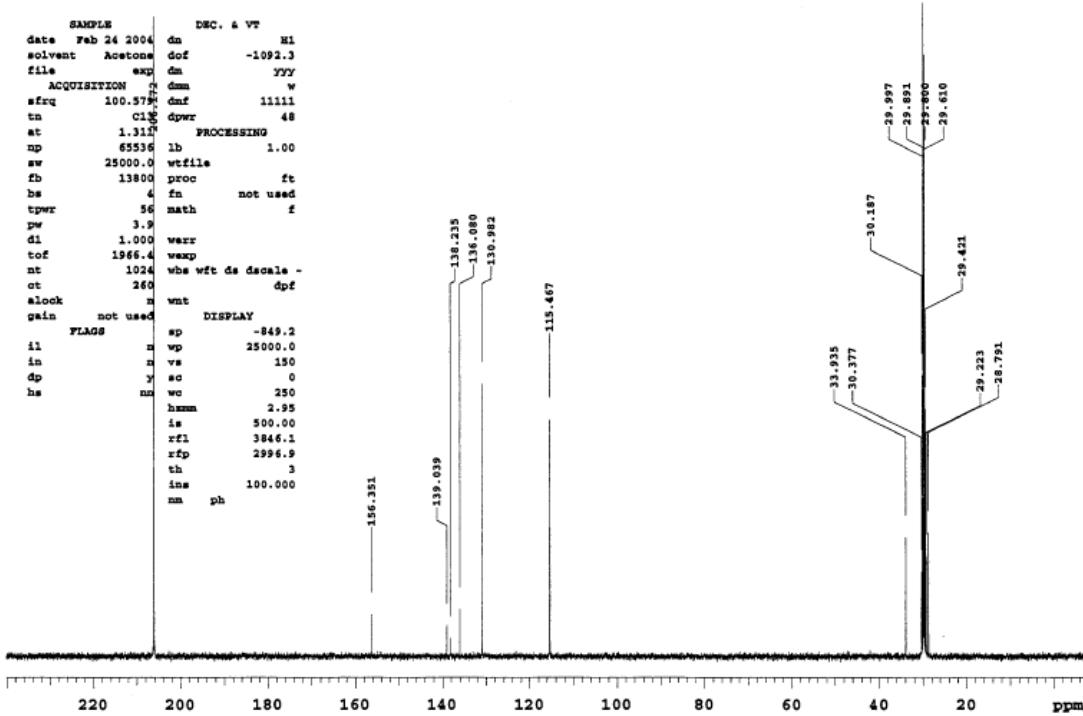


^{13}C NMR (100 MHz, acetone- d_6)

Bis(4-hydroxyphenyl)methylenecycloheptane (**5**)

exp2 std13c

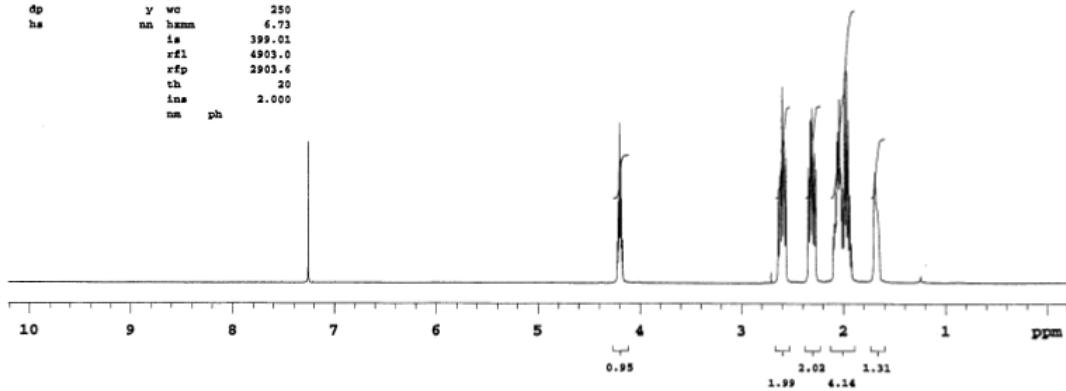
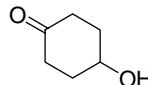
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solvent   Acetone dof    -1092.3
file      exp dm      YY
ACQUISITION dm      w
sfrq     100.378 dmf    11111
tn       C13 dpwv    48
at       1.311 PROCESSING
np       65536 lb      1.00
sw       25000.0 wtf
fb       13800 proc    ft
hs       4 fn      not used
tpwr    56 math    f
pw       3.9
dl       1.000 warr
tof     1966.4 wexp
nt       1024 wba wft ds decalc -
ot       260 dfp
clock   n
gain    not used DISPLAY
FLAGS
il       n wp      25000.0
in       n vs      150
dp       y sc      0
ha       nn wo      250
        hsmn    2.95
        is      500.00
        rfl     3846.1
        rfp     2996.9
        th      3
        ins     100.000
        nm     ph
```



4-Hydroxyhexanone (7). ^1H NMR (400 MHz, CDCl_3)

4-Hydroxyhexanone (7)

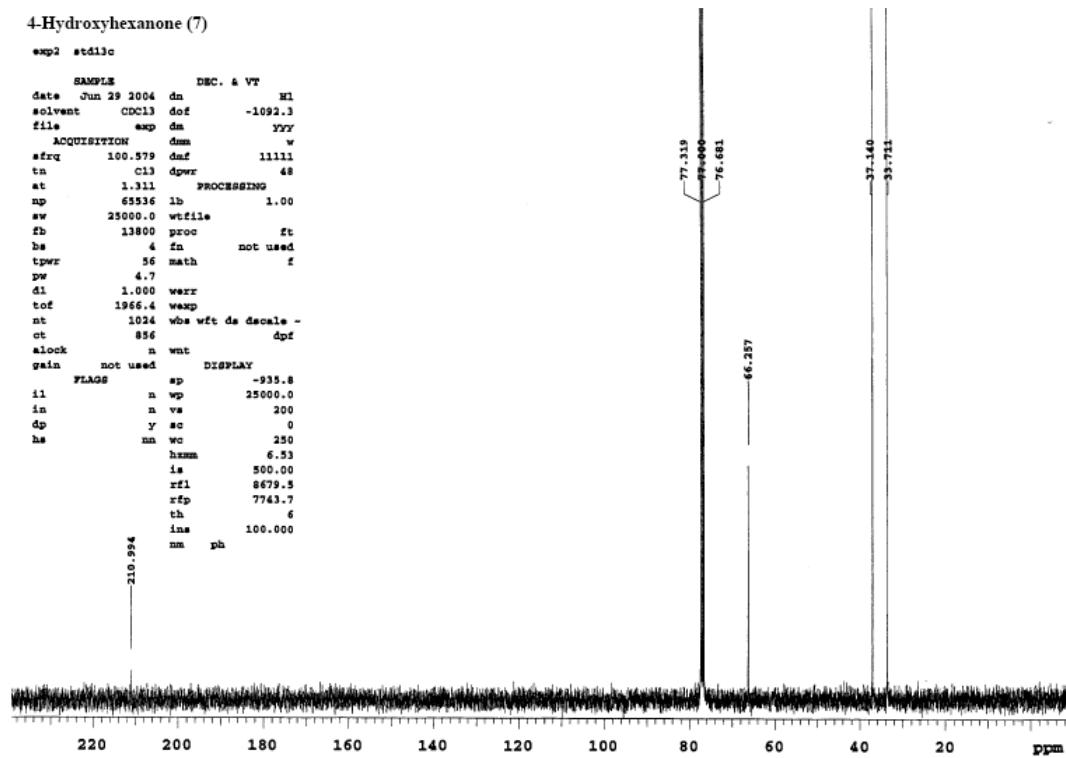
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expt stdib
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date  Jun 29 2004 dn      HL
solvent   CDCl3 dof      0
file      exp dm      nnn
ACQUISITION dm      c
sfrq     399.951 dmf    200
tn       HL dpower 20
at       4.096 PROCESSING
np       65536 lb      0.30
sw      8000.0 wtf file
fb       4400 proc      ft
bs        2 fn      not used
tpwr      63 math      f
pw       10.1
d1        0 warr
tof      -425.7 wmp
nt       64 wbs
ct        34 wnt
slock      n DISPLAY
gain      not used sp      -84.7
FLAGS      wp      4168.3
il        n vs      50
in        n sc      0
dp        y wc      250
hs        nn hzma  6.73
        is      399.01
        rfl      4903.0
        rfp      2903.6
        th      20
        ins      2.000
        nm      ph
```



^{13}C NMR (100 MHz, CDCl_3).

4-Hydroxyhexanone (7)

```
expt stdibc
SAMPLE      DSC. & VT
date  Jun 29 2004 dn      HL
solvent   CDCl3 dof      -1092.3
file      exp dm      YYV
ACQUISITION dm      w
sfrq     100.579 dmf    11111
tn       C13 dpower 48
at       1.311 PROCESSING
np       65536 lb      1.00
sw      25000.0 wtf file
fb       13800 proc      ft
bs        4 fn      not used
tpwr      56 math      f
pw       4.7
d1        1.000 warr
tof      1966.4 wmp
nt       1024 wbs wft ds decalc -
ct        856      dpf
slock      n wnt
gain      not used DISPLAY
FLAGS      sp      -935.8
il        n wp      25000.0
in        n vs      200
dp        y sc      0
hs        nn wc      250
        hzma  6.53
        is      500.00
        rfl      8679.5
        rfp      7743.7
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        nm      ph
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4-Acetoxy cyclohexanone (8). ^1H NMR (400 MHz, CDCl_3) Registry No. 41043-88-3

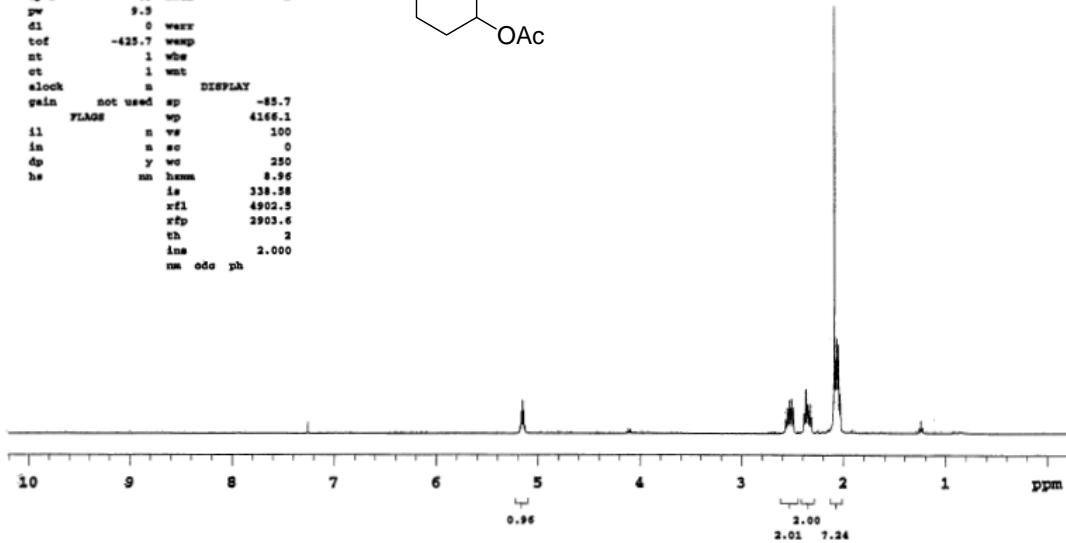
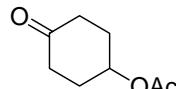
4-Acetoxy cyclohexanone (8)

exptl stdinh

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solvent CDCl3 dof   0
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sfrq  399.951 daf  200
tn     H1 dppr   30
at    4.096 PROCESSING
np    65536 lb   0.10
sw   8000.0 wtf file
fb    4400 proc   ft
bs     16 fn   not used
tpwr   63 math   f
pw    9.5
dl    0 warr
tof   -425.7 wmp
nt     1 wbs
ct     1 wnt
alock  n
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        th   2
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nm   odo ph

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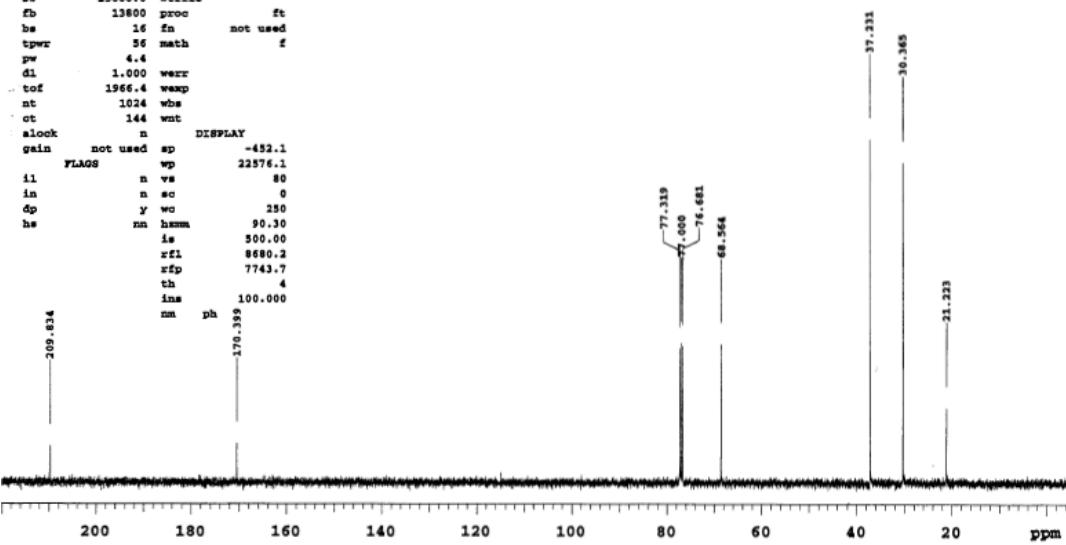
^{13}C NMR (100 MHz, CDCl_3).

4-Acetoxy cyclohexanone (8)

```

SAMPLE      DSC. & VT
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solvent CDCl3 dof   -1092.3
file   exp dm   377
ACQUISITION dm   w
sfrq  100.579 dmf  10101
tn     C13 dppr   48
at    1.311 PROCESSING
np    65536 lb   1.00
sw   25000.0 wtf file
fb    13800 proc   ft
bs     16 fn   not used
tpwr   56 math   f
pw    4.4
dl    1.000 warr
tof   1966.4 wmp
nt    1024 wbs
ct     144 wnt
alock  n
DISPLAY
gain  not used sp   -452.1
FLAGS  n wp   22576.1
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in    n sc   0
dp    y wd   250
hs    nn hsmm  90.30
        is   500.00
        rfl  8680.2
        rfp  7743.7
        th   4
        ins  100.000
nm   ph 359

```



Methyl (4-Oxocyclohexylidene)acetate. ^1H NMR (500 MHz, CDCl_3) Registry No 91158-10-0

Methyl (4-Oxocyclohexylidene)acetate

expt 12pul

```

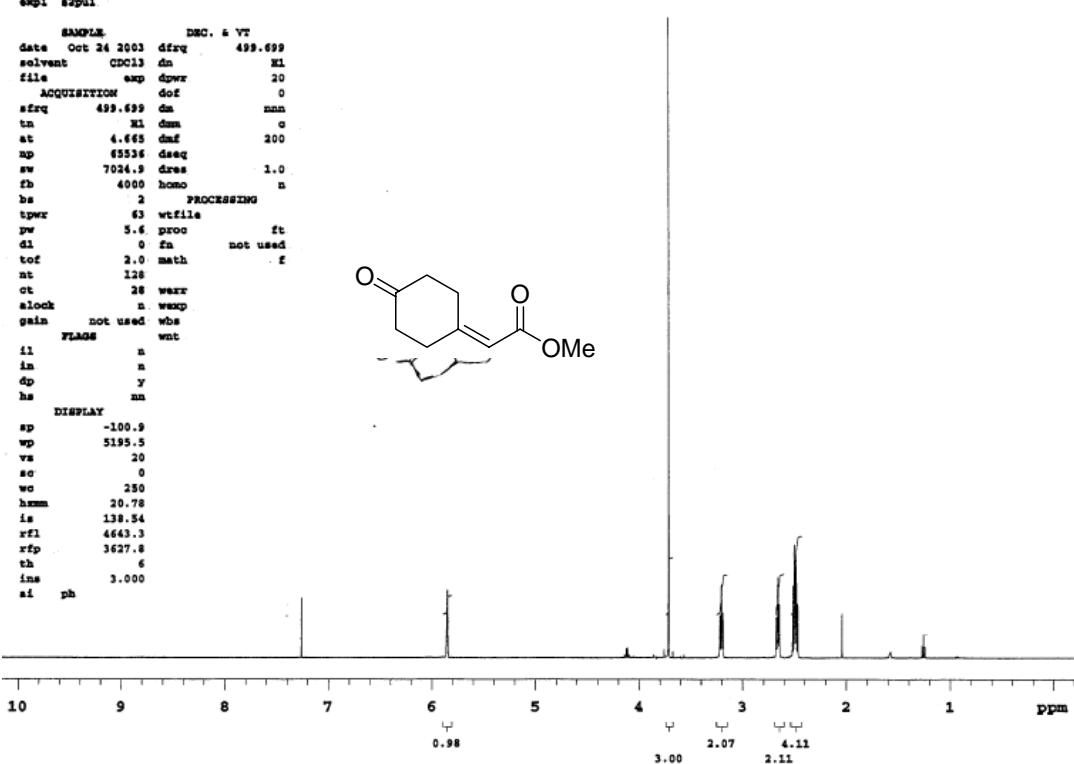
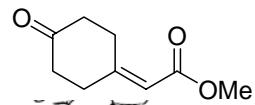
SAMPLE      DEC. & VT
date Oct 24 2003 dfrq 499.699
solvent CDCl3 dn   XL
file       exp dfrq 20
ACQUISITION dof 0
sfrq     499.699 dm  nnn
tn        XL dnm  c
at        4.665 dmf 200
ap        65536 dseq
sw        7024.9 dres 1.0
fb        4000 homo n
bs         2 PROCESSING
tppw    63 wtpw
pw        5.6 proc ft
d1        0 fn  not used
t0f      2.0 math
nt        128
ot        28 warr
clock    n wexp
gain    not used vbs
PLACES   wmt
il        n
in        n
dp        y
hs        nn

```

```

DISPLAY
sp      -100.9
wp      5195.5
vs      20
sc      0
wc      250
hmm    20.78
is      138.54
rf1    4643.3
rf2    3627.8
th      6
ins    3.000
si     ph

```

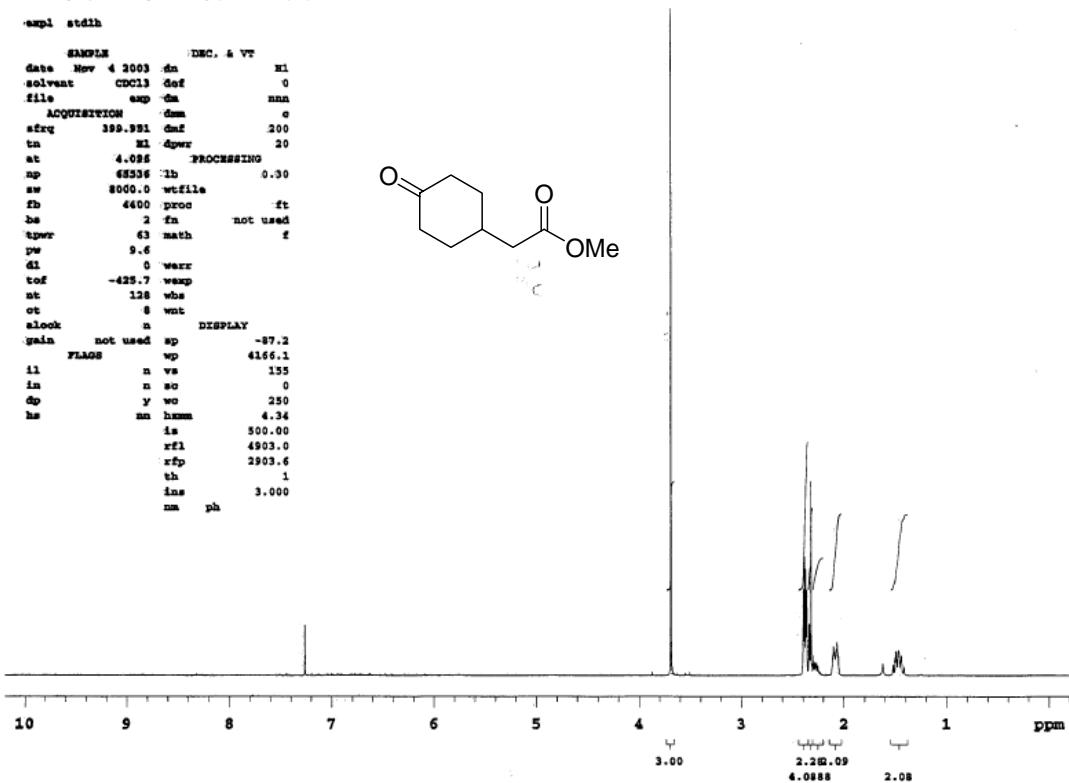
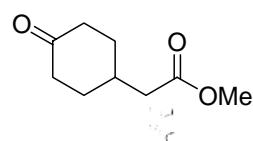


Methyl (4-Oxocyclohexyl)acetate (10). ^1H NMR (400 MHz, CDCl_3) Registry No. 66405-41-2

Methyl (4-Oxocyclohexyl)acetate (10)

exp1 std1h

```
SAMPLE      DSC. & VT
date Nov 4 2003 dn   H1
solvent  CDCl3 dcf    0
file      exp da   nnn
ACQUISITION ddm   e
sfrq     400.001 dmf  200
tn       H1 dppr  20
at       4.095   PROCESSING
np       65536 1b   0.30
sw      8000.0 wtfila
fb       4400  proc   ft
bs        2 fn    not used
tppw     63 math   f
pw       9.6
dl       0 warr
t0f     -425.7 wexp
nt       128 wbs
ot        8 wmt
alock    n
gain    not used sp   -87.2
PLAQS
  wp    4166.1
il      n vs    155
in      n sc    0
dp      y wo   250
hs      nn hsmn  4.34
  is    500.00
  rfl   4903.0
  rfp   2903.6
  th    1
  ins   3.000
  nm   ph
```

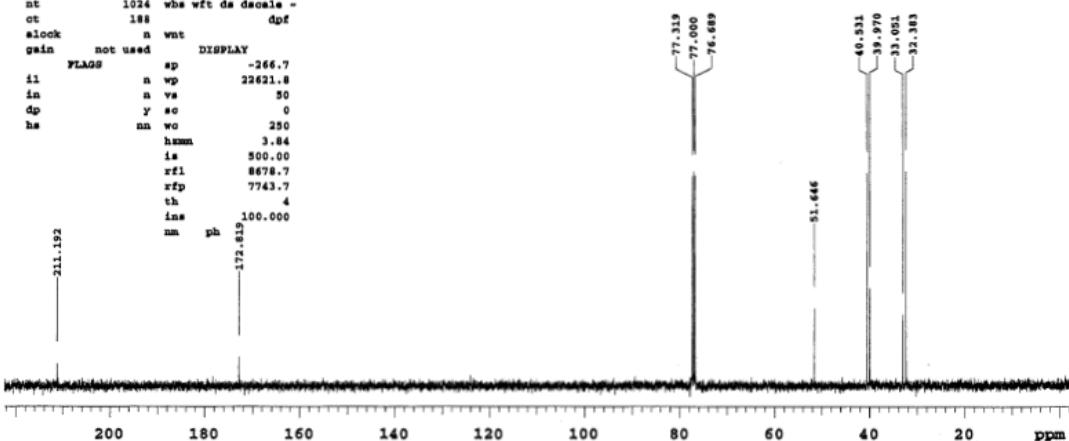


^{13}C NMR (100 MHz, CDCl_3)

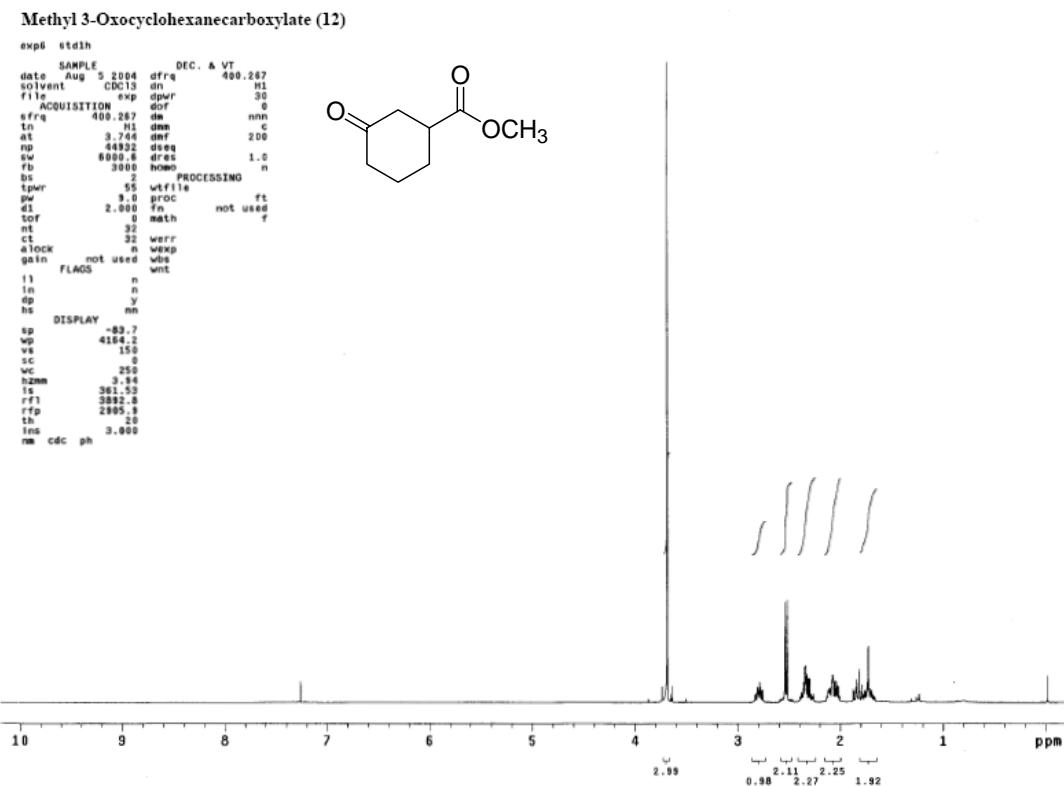
Methyl (4-Oxocyclohexyl)acetate (10)

exp2 std13c

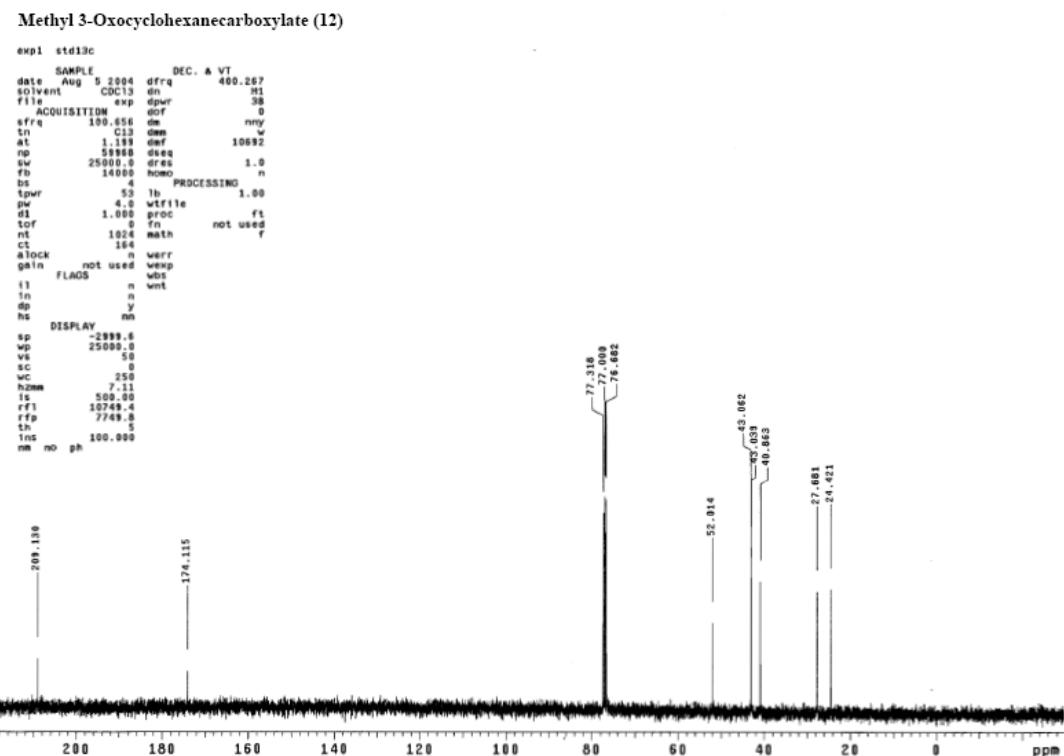
```
SAMPLE      DSC. & VT
date Nov 4 2003 dn   -1092.3
solvent  CDCl3 dcf    48
file      exp da   nnn
ACQUISITION ddm   e
sfrq     100.579 dmf  11111
tn       C13 dppr  48
at       1.311   PROCESSING
np       65536 1b   1.00
sw      25000.0 wtfila
fb       13800  proc   ft
bs        4 fn    not used
tppw     56 math   f
pw       1.9
dl       1.000 warr
t0f     1986.4 wexp
nt       1024 wbs wft ds dscale -
ot        188 dpf
alock    n
gain    not used sp   -266.7
PLAQS
  wp    22621.8
il      n vs    50
in      n sc    0
dp      y wo   250
hs      nn hsmn  3.84
  is    500.00
  rfl   8678.7
  rfp   7743.7
  th    4
  ins   100.000
  nm   ph
```



Methyl 3-Oxocyclohexanecarboxylate (12). ^1H NMR (400 MHz, CDCl_3) Registry No. 13148-83-9



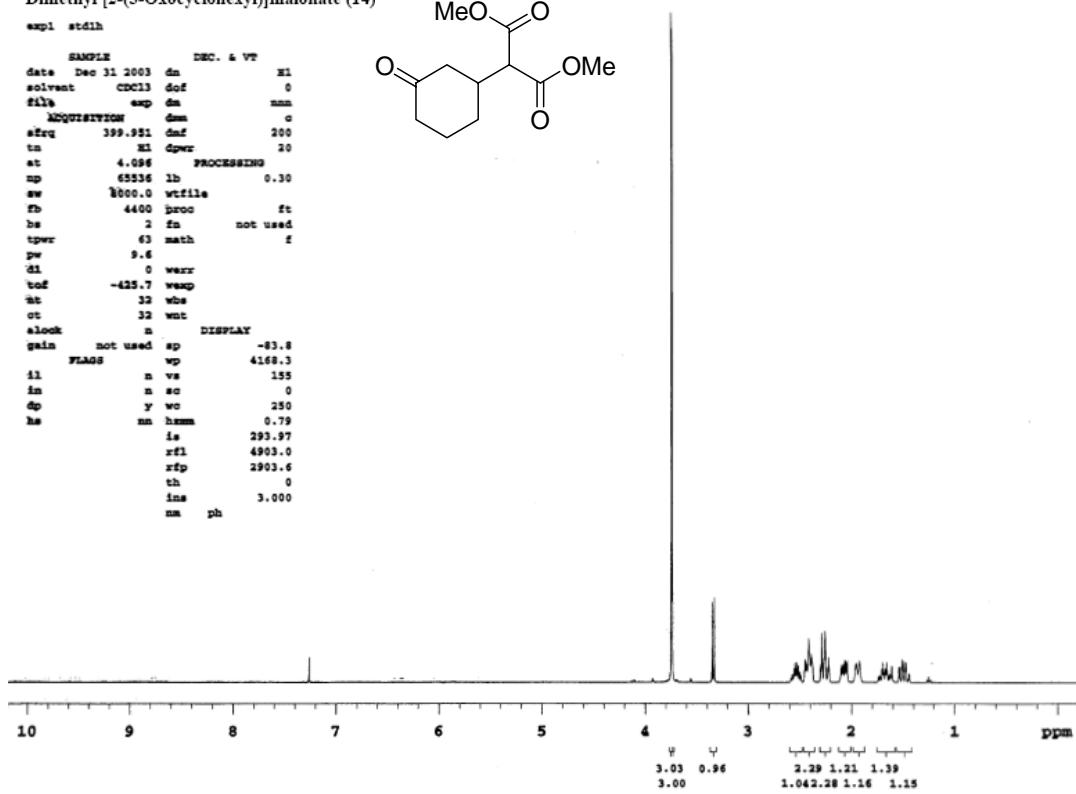
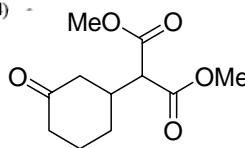
^{13}C NMR (100 MHz, CDCl_3)



Dimethyl [2-(3-Oxocyclohexyl)]malonate (14). ^1H NMR (400 MHz, CDCl_3) Registry No. 33646-18-3

Dimethyl [2-(3-Oxocyclohexyl)]malonate (14)

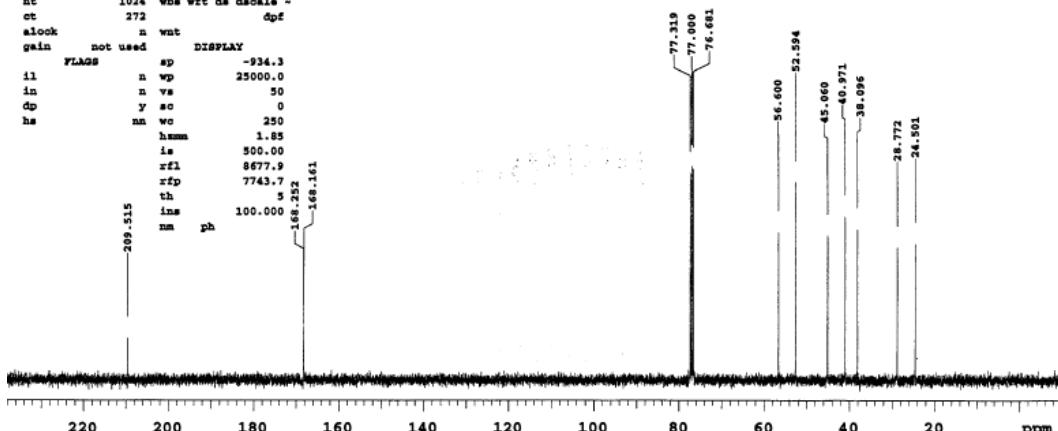
```
exp1 std1h
SAMPLE          DEC. & VT
date  Dec 31 2003  dn      H1
solvent   CDCl3  dof      0
file     exp  dm      nnn
ACQUISITION  dnmw  w
afrq    399.951  dnf     200
tn      H1  dppr     10
at      4.098  PROCESSING
np      65536  lb      0.30
sw     16000.0  wtf file
fb      4400  proc      ft
bs      2  fn      not used
tpwr    63  math      f
pw      9.6
dl      0  warr
tof    -425.7  wexp
ab      32  wba
ct      32  wnt
alock    n  DISPLAY
gain  not used sp      -83.8
FLAGS    wp      4168.3
il      n  vs      155
in      n  sc      0
dp      y  wo      250
hs      nn  hmn      0.79
           is      293.97
           rfl     4903.0
           rfp     2903.6
           th      0
           ins     3.000
           ms      ph
```



^{13}C NMR (100 MHz, CDCl_3)

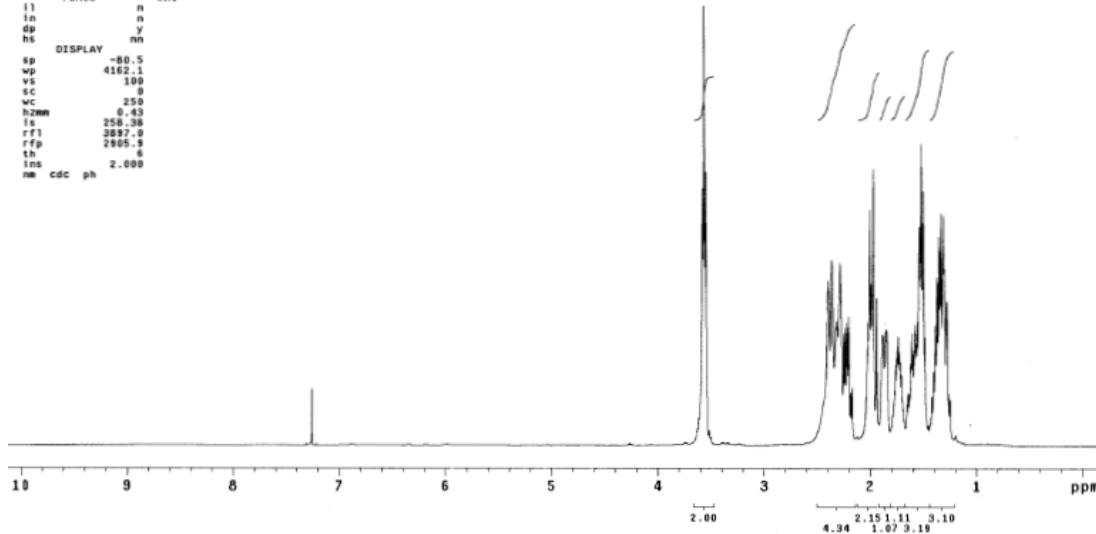
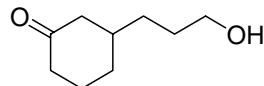
Dimethyl [2-(3-Oxocyclohexyl)]malonate (14)

```
exp2 std1c
SAMPLE          DEC. & VT
date  Dec 31 2003  dn      H1
solvent   CDCl3  dof     -1092.3
file     exp  dm      VVY
ACQUISITION  dnmw  w
afrq    100.579  dnf     11111
tn      C13  dppr     48
at      1.311  PROCESSING
np      65536  lb      1.00
sw     25000.0  wtf file
fb      13800  proc      ft
bs      4  fn      not used
tpwr    56  math      f
pw      3.9
dl      1.000  warr
tof    1966.4  wexp
nt      1024  wba wft da dscale -
et      272  dpf
alock    n  wnt
gain  not used DISPLAY
FLAGS    sp      -934.3
il      n  wp      25000.0
in      n  vs      50
dp      y  sc      0
hs      nn  ph      250
           hmn      1.85
           is      500.00
           rfl     8677.9
           rfp     7743.7
           th      5
           ins     100.000
           ms      ph
           hmc      1168.161
           lmc      1168.161
```



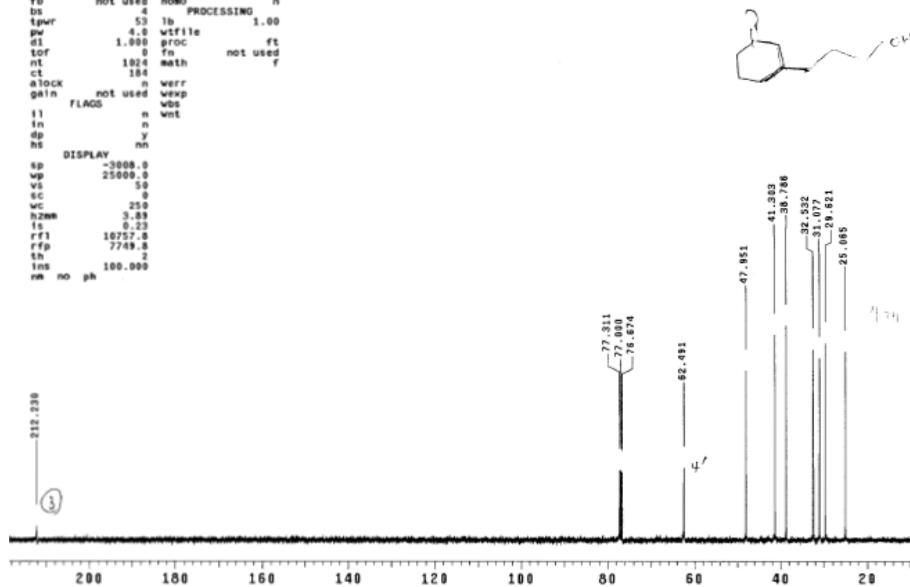
3-(3-Hydroxy-*n*-propyl)cyclohexanone ^1H NMR (400 MHz, CDCl_3) Registry No.69441-81-2.
3-(3-Hydroxy-*n*-propyl)cyclohexanone

```
exp4 std1h
SAMPLE          DEC. & VT      400.267
date   Nov 10 2004 dfraq   H1
solvent    CDCl3 dn       H1
file     exp dppr      38
ACQUISITION dof        0
sfrq    400.267 nms      n
tn      3.741 def       200
np      44932 dppg      0
sw      6000.0 dppf      1.0
fb      not used homo    n
bs      2      PROCESSING
tpwr    55 wtf file
pw      50 proc      ft
di      2.000 fm      not used
tof      0 math
nt      16
ct      16 werr
at      16 wexp
alock    n wexp
gain      not used wbs
FLAGS      n wnt
i1      n
in      n
dp      y
hs      nn
DISPLAY
sp      -80.5
wp      4162.1
vs      109
sc      9
wc      250
h2wm      0.43
is      258.38
rf1      350.0
rf2      2865.9
th      6
tms      2.000
nm cdc ph
```



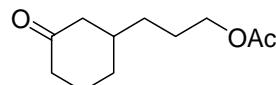
^{13}C NMR (100 MHz, CDCl_3)
3-(3-Hydroxy-*n*-propyl)cyclohexanone

```
exp3 std13c
SAMPLE          DEC. & VT      400.267
date   Nov 10 2004 dfraq   H1
solvent    CDCl3 dn       H1
file     exp dppr      38
ACQUISITION dof        0
sfrq    100.655 nms      n
tn      1.159 def       10692
np      31250 dppg      0
sw      25000.0 dppf      1.0
fb      not used homo    n
bs      4      PROCESSING
tpwr    50 lb      1.00
pw      4.0 wtf file
di      1.000 proc      ft
tof      0 fm      not used
nt      1000 math
ct      184
alock    n werr
gain      not used wexp
FLAGS      n wnt
i1      n
in      n
dp      y
hs      nn
DISPLAY
sp      -30000.0
wp      25000.0
vs      50
sc      9
wc      250
h2wm      0.23
is      10757.8
rf1      10757.8
rf2      7749.8
th      2
tms      100.000
nm no ph
```

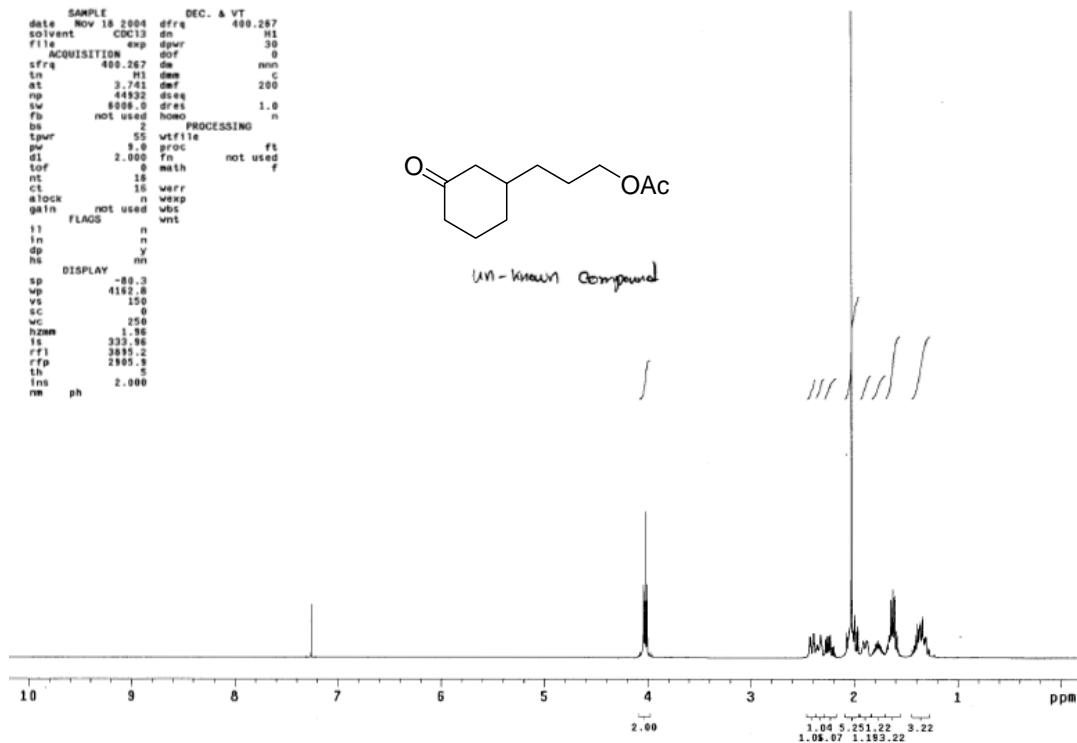


3-(3-Acetoxy-*n*-propyl)cyclohexanone (15). ^1H NMR (400 MHz, CDCl_3)
 3-(3-Acetoxy-*n*-propyl)cyclohexanone (15)

```
exp3 stdin
SAMPLE           DEC. & VT
date   Nov 18 2004 dfrq  400.267
solvent  CDCl3  dm      H1
file   exp dpur  38
      ACQUISITION dof      0
sfreq  400.267 dm      nnn
tn     3.741 def     200
np     44932 dseq
sw     8000.0 dres    1.0
re    not used  home
bs      2          PROCESSING
tpwr   55 wtf file
pw     9.0 proc
dl    2.000 fm      not used
tof      0 math
nt      16
ct      16 warr
clock   2000000000 wps
gain   not used wbs
FLAGS
ii      n
in      n
dp      y
hs      nn
DISPLAY
sp     -80.3
wp     4162.8
vs      150
sc      0
wc      250
hznm   1.36
ts     323.8
rf1    3885.2
rf2    2905.9
th      5
ins    2.000
nm      ph
```



Un-known Compound

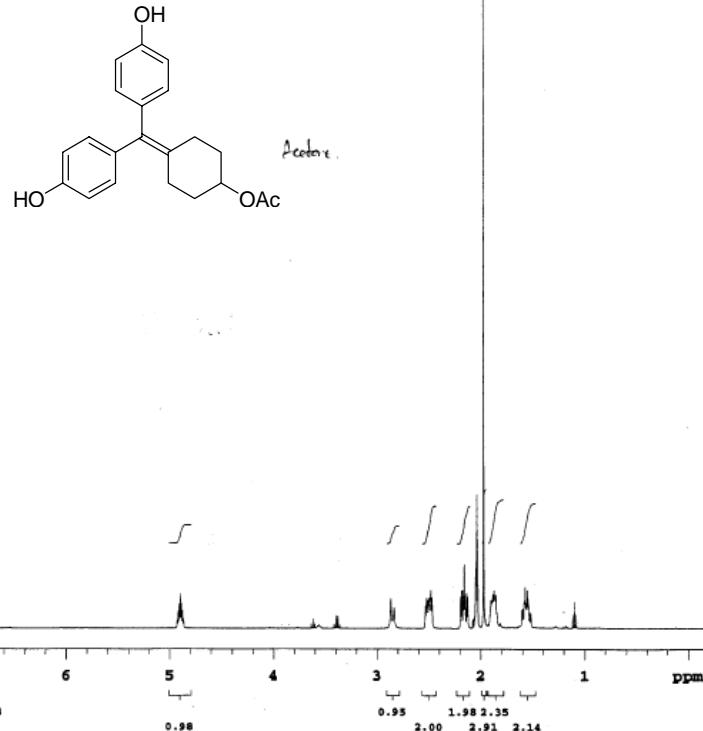


4-Acetoxy-1-[bis(4-hydroxyphenyl)methylene]cyclohexane (16).

¹H NMR (400 MHz, acetone-*d*₆)

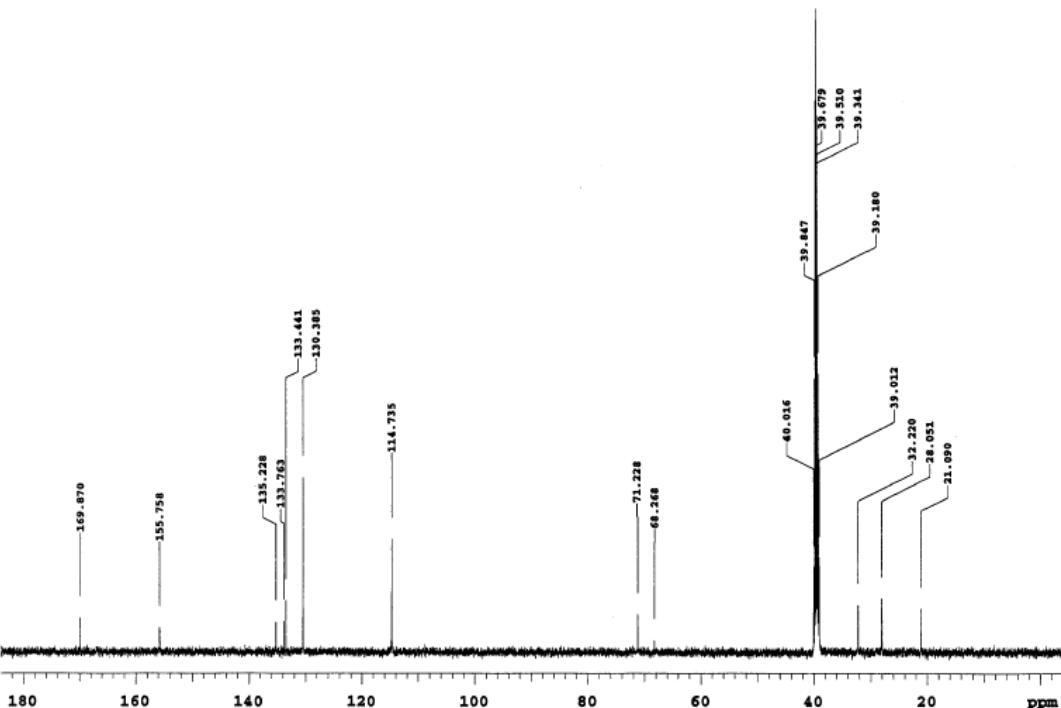
4-Acetoxy-1-[bis(4-hydroxyphenyl)methylene]cyclohexane (16)

```
expt stdth
SAMPLE      DEC. & VT
date Mar 6 2004 dn   H1
solvent Acetone dof   0
file      exp dm   nnn
ACQUISITION dm   c
sfrq    399.953 dmf  200
tn      H1 dppr  20
at      4.056 PROCESSING
np      65536 lb   0.30
sw     8000.0 wtflic
fb      4400 proc  ft
bs      2 fn   not used
tper     63 math  f
nw      9.6
d1      0 warr
t0f    -425.7 weak
nt      32 wba
ot      32 wnt
clock    n
DISPLAY
gain not used sp   -84.8
FLAGS    wp   4168.3
il      n vs   185
in      n sc   0
dp      y wc   250
hs      nn hsmm  2.78
        is   346.91
        rfl  2821.4
        rfp  815.9
        th   6
        ins  2.000
        nm ph
```



¹³C NMR (125 MHz, DMSO-*d*₆)

4-Acetoxy-1-[bis(4-hydroxyphenyl)methylene]cyclohexane (16)



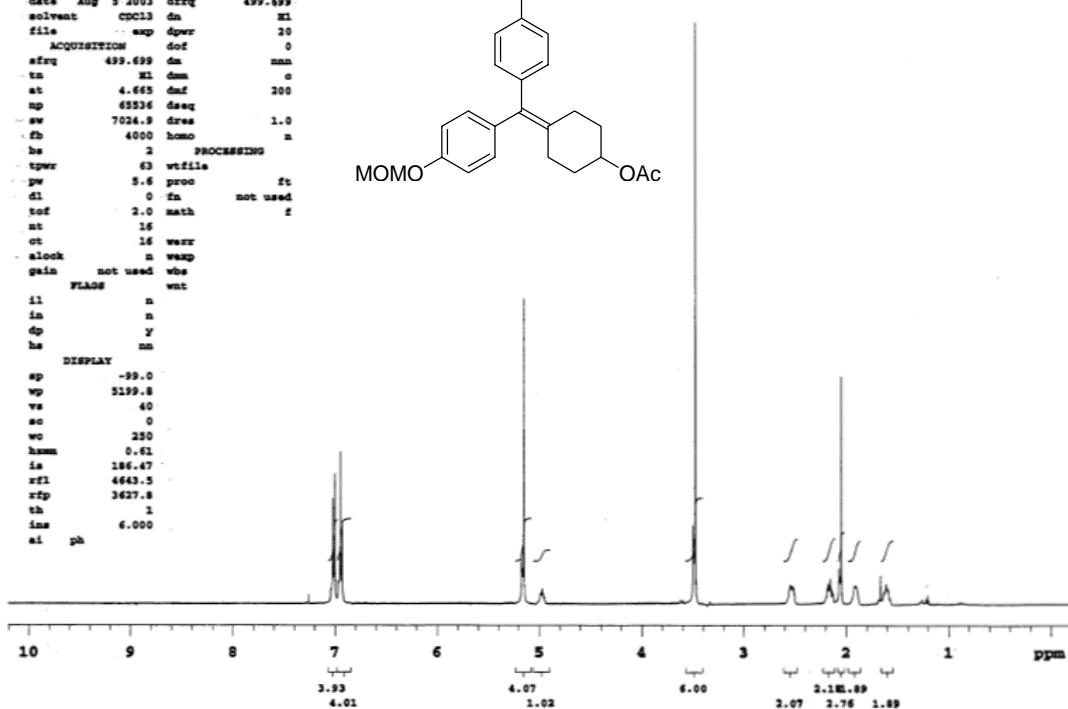
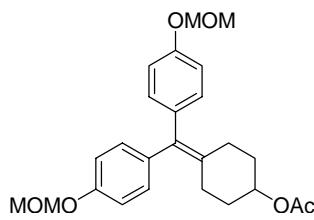
4-Acetoxy-1-[bis(4-methoxymethoxyphenyl)methylene]cyclohexane (17a).

¹H NMR (500 MHz, CDCl₃)

4-Acetoxy-1-[bis(4-methoxymethoxyphenyl)methylene]cyclohexane (17a)

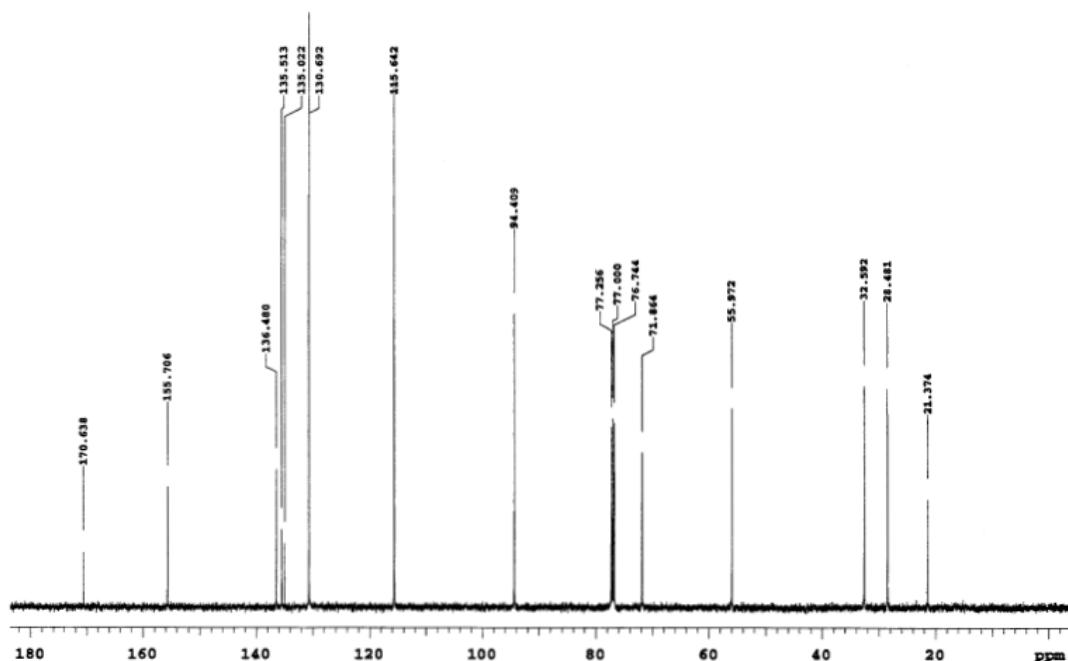
expt1 s1pul

```
SAMPLE          DSC. & VT
date Aug 5 2003 dfrq   499.699
solvent CDCl3 da      NL
file      wmp dppr    20
ACQUISITION dof      0
sfrq   499.699 da     nnn
tin     NL ddm      c
at     4.465 daf    200
np     45336 dseq
sw     7024.8 dres    1.0
fb     4000 homo
hs     2
PROCESSING
tpwr   63 wtfile
pw     5.6 proc     ft
dl     0 fn       not used
t0f    2.0 math
nt     16
ct     16 warr
alock  n wmp
gain   not used whs
PLACES
il     n
in     n
dp     Y
hs     nn
DISPLAY
sp     -99.0
wp     5199.8
vs     40
sc     0
wc     250
hmax  0.6L
is     186.47
rf1    4443.5
rf2    3427.8
tb     1
im    6.000
ai    ph
```



¹³C NMR (125 MHz, CDCl₃)

4-Acetoxy-1-[bis(4-methoxymethoxyphenyl)methylene]cyclohexane (17a)



4-[Bis(4-methoxymethoxyphenyl)methylene]cyclohexanol (17b).

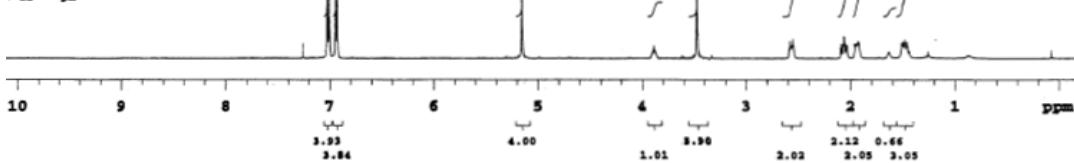
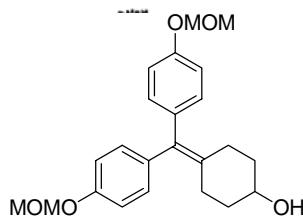
¹H NMR (500 MHz, CDCl₃)

4-[Bis(4-methoxymethoxyphenyl)methylene]cyclohexanol (17b)

```

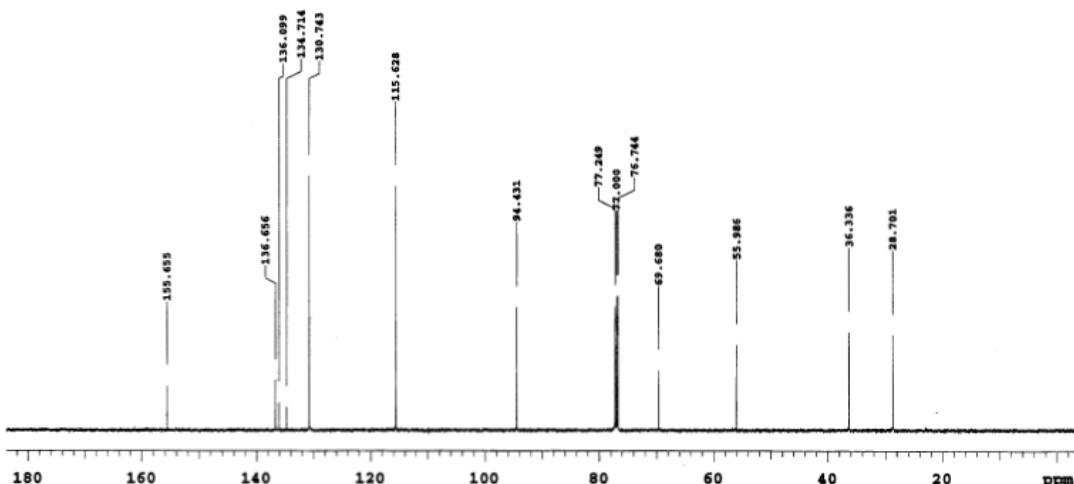
sample s2pul
      DSC. & VT
date Aug 7 2003 dfrq 499.699
solvent CDCl3 dn x1
file   wwp dfrw 20
ACQUISITION dof 0
dfrq 499.699 dn nra
tn    4.665 dnf 200
sp    65536 dswq
sw    7024.9 dsw 1.0
fb    4000 homo n
bs    2 PROCESSING
tpwv 63 wvfile
pw    5.6 proc ft
dl    0 fn not used
tof   2.0 math f
nt    16
ot    4. warr
clock n wexp
gain  not used whs
PLATE wmt
il    n
in    n
dp    y
hs    mn
DISPLAY
sp    -101.8
wp    5235.5
vs    26
sc    0
wo    230
hmn  5.45
is    221.72
rfl  1016.4
rfp  0
th    7
ins  4.000
ai    ph

```



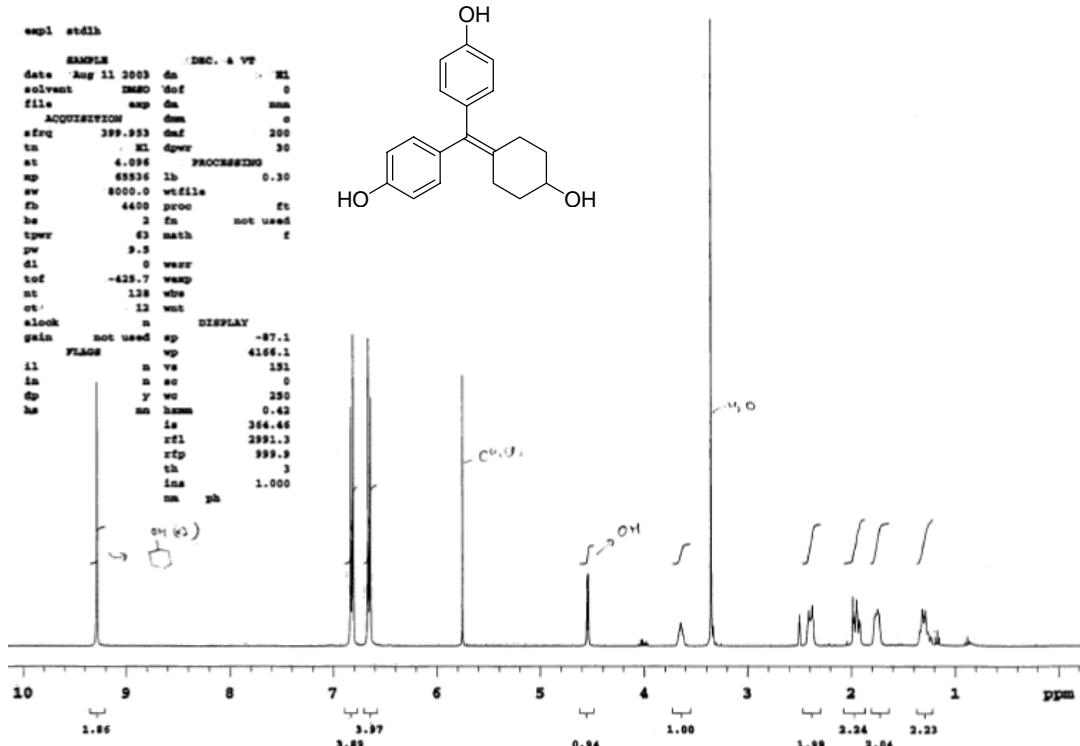
¹³C NMR (125 MHz, CDCl₃)

4-[Bis(4-methoxymethoxyphenyl)methylene]cyclohexanol (17b)



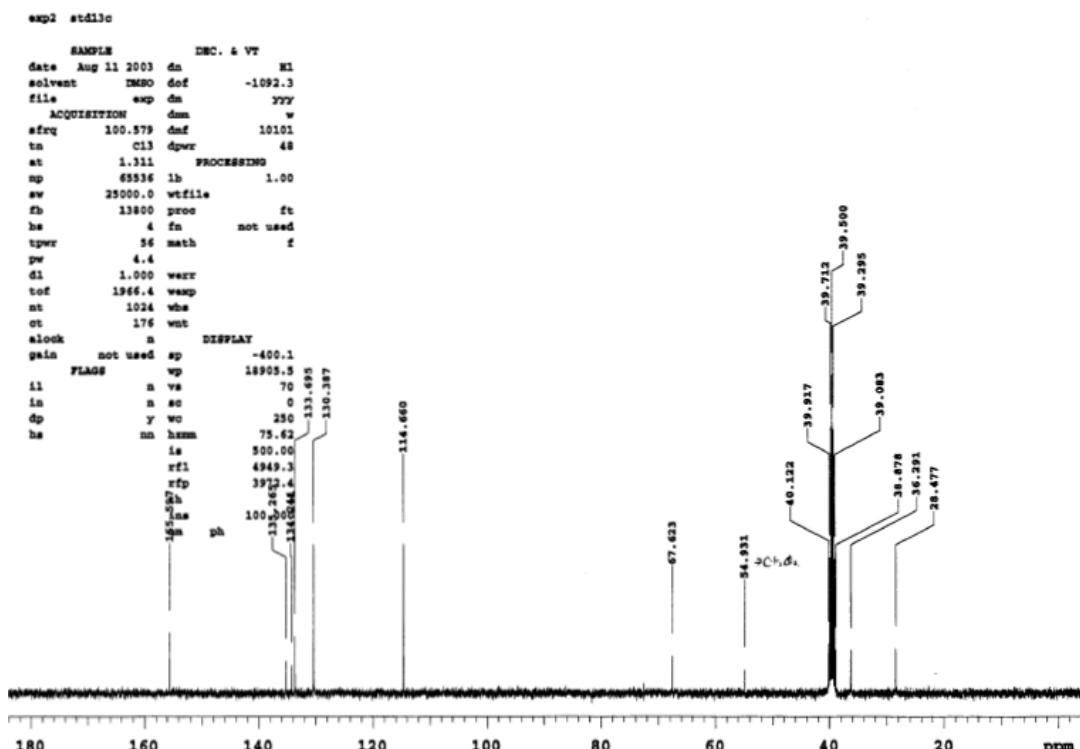
4-[Bis(4-hydroxyphenyl)methylene]cyclohexanol (18). ^1H NMR (400 MHz, $\text{DMSO}-d_6$)

4-[Bis(4-hydroxyphenyl)methylene]cyclohexanol (18)



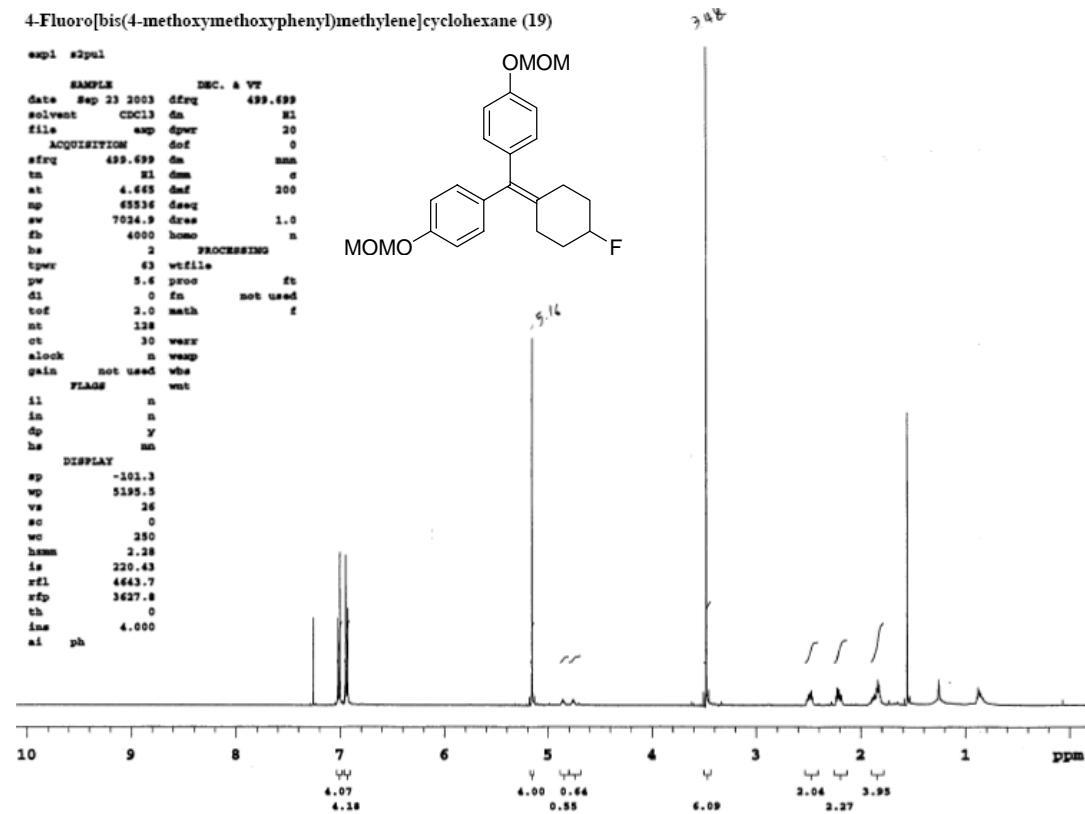
¹³C NMR (100 MHz, DMSO-*d*₆)

4-[Bis(4-hydroxyphenyl)methylene]cyclohexanol (18)



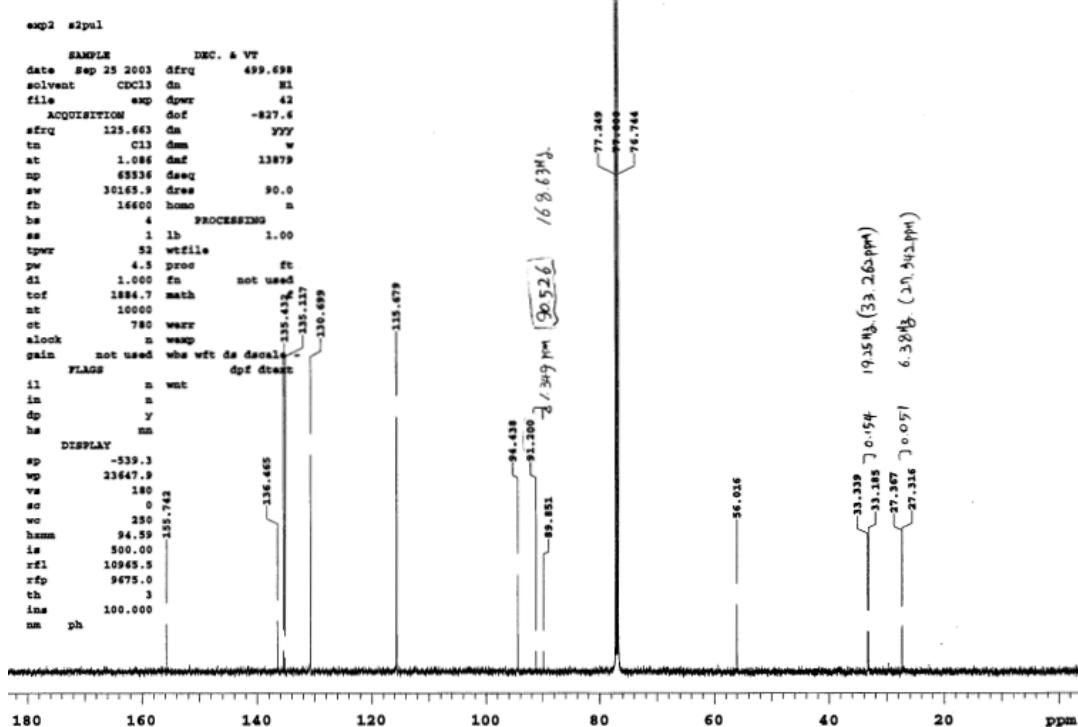
4-Fluoro[bis(4-methoxymethoxyphenyl)methylene]cyclohexane (19).

¹H NMR (500 MHz, CDCl₃)



¹³C NMR (125 MHz, CDCl₃)

4-Fluoro[bis(4-methoxymethoxyphenyl)methylene]cyclohexane (19)



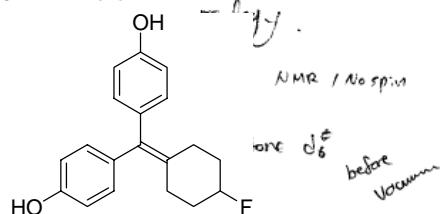
4-Fluoro[bis(4-hydroxyphenyl)methylene]cyclohexane (20).

¹H NMR (500 MHz, acetone-*d*₆)

4-Fluoro[bis(4-hydroxyphenyl)methylene]cyclohexane (20)

exp1 s2pul

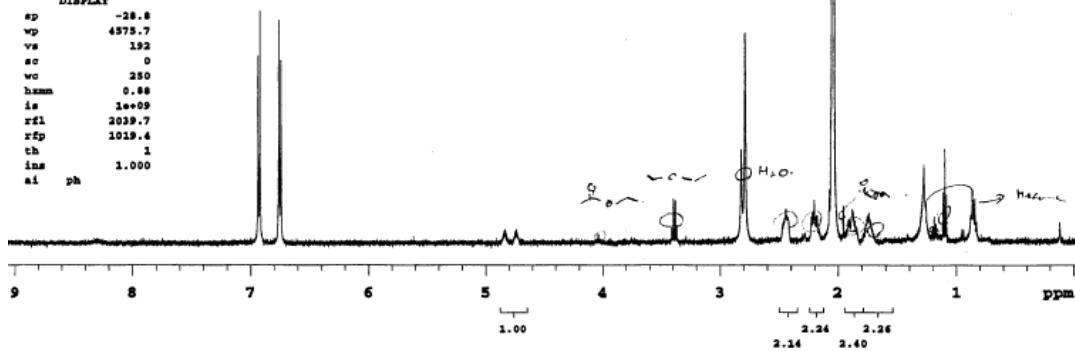
```
SAMPLE DEC. & VT
date Sep 29 2003 dfrq 499.702
solvent Acetone dn NL
file exp dpwr 20
ACQUISITION dof 0
sfrq 499.702 dm nnn
tn 499.702 NL dnm a
at 4.665 dmf 200
np 65536 dseq
sw 7024.9 drss 1.6
fb 4000 homo n
bs 2 PROCESSING
tpwr 5.0 wtfile
pw 5.0 prod ft
d1 0 fn not used
tof 2.0 math f
nt 128
ct 74 warr
clock n wexp
gain not used wbs
FLAGS wmt
il n
in n
dp y
hs nm
DISPLAY
sp -28.8
wp 4575.7
vs 192
sc 0
wc 250
hzmw 0.68
is 1e+09
rfl 2039.7
rfp 1019.4
th 1
ins 1.000
ai ph
```



NMR / No spin

one J₆
before
vacuum

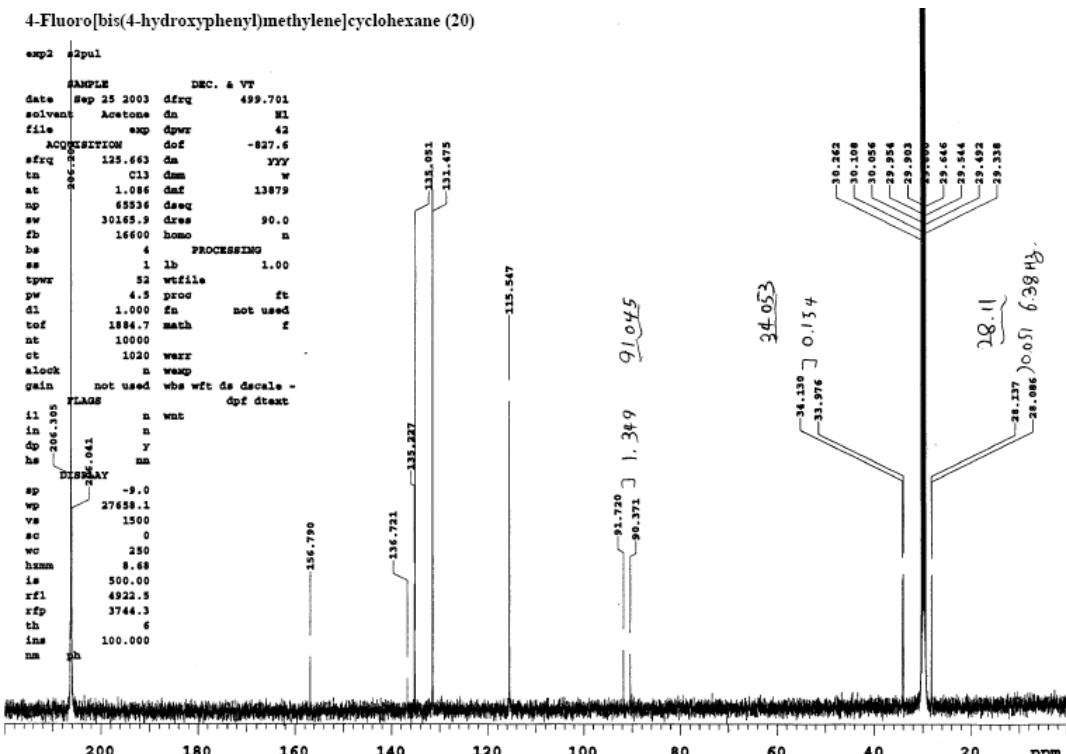
95%



¹³C NMR (125 MHz, acetone-*d*₆)

4-Fluoro[bis(4-hydroxyphenyl)methylene]cyclohexane (20)

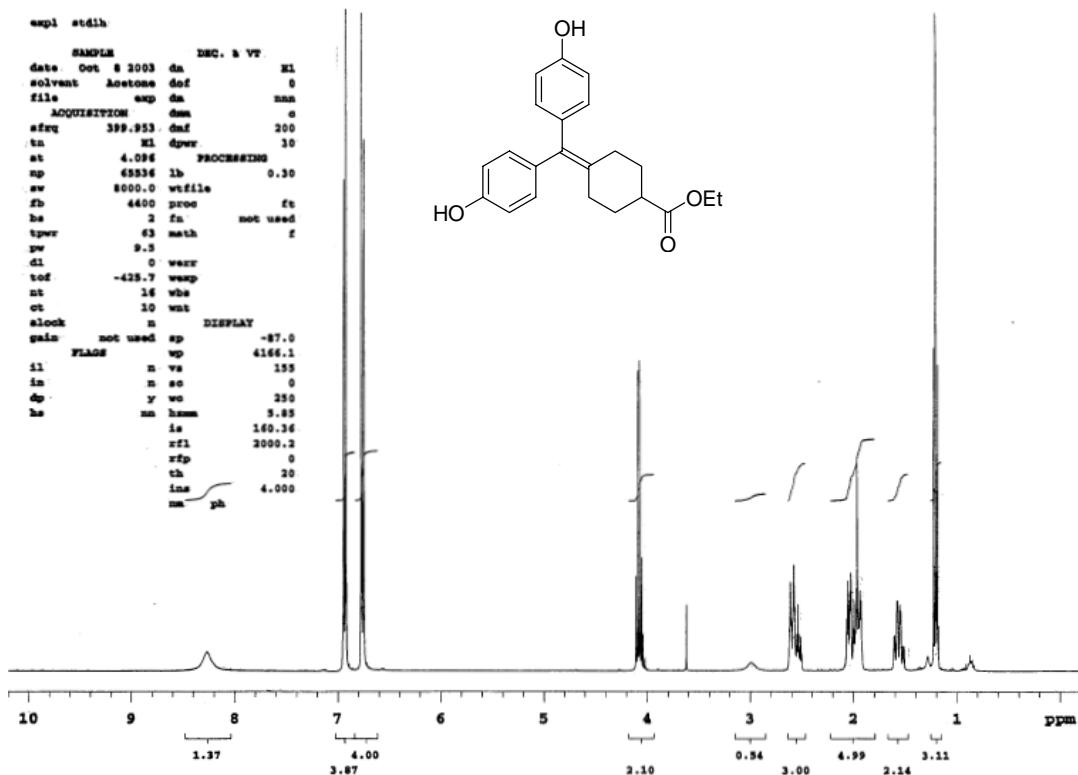
```
exp2 s2pul
SAMPLE DEC. & VT
date Sep 29 2003 dfrq 499.701
solvent Acetone dn NL
file exp dpwr 42
ACQUISITION dof -827.6
sfrq 125.663 dm VVY
tn C13 dnm w
at 1.086 dmf 13879
np 65536 dseq
sw 30165.9 drss 90.0
fb 16600 homo n
bs 4 PROCESSING
ss 1 lb 1.00
tpwr 52 wtfile
pw 4.5 prod ft
d1 1.000 fm not used
tof 1884.7 math f
nt 10000
ct 1020 warr
clock n wexp
gain not used wbs wft ds dscale -
FLAGS dpt dtaxt
il n wmt
in n
dp 256.041 y
hs nm
DISPLAY
sp -9.0
wp 27658.1
vs 1500
sc 0
wc 250
hzmw 0.68
is 500.00
rfl 4932.5
rfp 3744.3
th 6
ins 100.000
nm ph
```



Ethyl 4-[Bis(4-hydroxyphenyl)methylene]cyclohexanecarboxylate (21a).

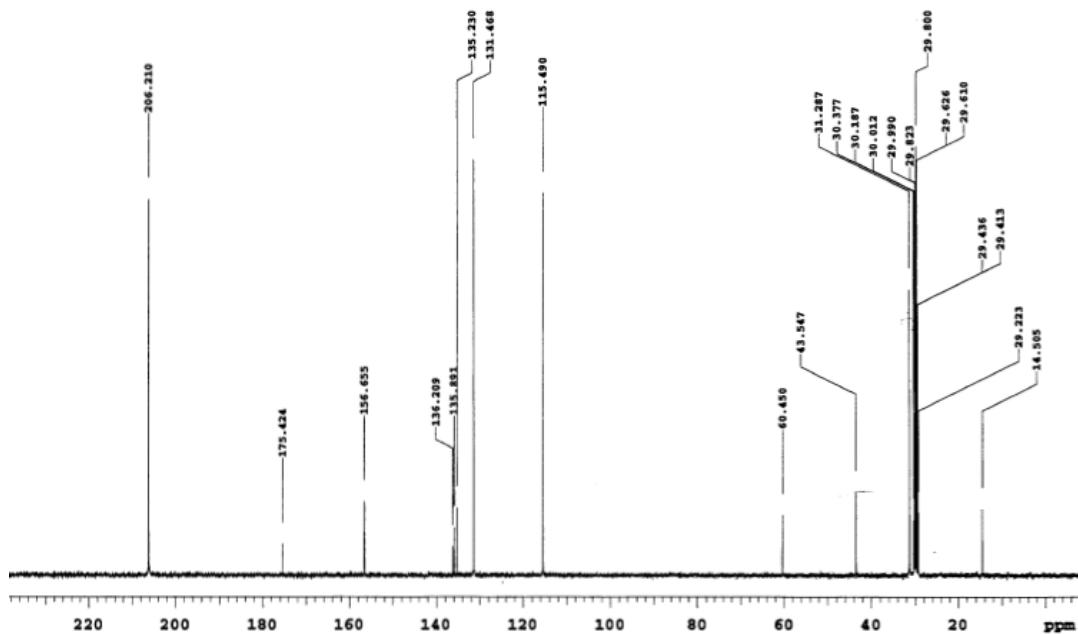
¹H NMR (400 MHz, acetone-*d*₆)

Ethyl 4-[Bis(4-hydroxyphenyl)methylene]cyclohexanecarboxylate (21a)



¹³C NMR (100 MHz, acetone-*d*₆)

Ethyl 4-[Bis(4-hydroxyphenyl)methylene]cyclohexanecarboxylate (21a)



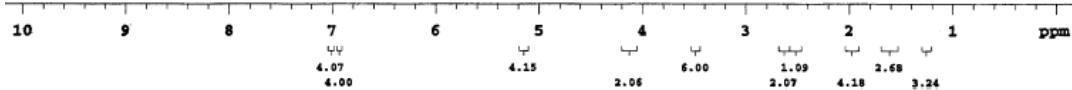
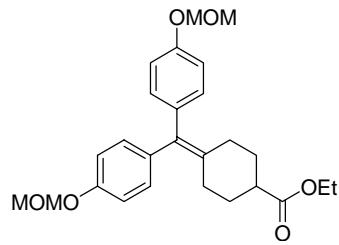
Ethyl 4-[Bis(4-methoxymethylphenyl)methylene]cyclohexanecarboxylate (22a).

¹H NMR (400 MHz, CDCl₃)

Ethyl 4-[Bis(4-methoxymethylphenyl)methylene]cyclohexanecarboxylate (22a)

expl. stalk

SAMPLE		DEC.	AT
date	Oct 3 2003	dn	
solvent	(CHCl ₃)	dof	
file		dn	
ACQUISITION		dnm	
sfrq	399.851	dof	20
tn	11	dper	3
at	4.056	PROCESSING	
sp	65536	lb	0.3
sw	.00000	wtfille	
fb	4400	proc	f
bs	2	fn	not use
tper	63	math	
pw	9.5		
dl	0	warr	
tof	-425.7	wexp	
nt	16	wba	
ot	8	wut	
alock	n	DISPLAY	
gain	not used	sp	-78.
PLATES		wp	4158.
il	n	vs	15
in	n	sc	
dp	y	wo	25
hs	nn	hsmm	0.9
		iz	278.4
		rfl	4903.
		rtp	2903.
		th	
		ins	6.00
		ns	
		ub	

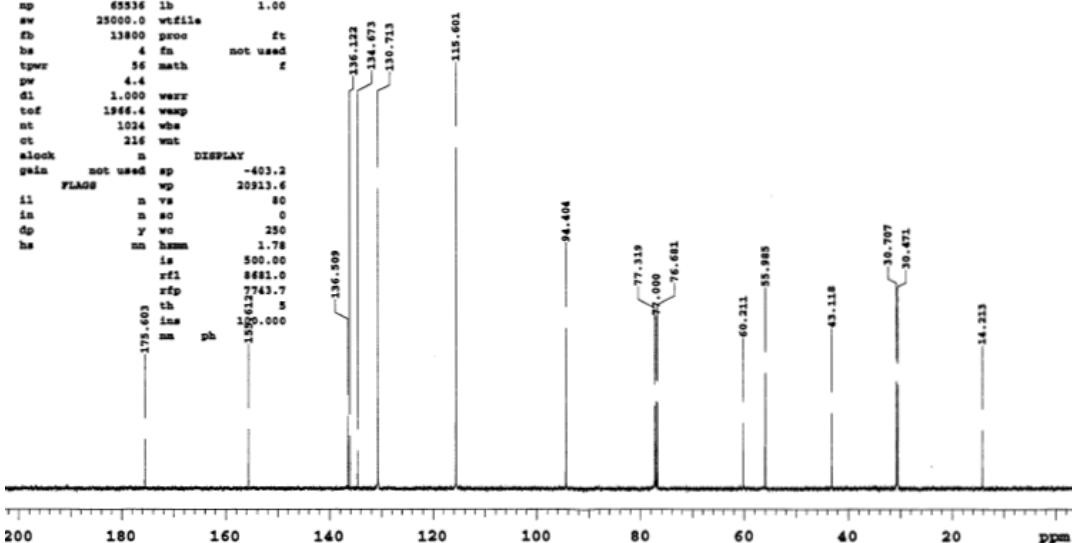


¹³C NMR (100 MHz, CDCl₃)

Ethyl 4-[Bis(4-methoxymethylphenyl)methylene]cyclohexanecarboxylate (22a)

exp2 std13c

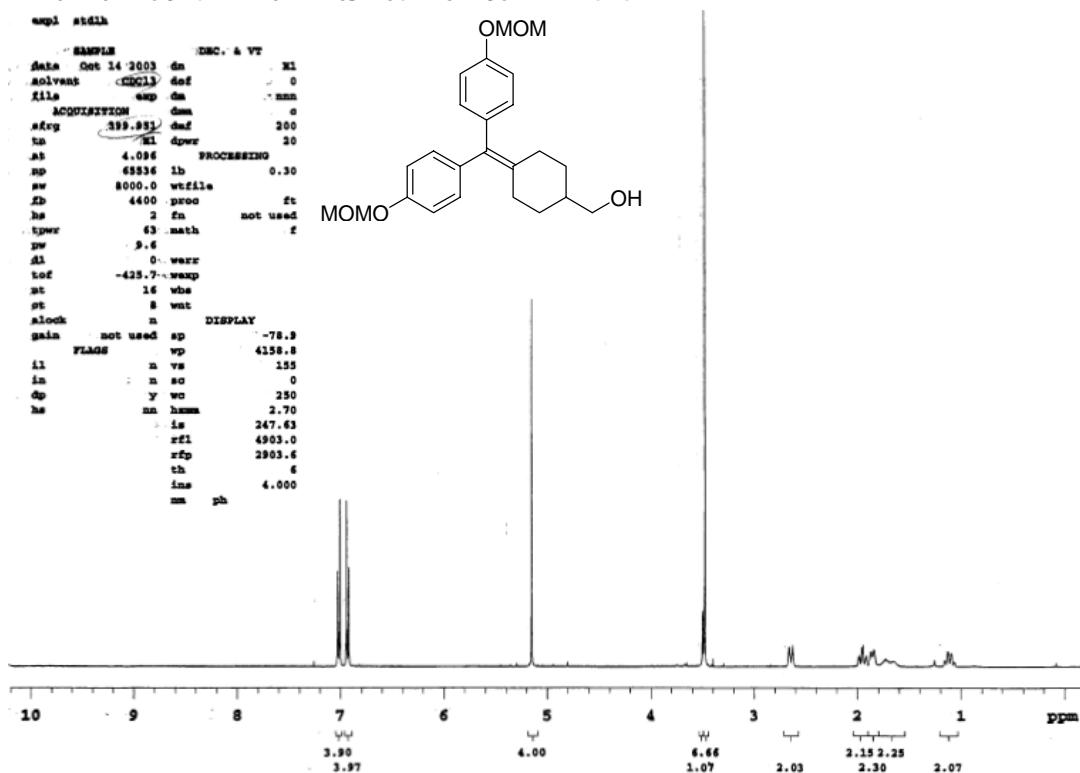
SAMPLE		DEC. & VT	
date	Oct 9 2003	da	HE
solvent	CDC13	dof	-1093.
file		da	Y
ACQUISITION			
sfreq	100.579	daf	10103.
tn	C13	dpmr	41
at	1.311	PROCESSING	
np	65536	lb	1.00
sw	25000.0	wtfile	
fb	13800	proc	f
hs	4	fn	not used
tper	56	math	
psw	4.4		
dl	1.000	werr	
tof	1964.4	wemp	
nt	1034	whs	
ct	216	wut	
alock	n	DISPLAY	
gain	not used	sp	-403.
FLAGS		wp	20913.
il	n	vs	8
in	n	sc	
dp	y	wc	25
hs	nn	hmm	1.7
	is		500.0
	rfl		8881.
	rfp		7743.
	th		1843.
	in		1000.



4-Hydroxymethyl[bis(4-methoxymethoxyphenyl)methylene]cyclohexane (23a).

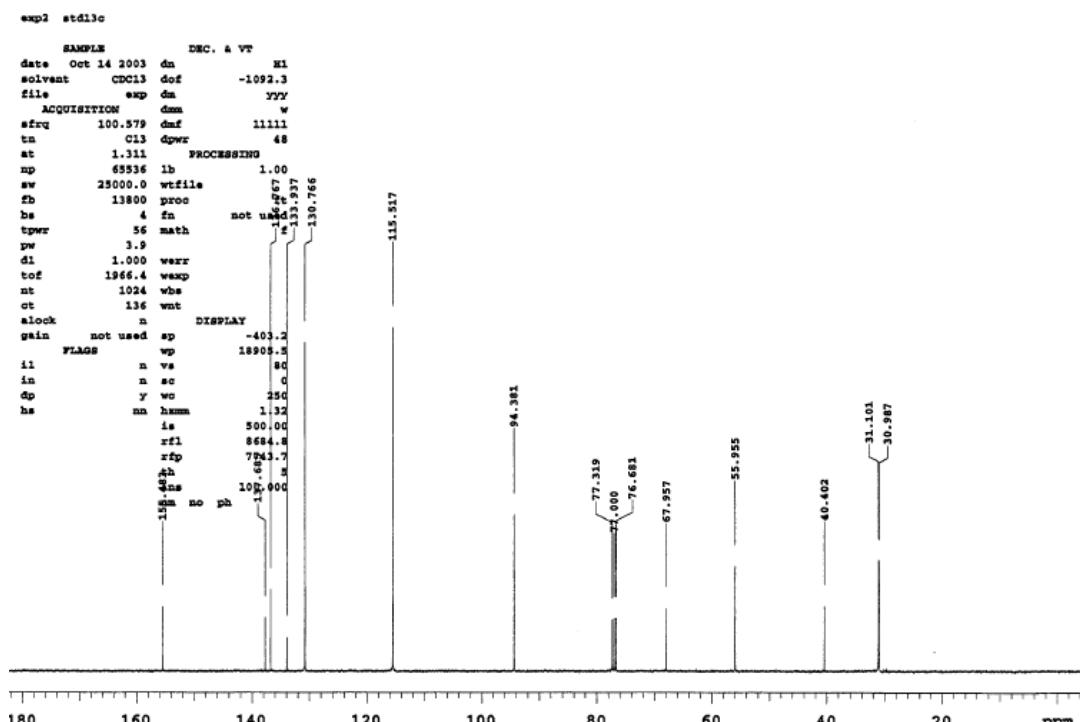
¹H NMR (400 MHz, CDCl₃)

4-Hydroxymethyl[bis(4-methoxymethoxyphenyl)methylene]cyclohexane (23a)



¹³C NMR (100 MHz, CDCl₃)

4-Hydroxymethyl[bis(4-methoxymethoxyphenyl)methylene]cyclohexane (23a)



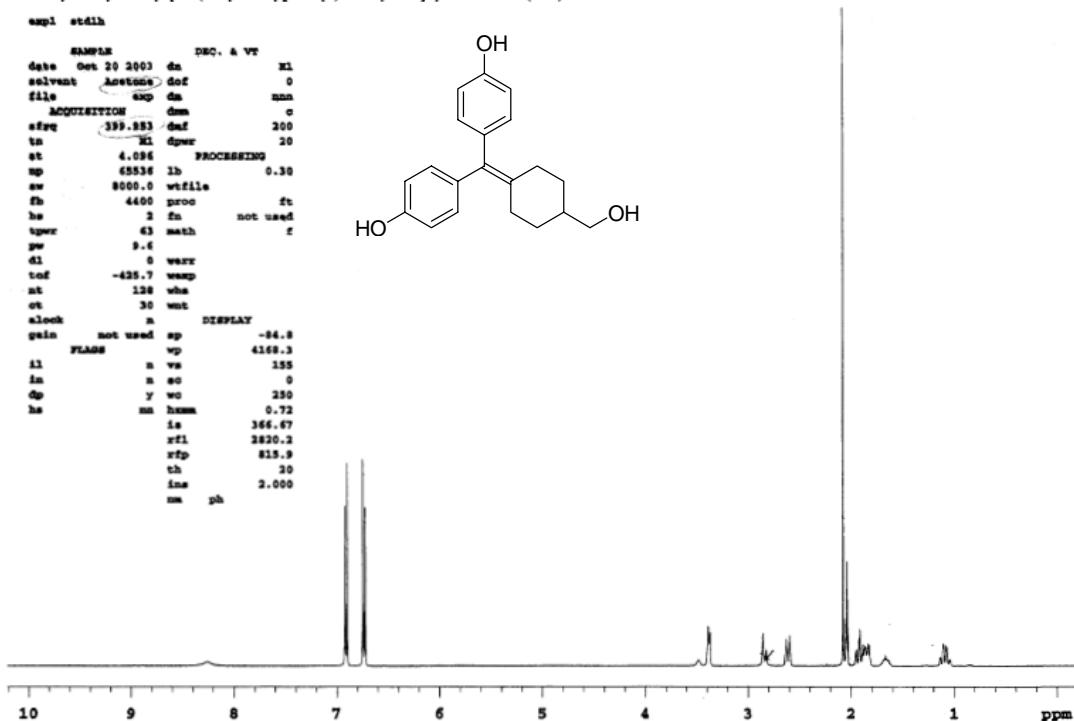
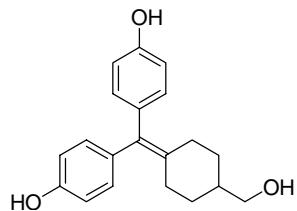
4-Hydroxymethyl[bis(4-hydroxyphenyl)methylene]cyclohexane (24a).

¹H NMR (400 MHz, acetone-*d*₆)

4-Hydroxymethyl[bis(4-hydroxyphenyl)methylene]cyclohexane (24a)

exp1 std1b

```
SAMPLE      DEC. & VT
date Oct 20 2003 dn   H1
solvent Acetone dof   0
file      exp dm   MM
ACQUISITION dm   c
sfreq   399.883 dof   200
tn      1H dppr   10
et      4.096   PROCESSING
sp      65536 1b   0.30
sw      8000.0 wfile
fb      4400 proc   ft
bs      2 fs   not used
tppw   63 math   f
pw      9.6
d1      0 warr
tof    -428.7 wexp
nt      128 wha
ct      30 wnt
alock   n
gain   not used sp   -84.8
FLAGS   wp   4166.3
il      n vs   155
in      n sc   0
dp      y wo   250
hs      nn hmax   0.72
        is   366.67
        r1l   2820.2
        rfp   815.9
        th   20
        ins   2.000
        nn ph
```

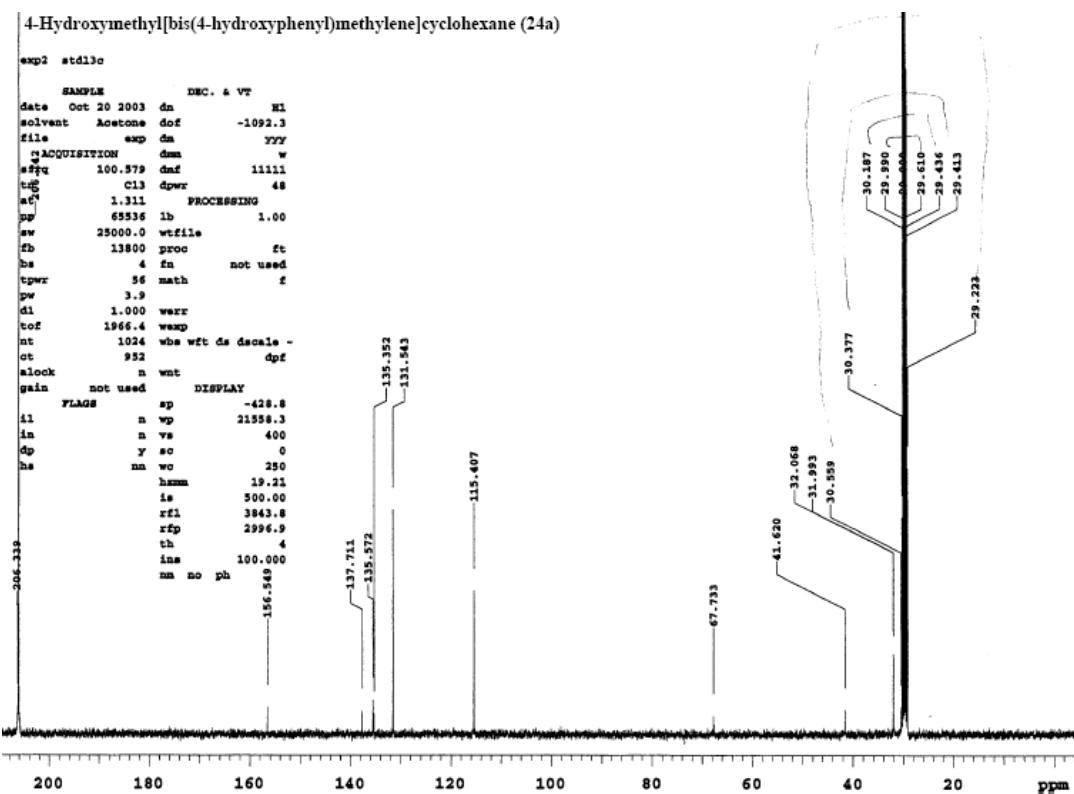


¹³C NMR (100 MHz, acetone-*d*₆)

4-Hydroxymethyl[bis(4-hydroxyphenyl)methylene]cyclohexane (24a)

exp2 std13c

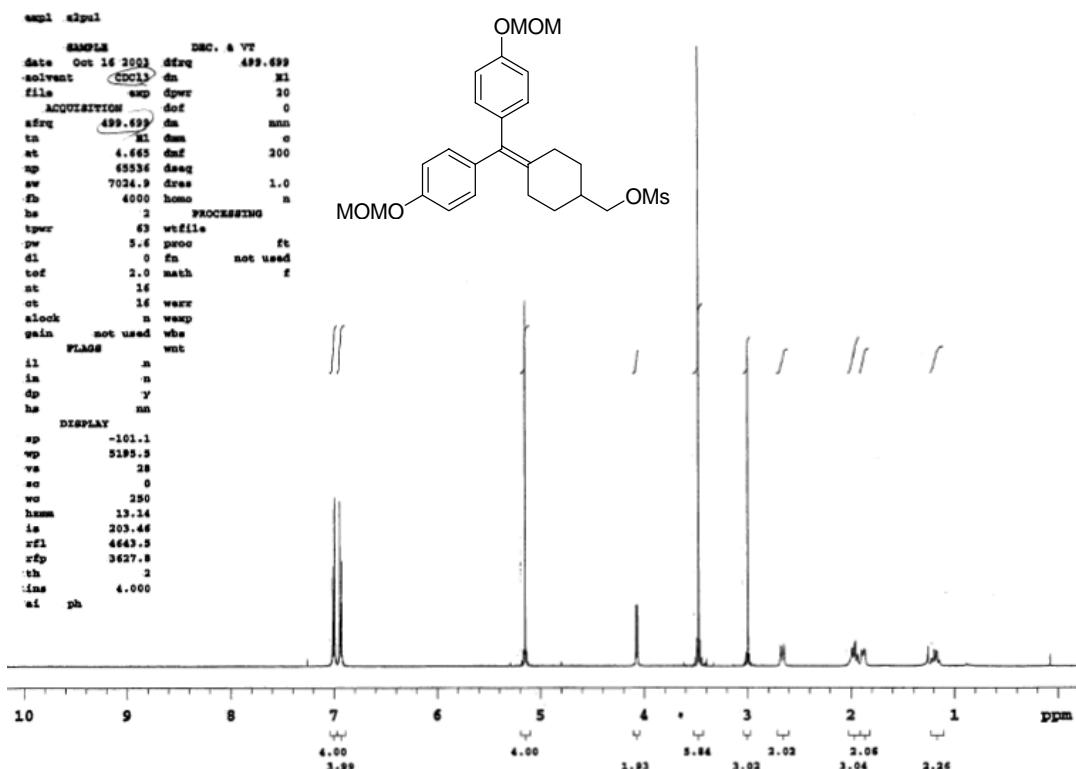
```
SAMPLE      DEC. & VT
date Oct 20 2003 dn   H1
solvent Acetone dof   -1092.3
file      exp dm   YY
ACQUISITION dm   w
sfq   100.579 dof   11111
tnf   C13 dppr   48
et      1.311   PROCESSING
sp      65536 1b   1.00
sw      25000.0 wfile
fb      13800 proc   ft
bs      4 fs   not used
tppw   36 math   f
pw      3.9
d1      1.000 warr
tof    1966.4 wexp
nt      1024 wha wft ds dascale -
ct      952 dppr
alock   n wnt
gain   not used DISPLAY
FLAGS   sp   -428.8
il      n vp   21558.3
in      n vs   400
dp      y sc   0
hs      nn wo   250
        hmax   19.21
        is   500.00
        r1l   3843.8
        rfp   2996.9
        th   4
        ins   100.000
        nn no ph
```



4-Methanesulfonyloxyethyl[bis(4-methoxymethoxyphenyl)methylene]cyclohexane (25a).

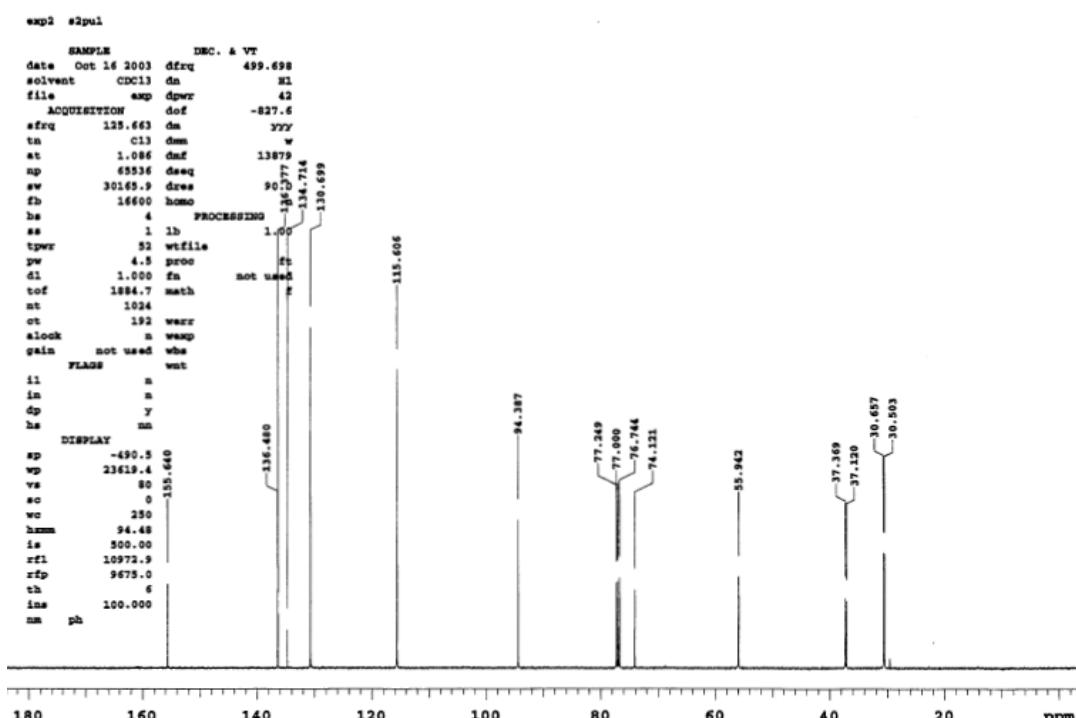
¹H NMR (500 MHz, CDCl₃)

4-Methanesulfonyloxyethyl[bis(4-methoxymethoxyphenyl)methylene]cyclohexane (25a)



¹³C NMR (125 MHz, CDCl₃)

4-Methanesulfonyloxyethyl[bis(4-methoxymethoxyphenyl)methylene]cyclohexane (25a)



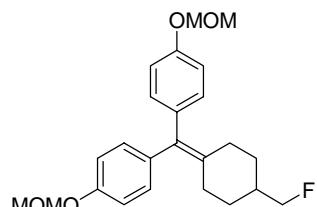
4-Fluoromethyl[bis(4-methoxymethoxyphenyl)methylene]cyclohexane.

¹H NMR (500 MHz, CDCl₃)

4-Fluoromethyl[bis(4-methoxymethoxyphenyl)methylene]cyclohexane

exp1 s2pul

```
SAMPLE          DSC. & VT
date  Oct 29 2003 dfrq    499.699
solvent   CDCl3 dn      XI
file     exp dpwr     20
ACQUISITION dof      0
sfrq    499.699 dn      nnn
tn      XI dmsc      c
at      4.645 dmf     200
np      65536 dseq
sw      7024.9 dres    1.0
fb      4000 homo     n
he      2 PROCESSING
tpwz    63 wtfille
pw      5.6 proc     ft
dl      0 fn        not used
t0f     2.0 math     f
nt      128
et      14 warr
alock   n wexp
gain    not used whs
PLAGE   wnt
ii      n
in      n
dp      Y
hs      nn
DISPLAY
sp      -182.0
wp      5195.5
vs      17
sc      0
wo      250
hwmn   4.93
is      263.05
rfl    4645.3
rfp    3627.8
th      1
ins    6.000
ai      ph
```



¹³C NMR (125 MHz, CDCl₃)

4-Fluoromethyl[bis(4-methoxymethoxyphenyl)methylene]cyclohexane

exp2 s2pul

```
SAMPLE          DSC. & VT
date  Oct 29 2003 dfrq    499.698
solvent   CDCl3 dn      XI
file     exp dpwr     42
ACQUISITION dof      -8270.6-377
sfrq    125.663 dm      XYV-377
tn      C13 dmsc      c
at      1.086 dmf     13879-130-780
np      65536 dseq
sw      30185.9 dres    90.0
fb      16600 homo     n
he      4 PROCESSING
ss      1 lb       1.00
tpwz    52 wtfille
pw      4.5 proc     ft
dl      1.000 fn        not used
t0f     1884.7 math     f
nt      1024
et      172 warr
alock   n wexp
gain    not used whs
PLAGE   wnt
ii      n
in      n
dp      Y
hs      nn
DISPLAY
sp      -510.8
wp      23619.4
vs      140
sc      0
wo      250
hwmn   1.57
is      500.00
rfl    10967.3
rfp    9675.0
th      3
ins    100.000
nm    ph
```



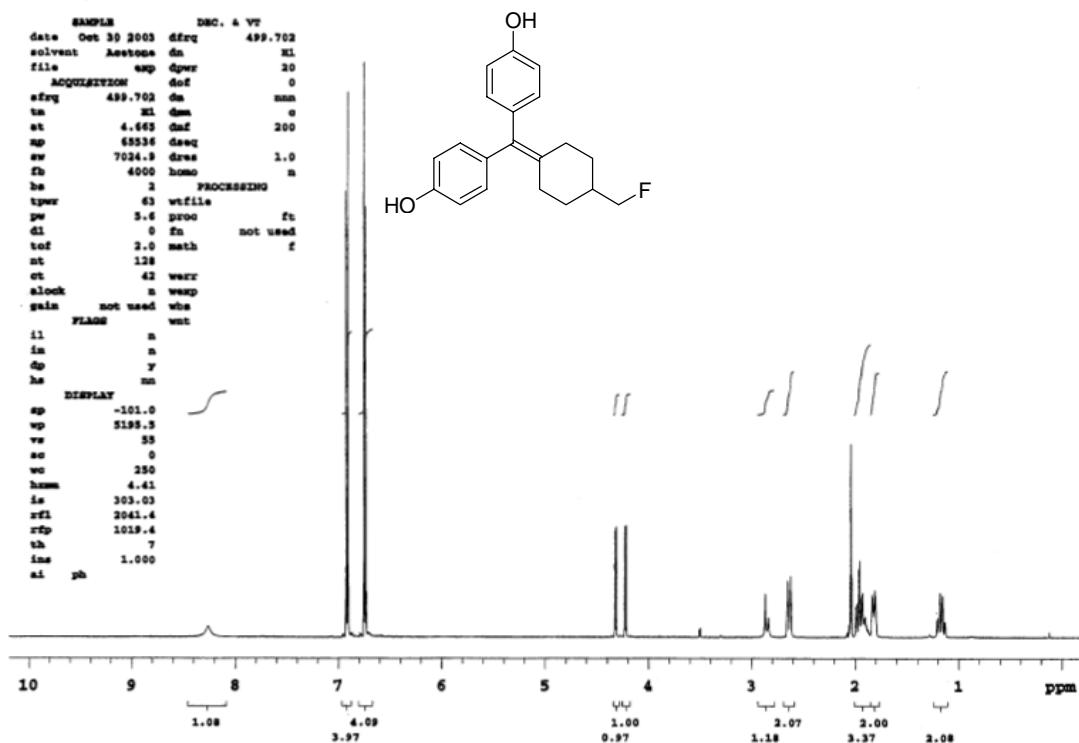
4-Fluoromethyl[bis(4-hydroxyphenyl)methylene]cyclohexane (26a).

¹H NMR (500 MHz, acetone-*d*₆)

4-Fluoromethyl[bis(4-hydroxyphenyl)methylene]cyclohexane (26a)

exp1 s2pul

```
SAMPLE      DSC. & VT
date Oct 30 2003 dfrq 499.702
solvent Acetone dn   H1
file      exp dpwr 20
ACQUISITION dof 0
sfrq 499.702 dm  nnn
tn      H1 dnm  o
st      4.463 dmf  200
sp      65536 dseq
sw      7024.9 dres 1.0
fb      4000 homo  n
hs      3 PROCESSING
tppr 63 wtf file
pw      5.6 proc  ft
d1      0 fm  not used
tof     2.0 math  f
nt      128
et      42 werr
clock   n wexp
gain   not used whs
PLANS  wmt
11      n
in      n
dp      y
hs      nn
DISPLAY
sp      -101.0
wp      5198.5
vs      55
sc      0
wo      250
hmn   4.41
is      303.03
rf1   2041.4
rf2p  1019.4
th      7
ins   1.000
ai      ph
```

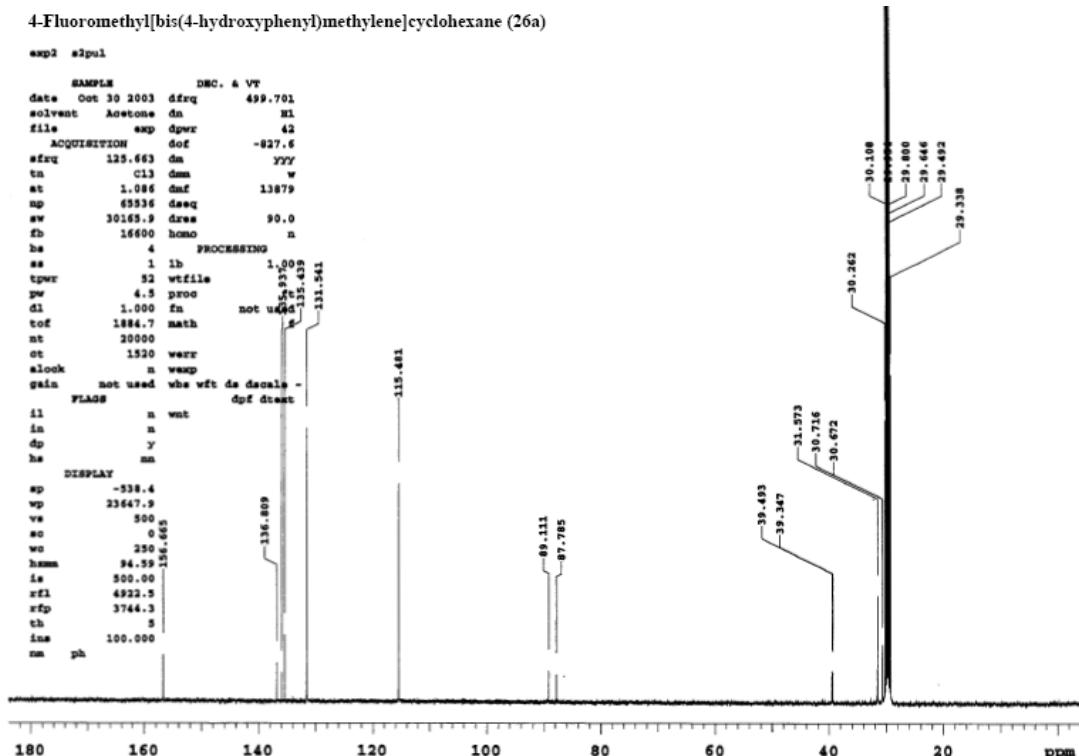


¹³C NMR (125 MHz, acetone-*d*₆)

4-Fluoromethyl[bis(4-hydroxyphenyl)methylene]cyclohexane (26a)

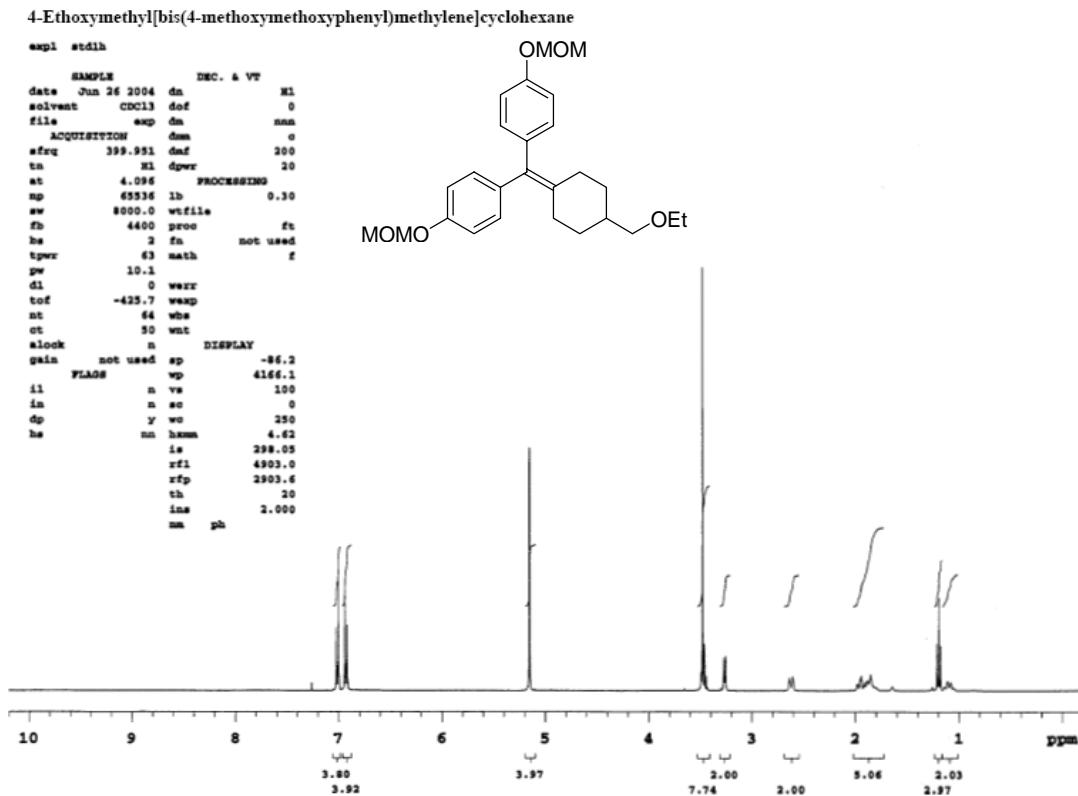
exp1 s2pul

```
SAMPLE      DSC. & VT
date Oct 30 2003 dfrq 499.701
solvent Acetone dn   H1
file      exp dpwr 42
ACQUISITION dof -827.6
sfrq 125.663 dm  YYY
tn      C13 dnm  w
st      1.086 dmf  13879
sp      65536 dseq
sw      30155.9 dres 90.0
fb      16600 homo  n
hs      4 PROCESSING
ss      1 lb      1.000
tppr 63 wtf file
pw      4.5 proc
d1      1.000 fm  not used
tof     1884.7 math
nt      20000
et      1520 werr
clock   n wexp
gain   not used whs wft da dscale -
PLANS  dpf dmaxt
11      n wmt
in      n
dp      y
hs      nn
DISPLAY
sp      -338.4
wp      23647.9
vs      500
sc      0.665
wo      250
hmn   94.59
is      500.00
rf1   4932.5
rf2p  3744.3
th      5
ins   100.000
nm   ph
```

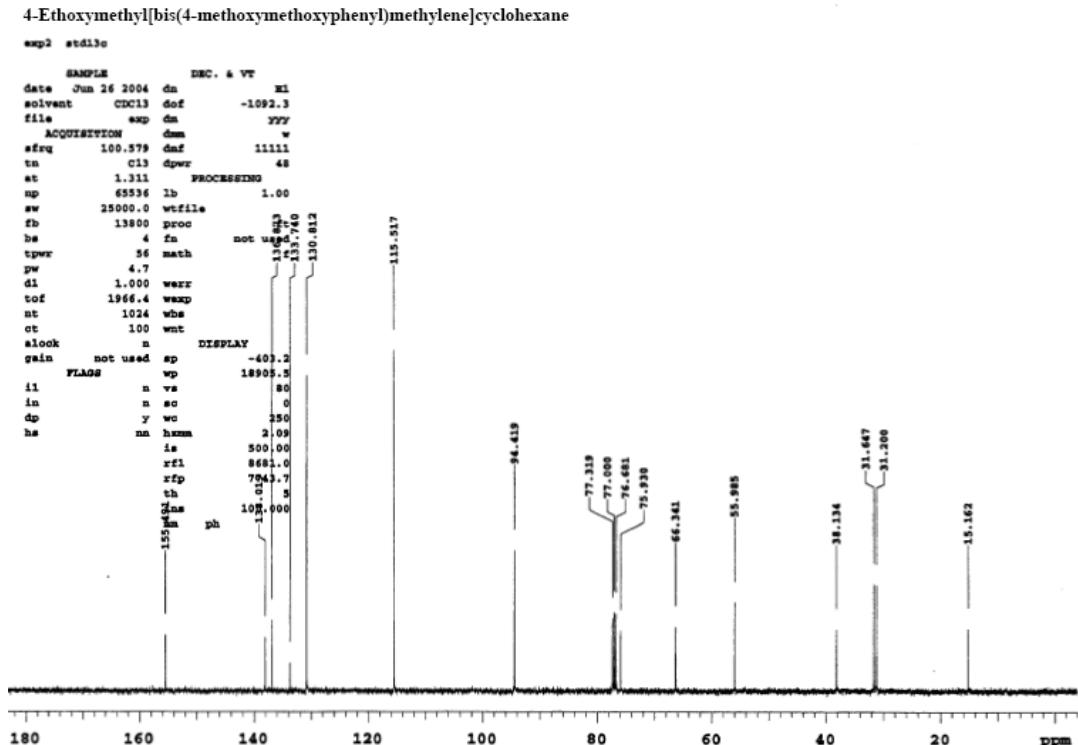


4-Ethoxymethyl[bis(4-methoxymethoxyphenyl)methylene]cyclohexane.

¹H NMR (400 MHz, CDCl₃)



¹³C NMR (100 MHz, CDCl₃)



4-Ethoxymethyl[bis(4-hydroxyphenyl)methylene]cyclohexane (27).

¹H NMR (400 MHz, acetone-*d*₆)

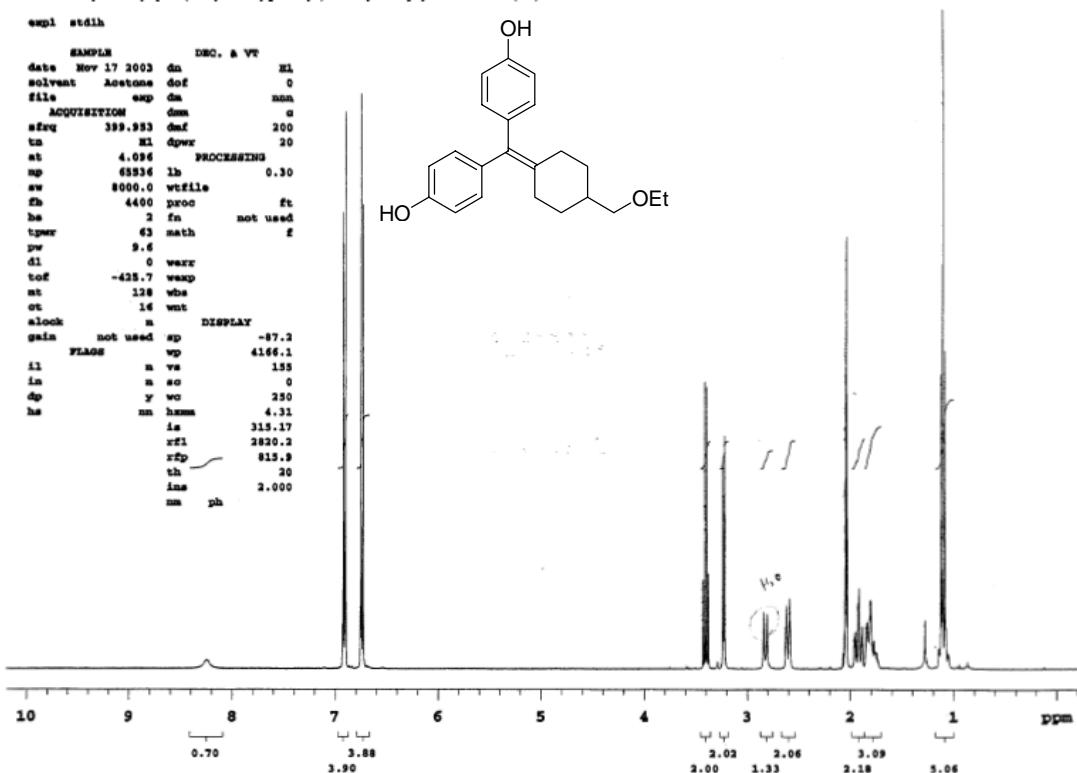
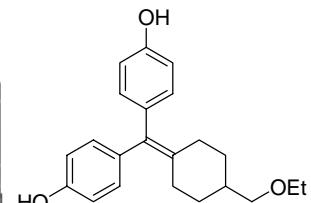
4-Ethoxymethyl[bis(4-hydroxyphenyl)methylene]cyclohexane (27)

exp1 stdinh

```

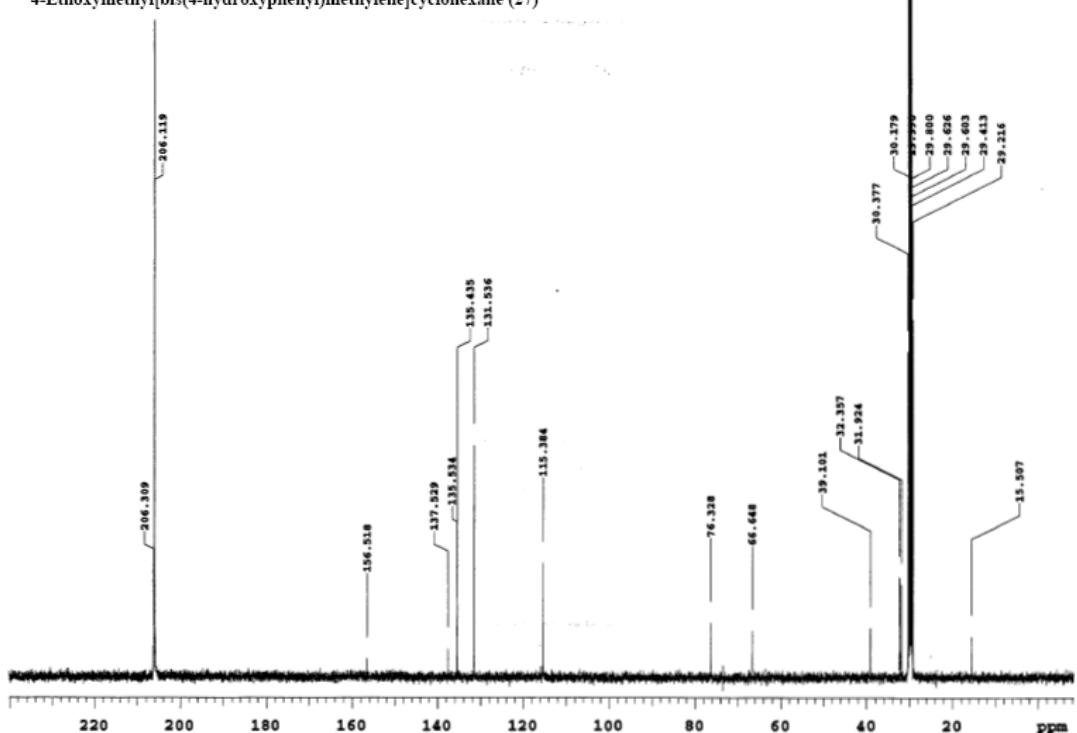
SAMPLE      DSC, & VT
date Nov 17 2003 dn   H1
solvent Acetone-d6 dof   0
file       exp da   nnn
ACQUISITION ddm   s
sfreq  399.933 dppr 200
tn      H1 dppr 20
tr      4.096 PROCESSING
np      65936 lb   0.30
sw     8000.0 wtf file
fb      4400 proc   ft
ns      2 fn   not used
tper    63 math   f
pw      9.6
dl      0 wexp
tof    -425.7 wexp
at      128 whs
ot      16 wnt
clock   n DISPLAY
gain   not used sp   -87.2
PLACES   vp   4166.1
il      n vs   155
in      n ac   0
dp      y vc   250
hs      nn hmax 4.31
        is   315.17
        r1f  2820.2
        r1p  813.9
        th   20
        ins  2.000
        ms   ph

```



¹³C NMR (100 MHz, acetone-*d*₆)

4-Ethoxymethyl[bis(4-hydroxyphenyl)methylene]cyclohexane (27)



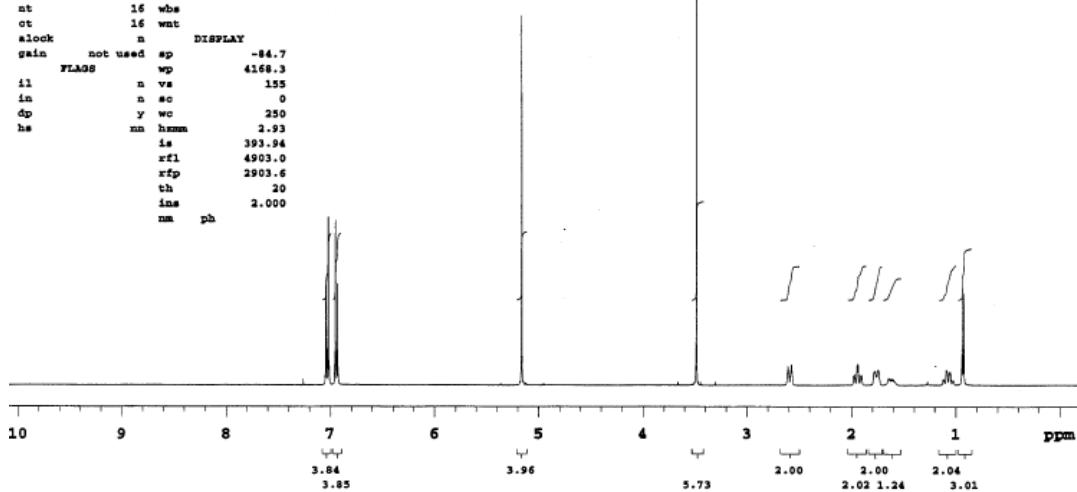
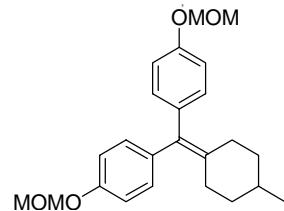
4-Methyl[bis(4-methoxymethoxyphenyl)methylene]cyclohexane.

¹H NMR (400 MHz, CDCl₃)

4-Methyl[bis(4-methoxymethoxyphenyl)methylene]cyclohexane

exp1 std1h

```
SAMPLE      DSC. & VT
date Apr 16 2004 dn   H1
solvent CDCl3 dof   0
file   exp dm   nnn
ACQUISITION ddm   c
sfrq  399.951 dmf  200
tn    65536 lb   0.30
sw   8000.0 wfile
fb    4400 proc   ft
hs    2 fn   not used
tper   63 math   f
pw    9.6
d1    0 warr
tof  -425.7 wexp
nt    16 whs
ot    16 wnt
alock n
gain not used sp   -84.7
FLAGS wp   4168.3
il    n vs   155
in    n sc   0
dp    y wc   250
hs    nn hwm   2.93
        is   393.94
        r1f  4903.0
        rfp  2903.6
        th   20
        ins  2.000
        nm   ph
```

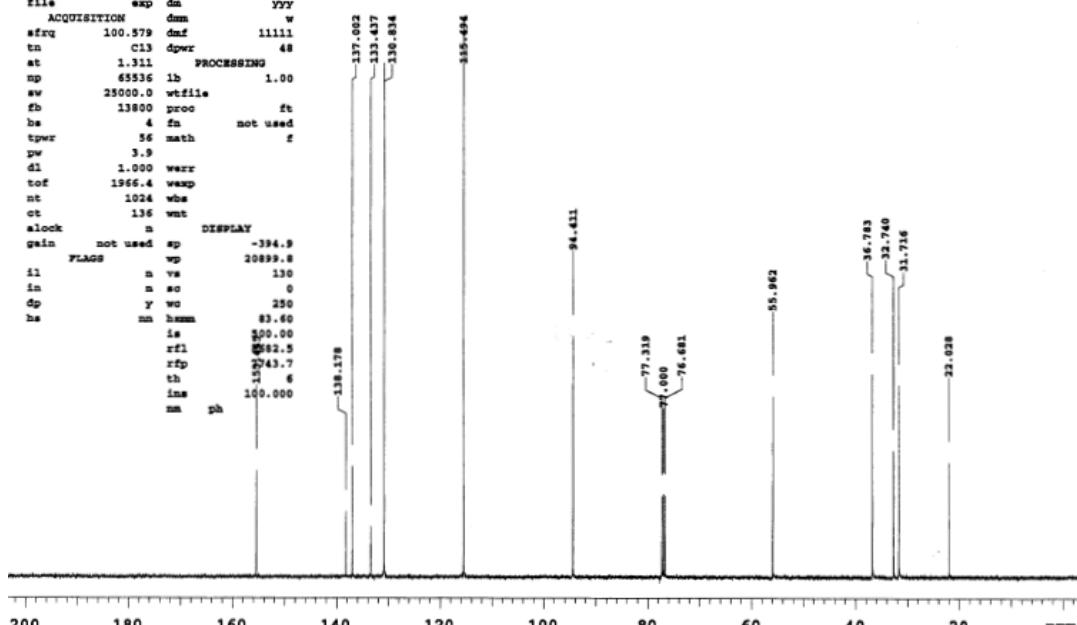


¹³C NMR (100 MHz, CDCl₃)

4-Methyl[bis(4-methoxymethoxyphenyl)methylene]cyclohexane

exp1 std13c

```
SAMPLE      DSC. & VT
date Apr 16 2004 dn   H1
solvent CDCl3 dof  -1092.3
file   exp dm   VVY
ACQUISITION ddm   w
sfrq  100.579 dmf  11111
tn    C13 dpwr  48
at    1.311 PROCESSING
np    65536 lb   1.00
sw   25000.0 wfile
fb    13800 proc   ft
hs    4 fn   not used
tper   56 math   f
pw    3.9
d1    1.000 warr
tof  1956.4 wexp
nt    1024 whs
ot    136 wnt
alock n
gain not used sp   -394.9
FLAGS wp   20899.8
il    n vs   130
in    n sc   0
dp    y wc   250
hs    nn hwm   83.40
        is   500.00
        r1f  482.5
        rfp  243.7
        th   6
        ins  100.000
        nm   ph
```



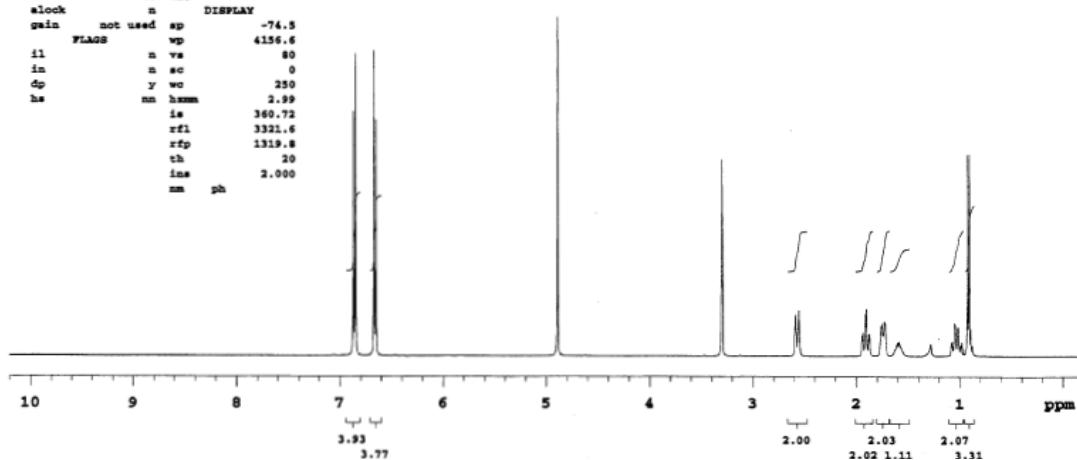
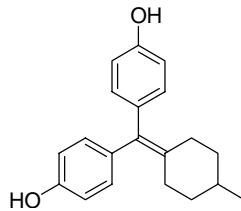
4-Methyl[bis(4-hydroxyphenyl)methylene]cyclohexane (29a).

¹H NMR (400 MHz, methanol-d₄)

4-Methyl[bis(4-hydroxyphenyl)methylene]cyclohexane (29a)

exp1 std1h

```
SAMPLE      DEC. & VT
date Apr 19 2004 dn   H1
solvent CD3OD dof   0
file      exp dm   mmn
ACQUISITION dm   g
sfrq     399.952 daf  200
tn       H1 dppr  20
at       4.096 PROCESSING
np       65536 lb   0.30
sw      8000.0 wtfle
fb       4400 proc  ft
hs       2 fn    not used
tpwr    63 math   f
pw       9.6
di       0 warr
tof     -423.7 wmp
nt       16 wbs
ot       16 wnt
alock   n
gain    not used sp   -74.5
        vp   4156.6
FLAGS   n vs   80
il       n sc   0
in       n wc   250
dp       y hm   2.99
hs       nn hmnm 2.99
        is   360.72
        rfi  3321.6
        rfp  1319.8
        th   20
        ins  2.000
        nm   ph
```

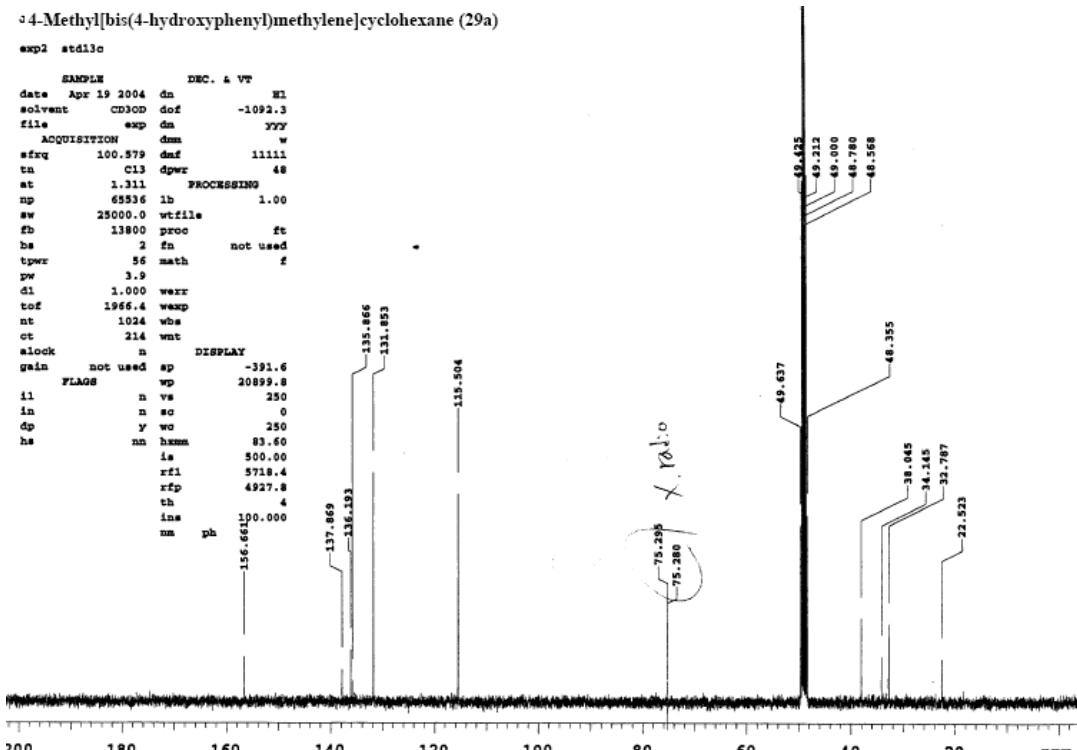


¹³C NMR (100 MHz, methanol-d₄)

4-Methyl[bis(4-hydroxyphenyl)methylene]cyclohexane (29a)

exp2 std1c

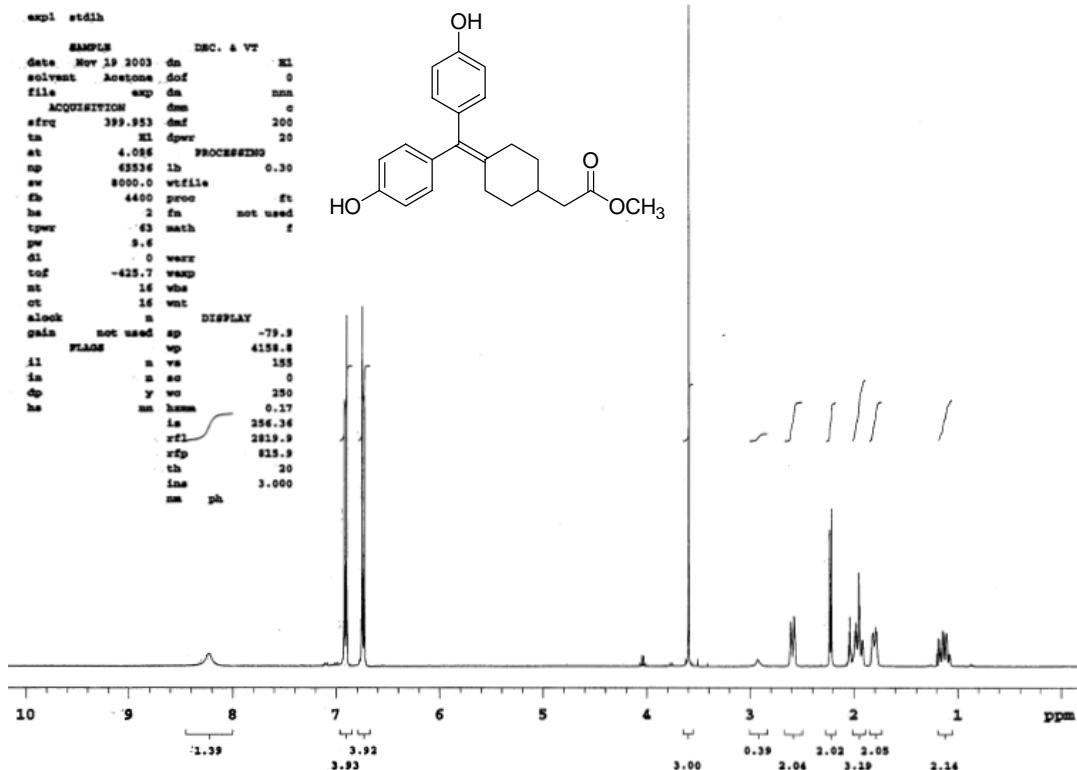
```
SAMPLE      DEC. & VT
date Apr 19 2004 dn   H1
solvent CD3OD dof   -1092.3
file      exp dm   YY
ACQUISITION dm   w
sfrq     100.579 dm   11111
tn       C13 dppr  48
at       1.311 PROCESSING
np       65536 lb   1.00
sw      25000.0 wtfle
fb       13800 proc  ft
hs       2 fn    not used
tpwr    56 math   f
pw       3.9
di       1.000 warr
tof     1966.4 wmp
nt       1024 wbs
ot       214 wnt
alock   n
gain    not used sp   -391.6
        vp   20899.8
FLAGS   n vs   250
il       n sc   0
in       n wc   250
dp       y hm   83.60
hs       nn hmnm 83.60
        is   500.00
        rfi  5718.4
        rfp  4927.8
        th   4
        ins  100.000
        nm   ph
```



Methyl {4-[Bis(4-hydroxyphenyl)methylene]cyclohexyl}acetate (21b).

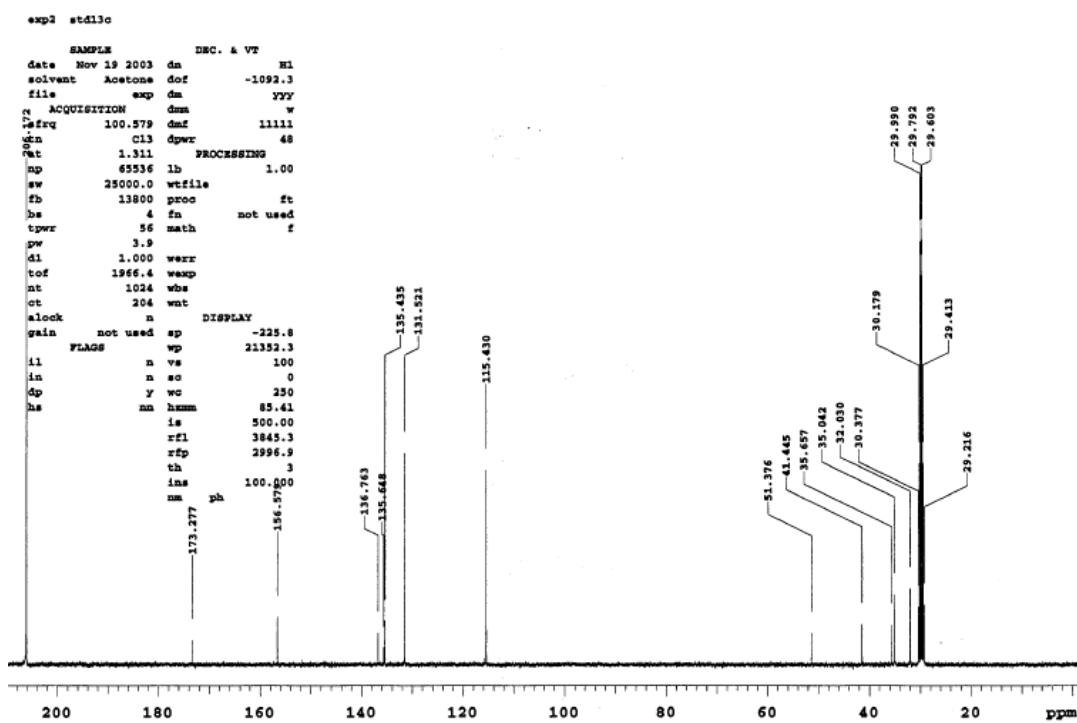
¹H NMR (400 MHz, acetone-*d*₆)

Methyl {4-[Bis(4-hydroxyphenyl)methylene]cyclohexyl}acetate (21b)



¹³C NMR (100 MHz, acetone-*d*₆)

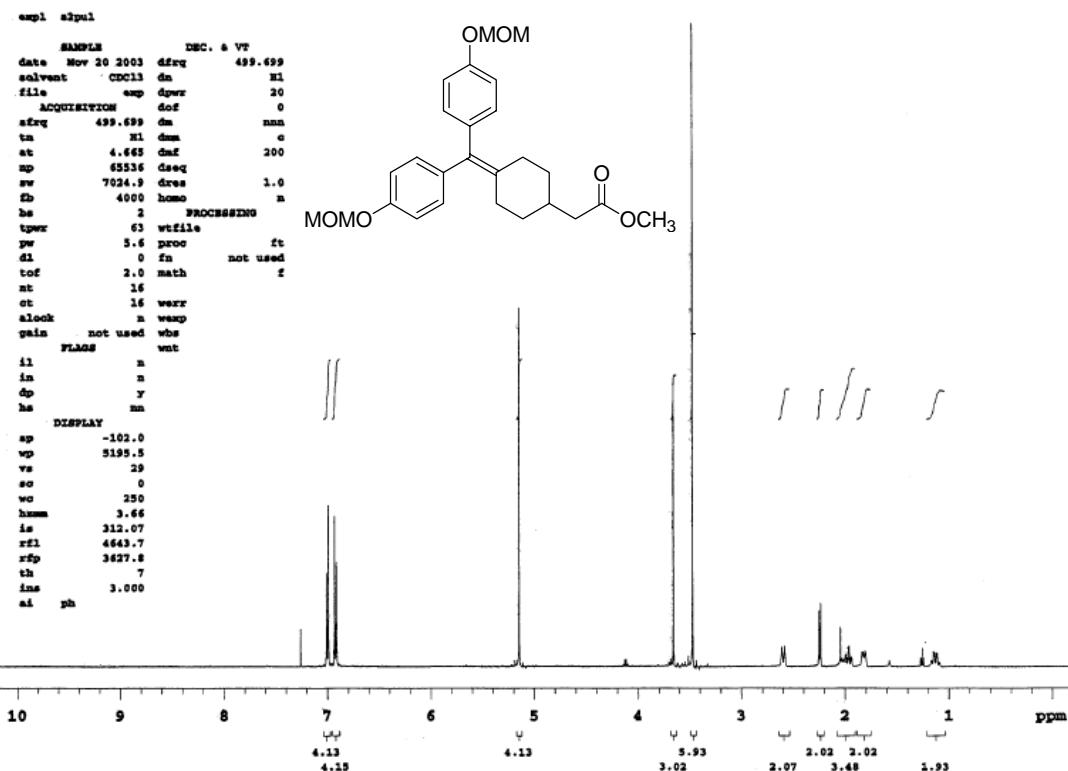
Methyl {4-[Bis(4-hydroxyphenyl)methylene]cyclohexyl}acetate (21b)



Methyl {4-[Bis(4-methoxymethoxyphenyl)methylene]cyclohexyl}acetate (22b).

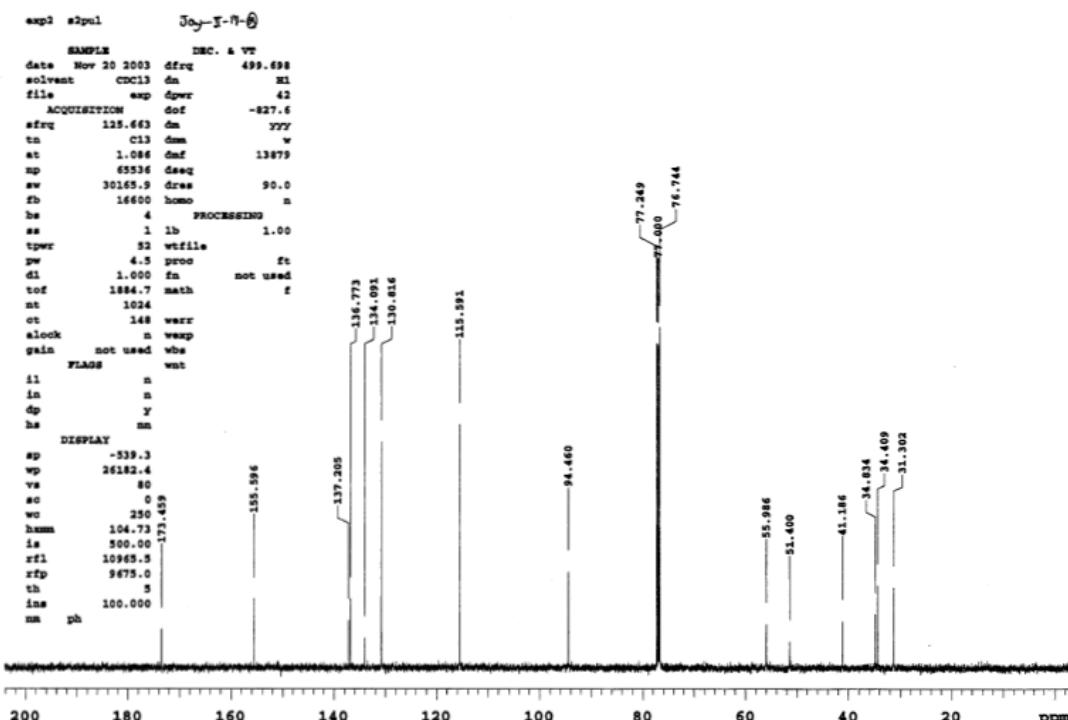
¹H NMR (500 MHz, CDCl₃)

Methyl {4-[Bis(4-methoxymethoxyphenyl)methylene]cyclohexyl}acetate (22b)



¹³C NMR (125 MHz, CDCl₃)

Methyl {4-[Bis(4-methoxymethoxyphenyl)methylene]cyclohexyl}acetate (22b)



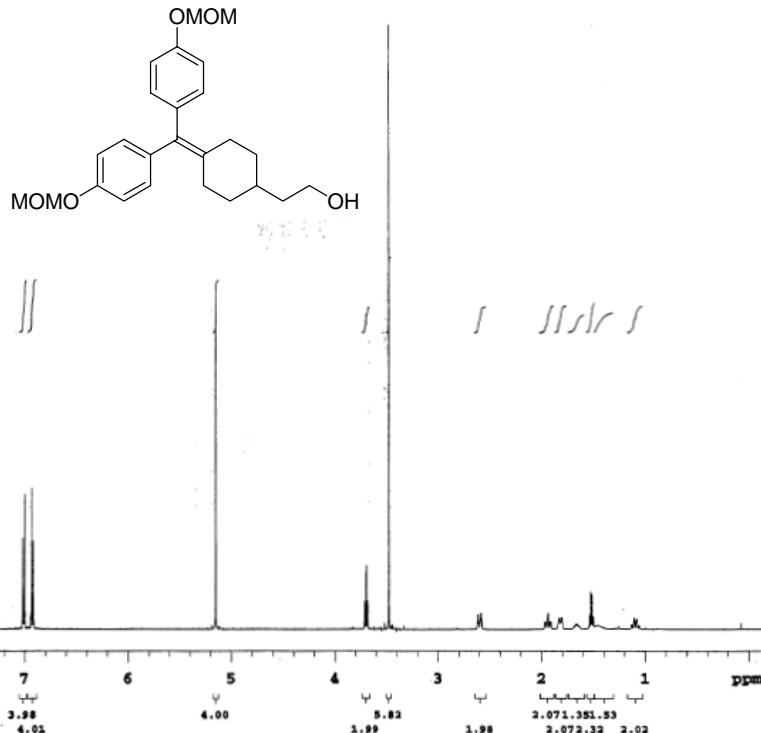
4-(2-Hydroxyethyl)[bis(4-methoxymethoxyphenyl)methylene]cyclohexane (23b).

¹H NMR (500 MHz, CDCl₃)

4-(2-Hydroxyethyl)[bis(4-methoxymethoxyphenyl)methylene]cyclohexane (23b)

exp1 s2pul

```
SAMPLE      DSC. & VT
date Nov 22 2003 dfrq 499.699
solvent CDCl3 dn   H1
file      exp dpwr 20
ACQUISITION dof 0
sfrq 499.699 da   nnn
tn       1.11 dnm   o
at       4.465 dmf  200
sp       65336 dsw
sw       7024.9 dres 1.0
fb       4000 homo   n
hs       2
PROCCESSING
tppw    63 wfile
pr       5.6 proc   ft
d1       0 fn     not used
tof      2.0 math   f
at       16
ct       16 werr
alock   n wemp
gain    not used whs
FLAGS   n wft
DISPLAY
sp       -101.1
wp       5199.5
vs       28
sc       0
wd       250
hmax   4.01
is       341.13
rfl    -4643.5
rfp    -3627.6
th       2
ins    -4.000
ai     ph
```

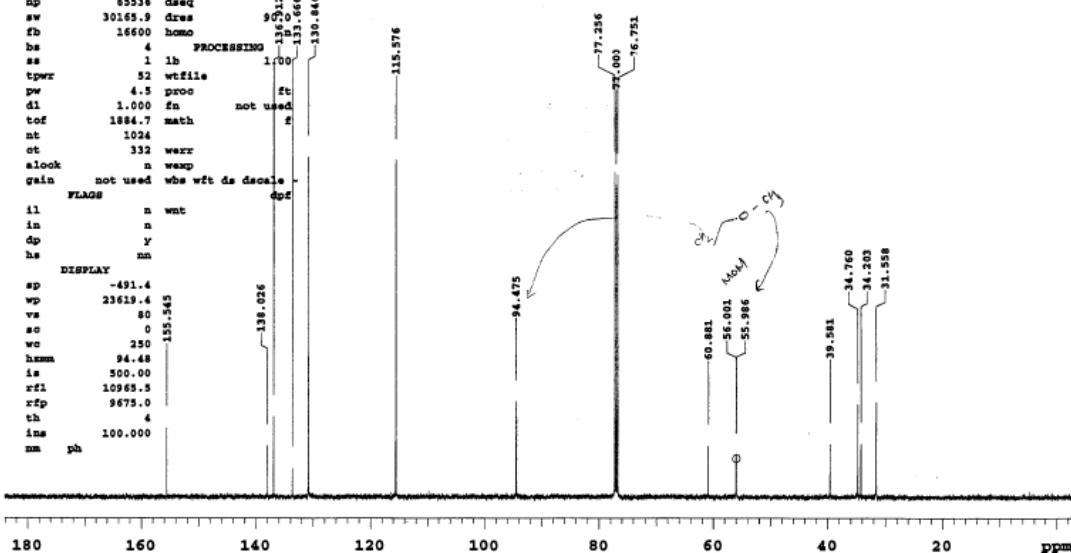


¹³C NMR (125 MHz, CDCl₃)

4-(2-Hydroxyethyl)[bis(4-methoxymethoxyphenyl)methylene]cyclohexane (23b)

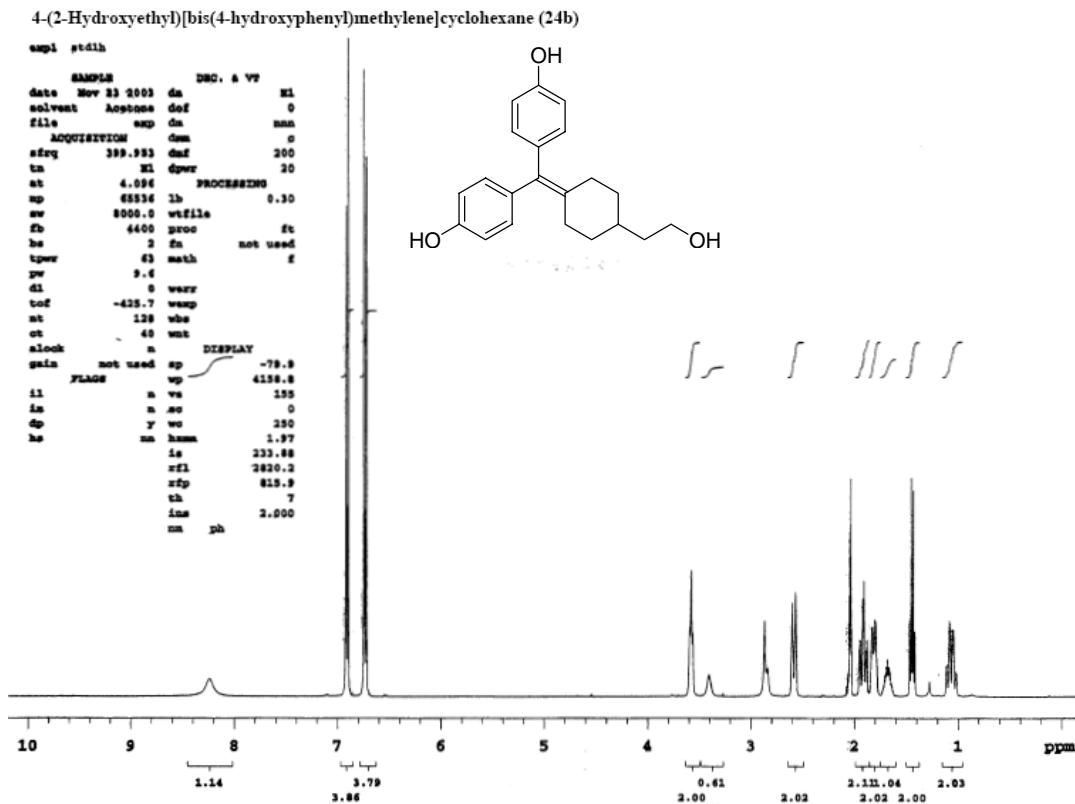
exp2 s2pul

```
SAMPLE      DSC. & VT
date Nov 22 2003 dfrq 499.698
solvent CDCl3 dn   H1
file      exp dpwr 42
ACQUISITION dof -827.6
sfrq 125.663 dm   vvv
tn       C13 dnm   w
at       1.086 dmf  13879
sp       65336 dsw
sw       30165.9 dres 90.0,66.6
fb       16600 homo   113,110,86.6
hs       4
PROCCESSING
ss       1 lb    1.000
tppw    52 wfile
pr       4.5 proc   ft
d1       1.000 fn     not used
tof      1884.7 math   f
nt       1024
ct       332 werr
alock   n wemp
gain    not used whs wft dscale -
FLAGS   n wft
DISPLAY
sp       -491.4
wp       23619.4 45
vs       80 54
sc       0 155
wd       250
hmax   94.48
is       500.00
rfl    10965.5
rfp    9675.0
th       4
ins    100.000
nm     ph
```

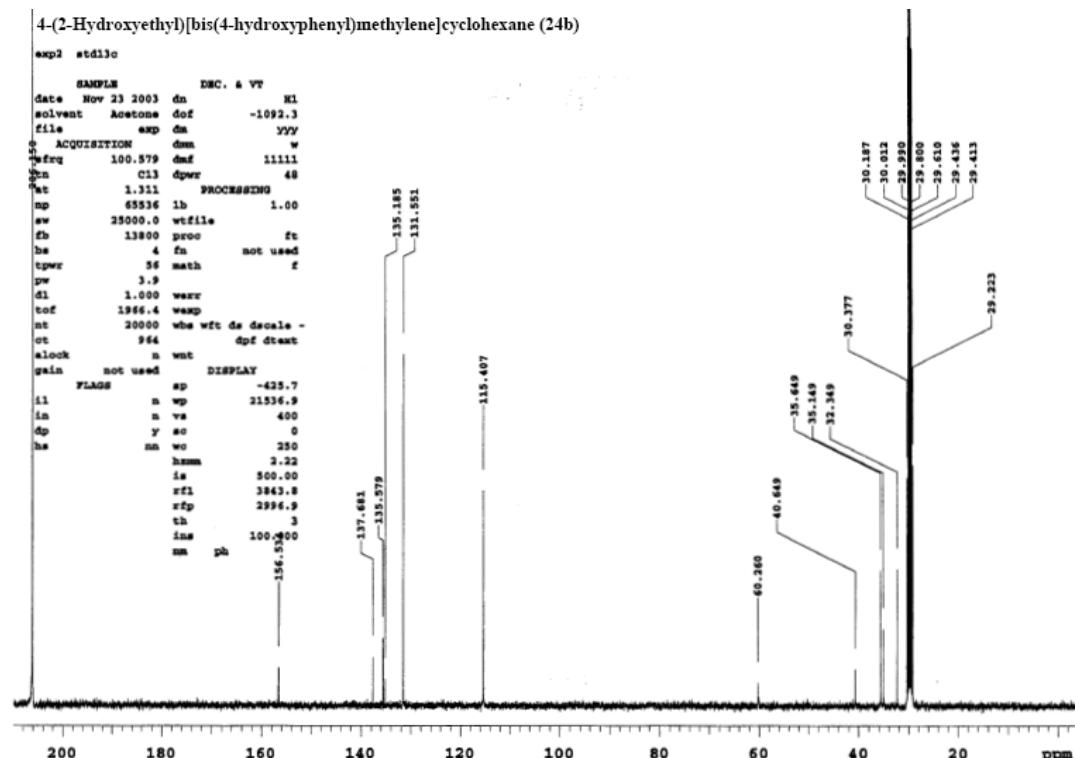


4-(2-Hydroxyethyl)[bis(4-hydroxyphenyl)methylene]cyclohexane (24b).

¹H NMR (400 MHz, acetone-*d*₆)

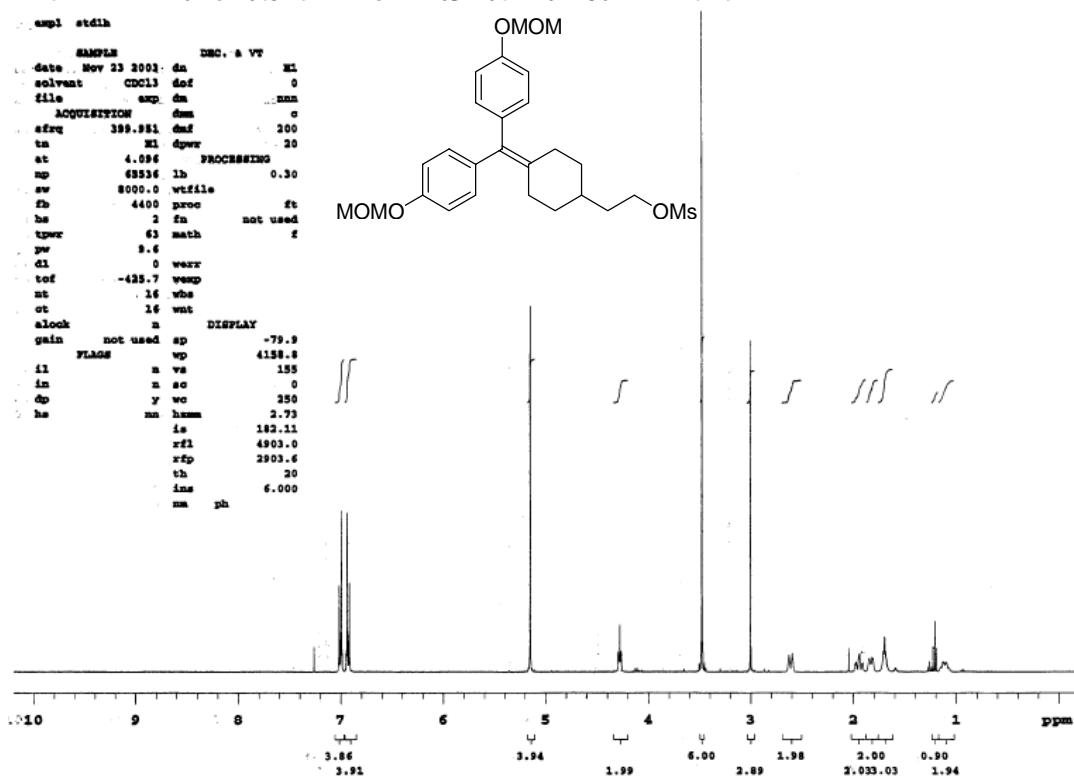


¹³C NMR (100 MHz, acetone-*d*₆)



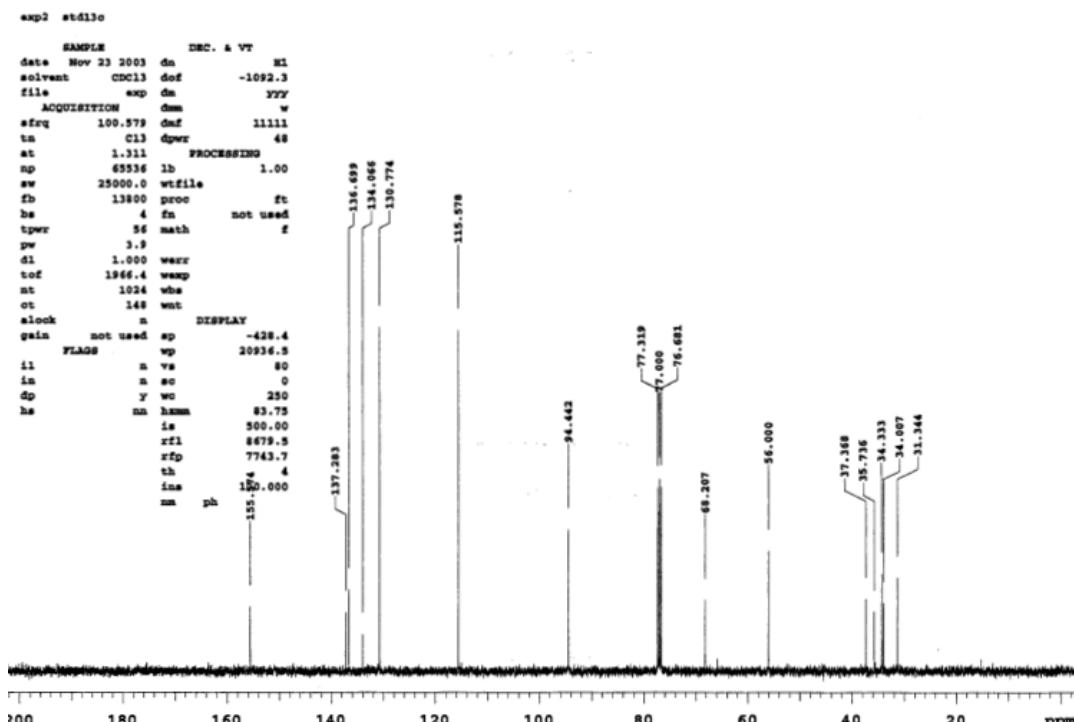
**4-(2-Methanesulfonyloxyethyl)[bis(4-methoxymethoxyphenyl)methylene]cyclohexane
(25b). ^1H NMR (400 MHz, CDCl_3)**

4-(2-Methanesulfonyloxyethyl)[bis(4-methoxymethoxyphenyl)methylene]cyclohexane (25b)



^{13}C NMR (100 MHz, CDCl_3)

4-(2-Methanesulfonyloxyethyl)[bis(4-methoxymethoxyphenyl)methylene]cyclohexane (25b)



4-(2-Fluoroethyl)[bis(4-methoxymethoxyphenyl)methylene]cyclohexane.

¹H NMR (500 MHz, CDCl₃)

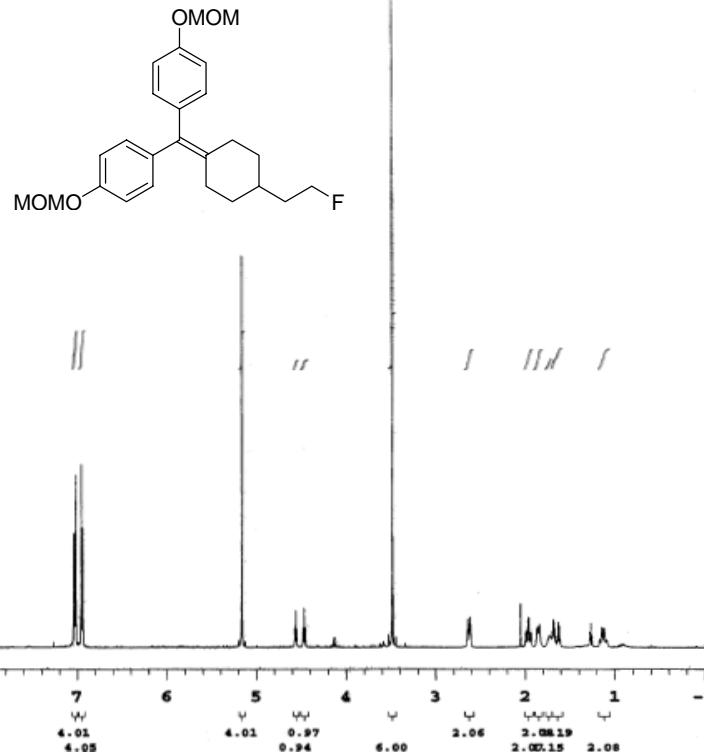
4-(2-Fluoroethyl)[bis(4-methoxymethoxyphenyl)methylene]cyclohexane

expt1 s2pul

```

SAMPLE      DSC. & VP
date Nov 24 2003 dfrq    499.698
solvent   CDCl3  dn      H1
file      exp  dpwr     20
ACQUISITION dof      0
sfrq     499.698 dn      nnn
tn        H1  dswm     c
at       4.465 dswf    200
wp      65536  dswq
sw      7024.9  dres     1.0
fb      40000 homo
he         2  PROCESSING
tpwr     63 wtfile
pw      5.6  proc
st
dl      0  fn      not used
tof      2.0  math
nt      16
ot      16  warr
alock    n  wscp
gain    not used wbs
FLAGS    wnt
il      n
in      n
dp      y
hs      nn
DISPLAY
sp      -1013.5
wp      7024.9
vs      42
sc      0
wo      250
hmnw    4.59
is      184.52
rf1     4643.3
rf2p    3627.8
th      7
ins     6.000
ai      ph

```



¹³C NMR (125 MHz, CDCl₃)

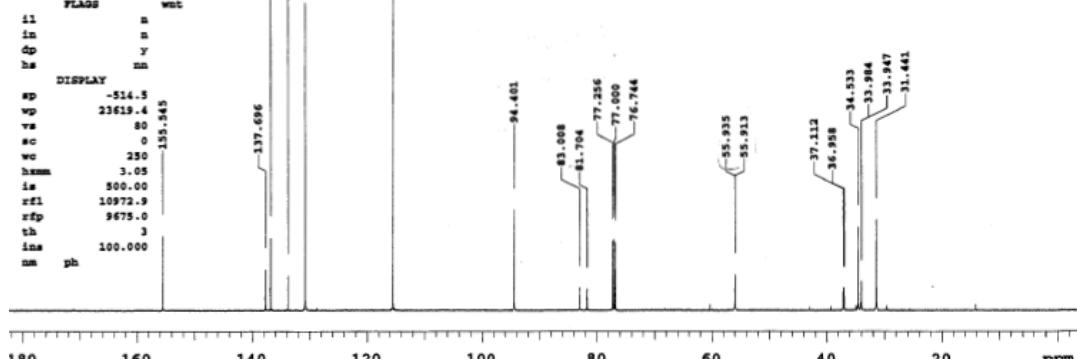
4-(2-Fluoroethyl)[bis(4-methoxymethoxyphenyl)methylene]cyclohexane

amp2 s2pul

```

SAMPLE      DSC. & VP
date Nov 24 2003 dfrq    499.698
solvent   CDCl3  dn      H1
file      exp  dpwr     42
ACQUISITION dof      -827.6
sfrq     125.663 dm      VVY
tn        C13  dswm     w
at       1.086 dswf    13879
wp      65536  dswq
sw      30165.9  dres    90.0
fb      16600  homo
he         4  PROCESSING
ss      1  1b      146.0-79.8
tpwr     52 wtfile
pw      4.5  proc
st
dl      1.0000 fn      not used
tof      1884.7  math
nt      1024
ot      156  warr
alock    n  wscp
gain    not used wbs
FLAGS    wnt
il      n
in      n
dp      y
hs      nn
DISPLAY
sp      -514.5
wp      23619.4 345
vs      80
sc      0 155
wo      250
hmnw    3.05
is      500.00
rf1     10972.9
rf2p    9675.0
th      3
ins     100.000
nm      ph

```



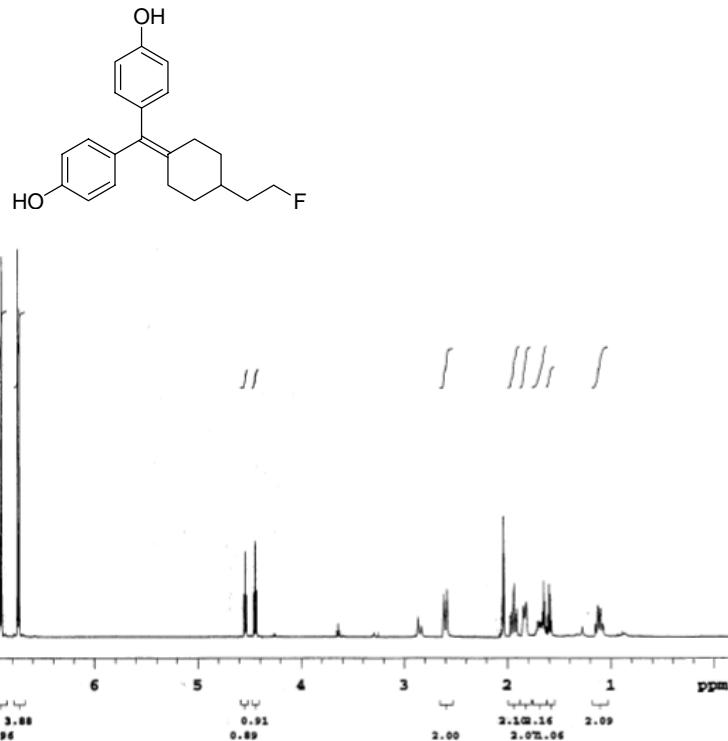
4-(2-Fluoroethyl)[bis(4-hydroxyphenyl)methylene]cyclohexane (26b).

¹H NMR (500 MHz, acetone-*d*₆)

4-(2-Fluoroethyl)[bis(4-hydroxyphenyl)methylene]cyclohexane (26b)

exp1 s2pul

```
SAMPLE      DSC. & VT
date Nov 25 2003 dfrq 499.702
solvent Acetone dn   NL
file          exp dpwr 20
ACQUISITION dof 0
sfrq 499.702 dm  nnn
tn      NL dnm  c
at     4.665 dmf  200
sp     65536 dseq
sw    7024.9 dres  1.0
fb    4000 homo  n
hs      2 PROCESSING
tppw 63 wtfille ft
pw    5.6 proc  ft
di    0 fn  not used
tof   2.0 math  f
nt    128
ct    20 warr
alock  n wexp
gain  not used wbs
FLAGS  wnt
DISPLAY
sp    -101.9
wp    5195.5
vs    40
sc    0
wo    250
hmn  0.78
is    281.04
rf1   2041.0
rf2p  1019.4
th    7
ins   2.000
ai    -ph
```

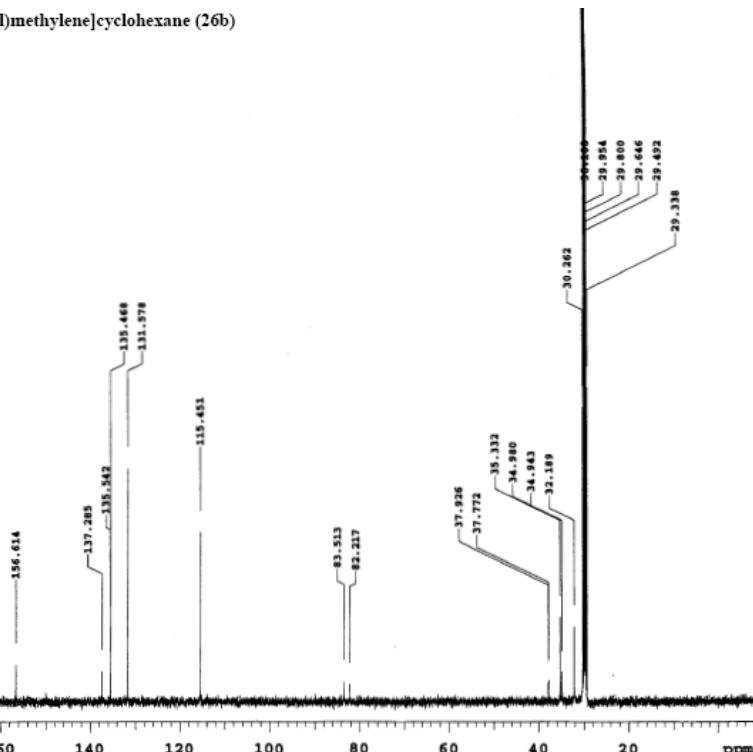


¹³C NMR (125 MHz, acetone-*d*₆)

4-(2-Fluoroethyl)[bis(4-hydroxyphenyl)methylene]cyclohexane (26b)

exp2 s2pul

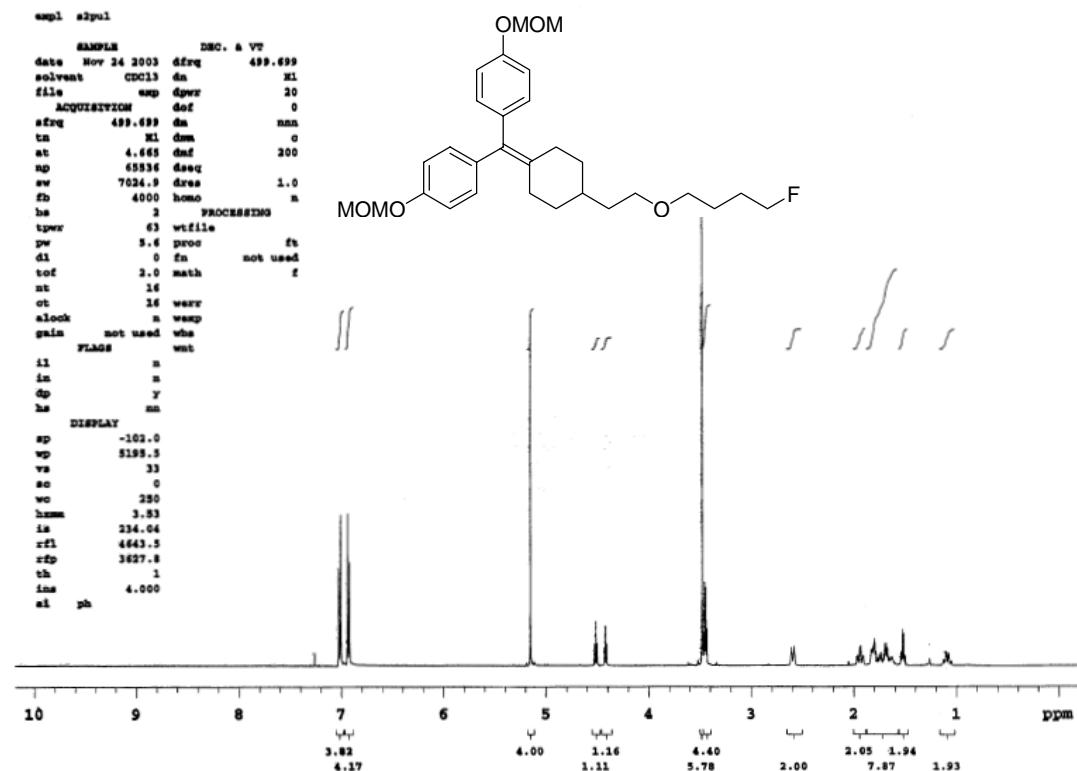
```
SAMPLE      DSC. & VT
date Nov 25 2003 dfrq 499.701
solvent Acetone dn   NL
file          exp dpwr 42
ACQUISITION dof -827.6
sfrq 100.663 dm  VVV
tn      NL C13 dnm  v
at     7.086 dmf  13879
sp     65536 dseq
sw    30165.9 dres  90.0
fb    16600 homo  n
hs      4 PROCESSING
ss    1 lb  1.00
tppw 52 wtfille
pw    4.5 proc  ft
di    1.000 fn  not used
tof   1884.7 math  f
nt    10000
ct    336 warr
alock  n wexp  svrf(nL)
gain  not used wbs wft da dscals -
FLAGS  ddp dtsmt
il    n wnt  wft('aqr')
in    n
dp    Y
hs    nn
DISPLAY
sp    -1178.2
wp    30143.9
vs    300
sc    0
wo    250
hmn  11.30
is    100.00
rf1   922.5
rf2p  744.3
th    3
ins   100.000
nm  ph
```



4-[2-(4-Fluorobutoxy)ethyl][bis(4-methoxymethoxyphenyl)methylene]cyclohexane.

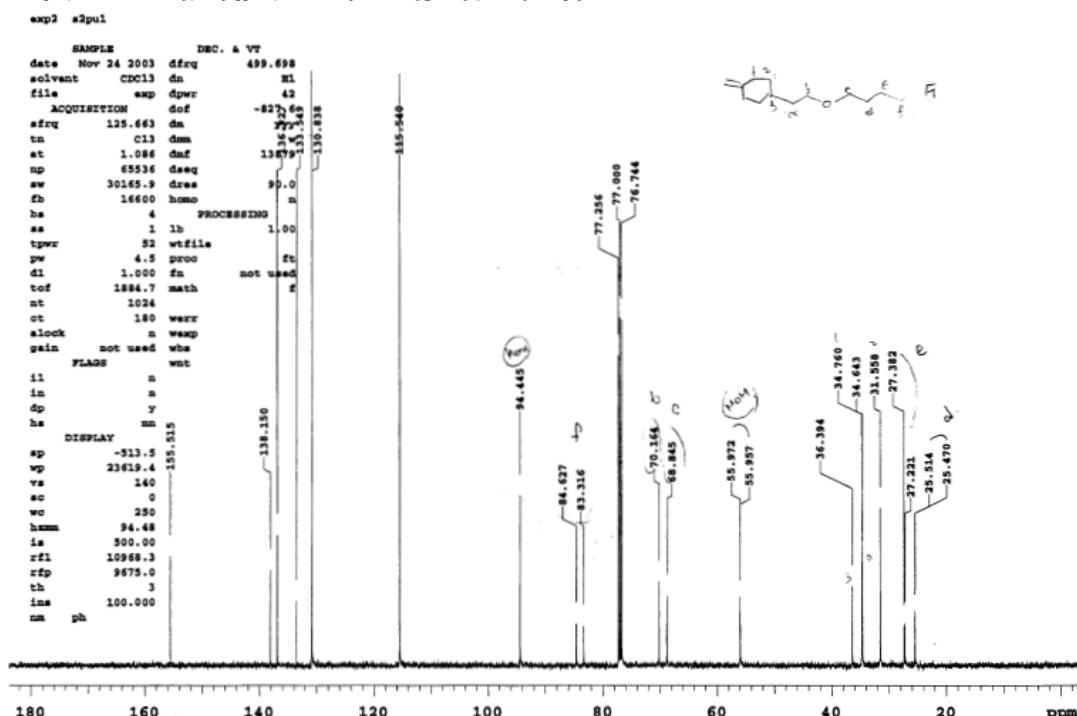
¹H NMR (500 MHz, CDCl₃)

4-[2-(4-Fluorobutoxy)ethyl][bis(4-methoxymethoxyphenyl)methylene]cyclohexane



¹³C NMR (125 MHz, CDCl₃)

4-[2-(4-Fluorobutoxy)ethyl][bis(4-methoxymethoxyphenyl)methylene]cyclohexane



4-[2-(4-Fluorobutoxy)ethyl][bis(4-hydroxyphenyl)methylene]cyclohexane (28).

¹H NMR (500 MHz, acetone-*d*₆)

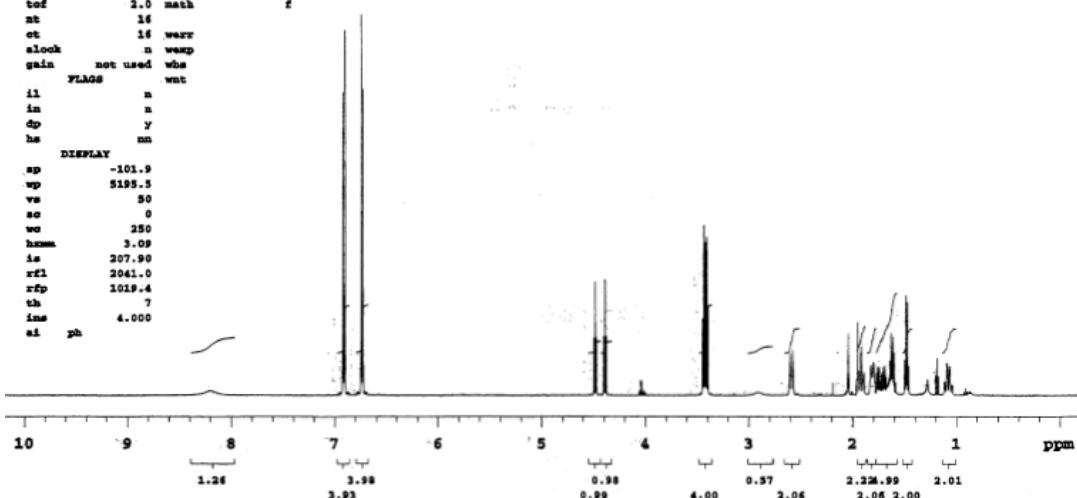
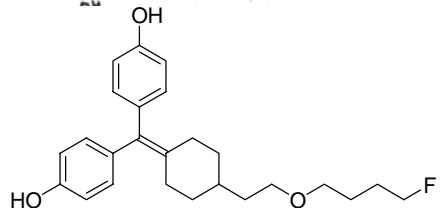
4-[2-(4-Fluorobutoxy)ethyl][bis(4-hydroxyphenyl)methylene]cyclohexane (28)

expi signi

```

SAMPLE          DEC. & VT
date Nov 26 2003 dfqrq    499.700
solvent Acetone dn      .ml
file      exp dpwr     20
ACQUISITION dof      0
sfqrq   499.702 dm      mmr
tn      EL dnm      0
st      4.665 dmf     100
np      63336 dsqf
sw      7024.9 dres    1.0
fb      4000 homo    n
ba      2      PROCESSING
tpwr   63 wtfille
pw      5.6 proc    ft
dl      0 fn      not used
tcf      2.0 math    f
nt      16
ct      16 warr
clock   n wexp
gain   not used whs
YFLAG   wnt
il      n
in      n
dp      y
hs      nm
DISPLAY
sp      -101.9
wp      5195.5
vs      30
se      0
wo      250
lmaxm   3.08
is      207.90
rf1     2041.0
rfp     1029.4
tb      7
inc     4.000

```

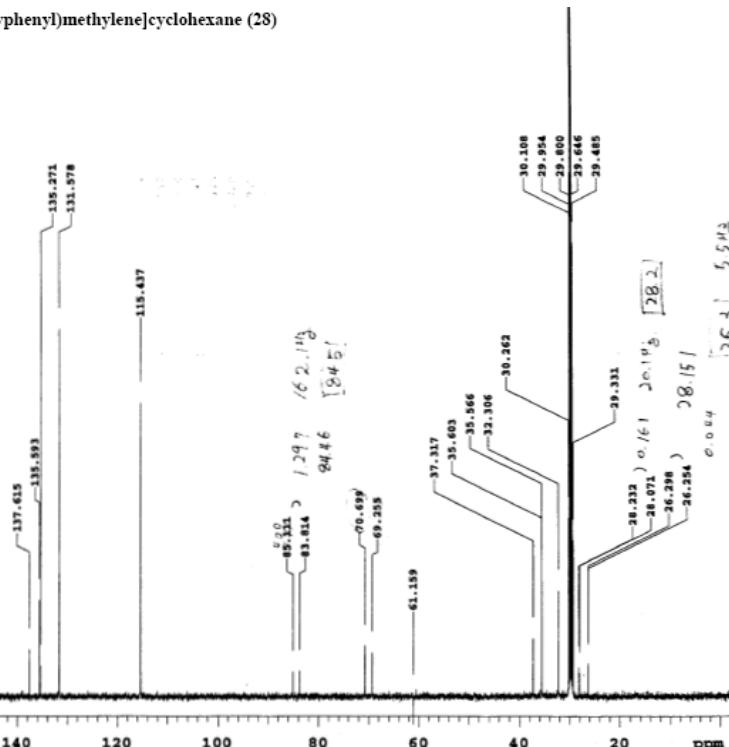


¹³C NMR (125 MHz, acetone-*d*₆)

4-[2-(4-Fluorobutoxy)ethyl]bis(4-hydroxyphenyl)methylene)cyclohexane (28)

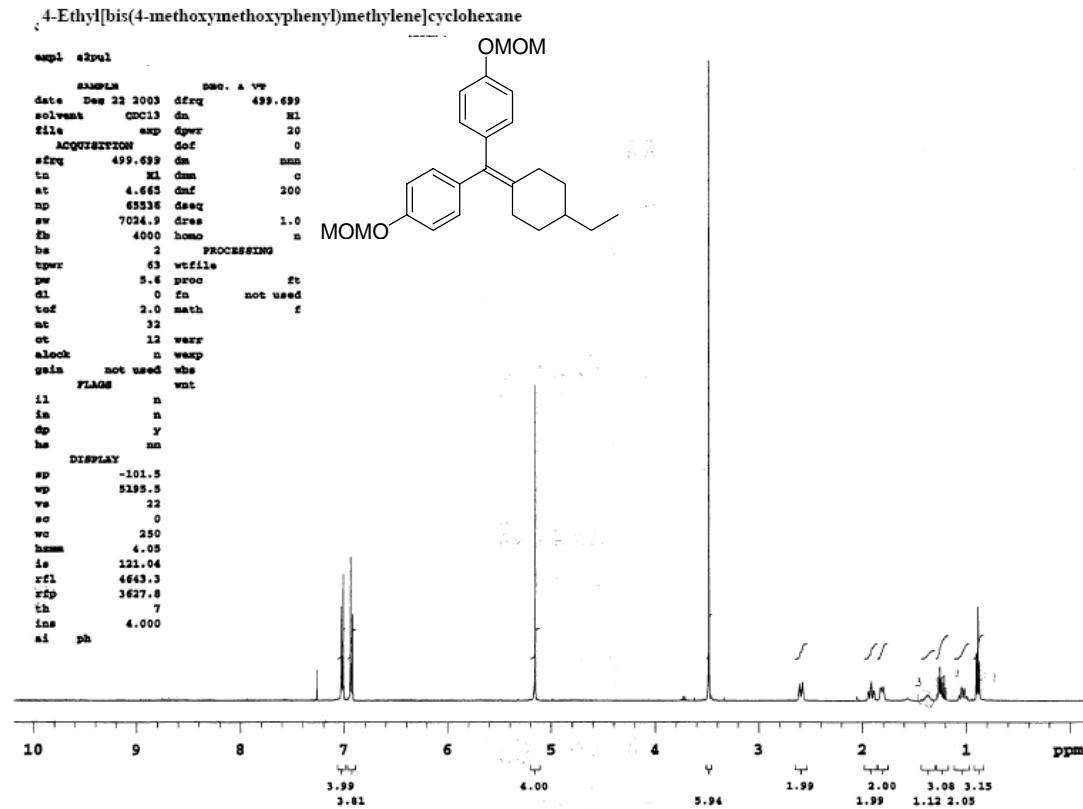
exp2 s2pu1

SAMPLE	DEC. & VT
date Nov 26 2003	dfrq 459.701
solvent Acetone	dn N1
file exp	dprw 42
ACQUISITION	dof -827.6
sfreq 125.663	dmz YYY
scn C13	dms w
atm 1.086	dmf 13879
ns 65536	dsqz
sw 30165.9	drss 90.0
fb 16600	homo n
bw 4	PROCESSING
ss 1 lb	1.00
tppr 32	wtf file
tr 4.5	proc ft
dt 1.000	fn not used
tdf 1884.7	math f
nt 1024	
op 264	werr
clock	wnsp
gain not used	wbs wft ds decade -
PLHSD	dpf



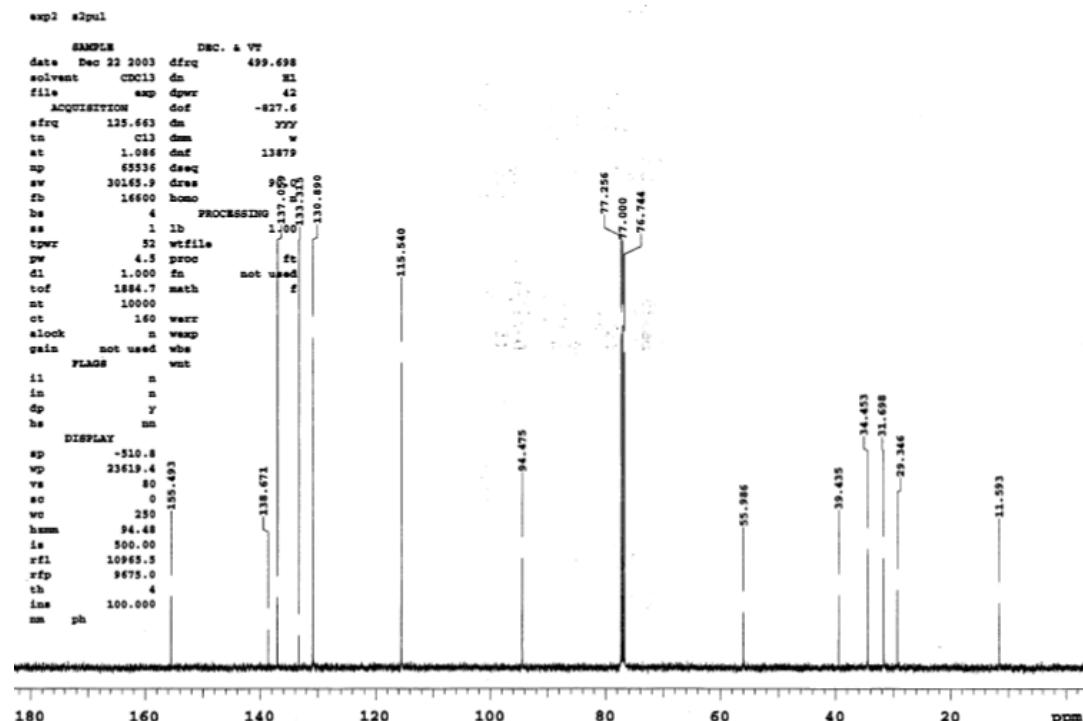
4-Ethyl[bis(4-methoxymethoxyphenyl)methylene]cyclohexane.

¹H NMR (500 MHz, CDCl₃)



¹³C NMR (125 MHz, CDCl₃)

4-Ethyl[bis(4-methoxymethoxyphenyl)methylene]cyclohexane



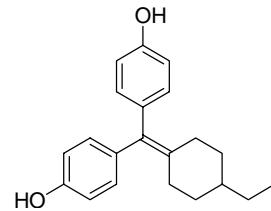
4-Ethyl[bis(4-hydroxyphenyl)methylene]cyclohexane (29b).

¹H NMR (500 MHz, CDCl₃)

4-Ethyl[bis(4-hydroxyphenyl)methylene]cyclohexane (29b)

exp1 *2pul

```
SAMPLE      DEC. & VT
date Dec 24 2003 dfrq 499.699
solvent CDCl3 dn   H1
file    exp dpwv 20
ACQUISITION dof 0
sfrq 499.699 dm  nnn
tn     4.645 dm  c
st     4.645 dmf 200
sp     61536 dseq
sw     7024.9 dres 1.0
fb     4000 homo n
bs     2 PROCESSING
tpwr  63 wtfile
pw     5.6 proc ft
d1     0 fn  not used
tof    2.0 math f
nt     32
ot     32 warr
slock  n wexp
gain   not used vbs
PLANS
      vmt
il     n
in     n
dp     Y
hs     nn
DISPLAY
sp     -101.7
wp     5135.5
vs     100
sc     0
wo     250
hmn   5.48
is     102.94
rfl   4644.1
rfp   3627.8
th     7
ins   2.000
si     ph
```



¹³C NMR (125 MHz, CDCl₃)

4-Ethyl[bis(4-hydroxyphenyl)methylene]cyclohexane (29b)

exp2 *2pul

```
SAMPLE      DEC. & VT
date Dec 24 2003 dfrq 499.698
solvent CDCl3 dn   H1
file    exp dpwv 42
ACQUISITION dof -827.6
sfrq 125.663 dm  vvv
tn     C13 dnm  w
st     1.086 dmf 13879
sp     61536 dseq
sw     30165.9 dres 90.0
fb     16600 homo n
bs     4 PROCESSING
ss     1 lb  1.00
tpwr  52 wtfile
pw     4.5 proc ft
d1     1.000 fn  not used
tof    1884.7 math f
nt     10000
ot     732 warr
slock  n wexp
gain   not used vbs wft ds discalc -
PLANS
      dpf
il     n wmt
in     n
dp     Y
hs     nn
DISPLAY
sp     -510.8
wp     23619.4
vs     140
sc     0
wo     250
hmn   94.48
is     500.00
rfl   10965.5
rfp   9675.0
th     5
ins   100.000
nm   ph
```

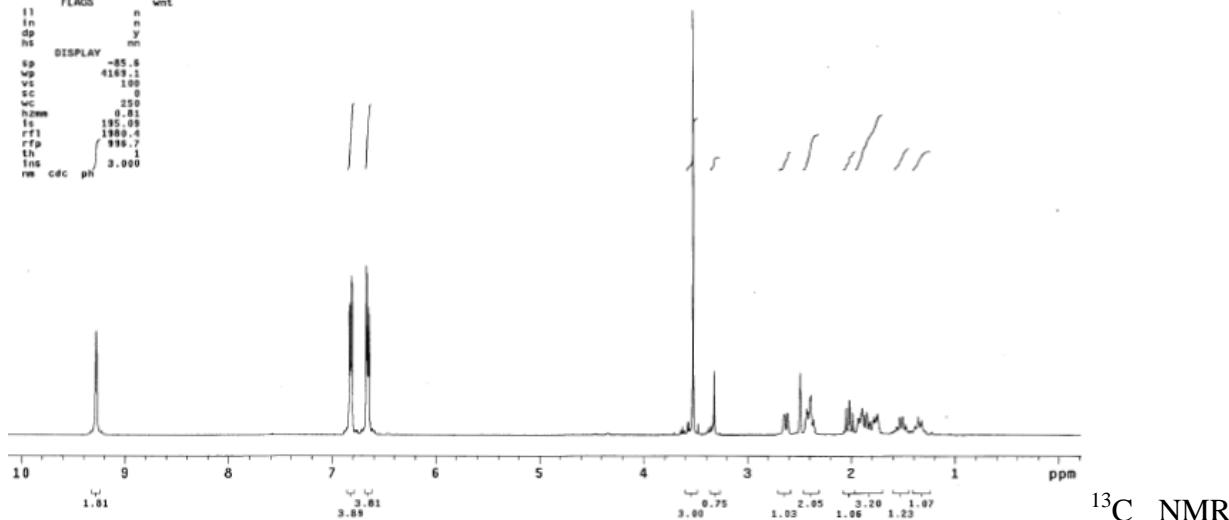
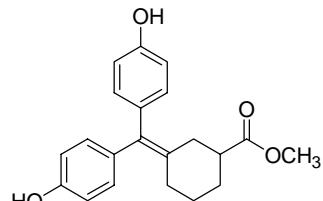
180 160 140 120 100 80 60 40 20 ppm

Methyl 3-[Bis(4-hydroxyphenyl)methylene]cyclohexanecarboxylate (30a).

¹H NMR (400 MHz, acetone-*d*₆)

Methyl 3-[Bis(4-hydroxyphenyl)methylene]cyclohexanecarboxylate (30a)

```
exp3 std1h
SAMPLE DEC. & VT
date Aug 31 2004 dfrq 400.269
solvent DMSO dn H1
file exp dpwrf 38
ACQUISITION dof 0
sfrq 400.269 dm nnn
tn 3.744 dmt 200
at 400.269 dmt 200
dt 6000.0 dres 1.0
fb 3000 homo n
bs 2 PROCESSING
pw 4.0 wfile
pw 1.0 proc
d1 2.000 fn not used
tof math
ct 24 werr
ct 24 wexp
alock not used wbs
gain 1.0 wts
FLAGS wmt
!l n
in n
dp y
hs mn
DISPLAY
sp -85.8
wp 4189.3
vs 100
sc 0
wc 250
hzw 0.81
tr 195.09
rt 1989.4
rfa 1991.7
th 1
ins cdc ph 3.080
```

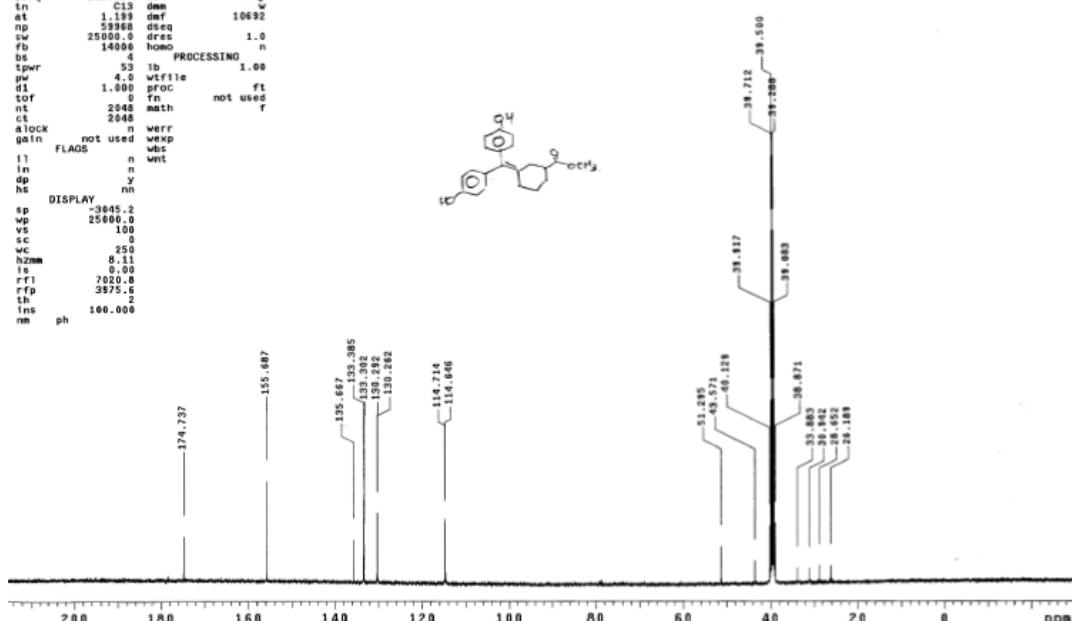
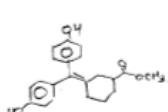


¹³C NMR

(100 MHz, acetone-*d*₆)

Methyl 3-[Bis(4-hydroxyphenyl)methylene]cyclohexanecarboxylate (30a)

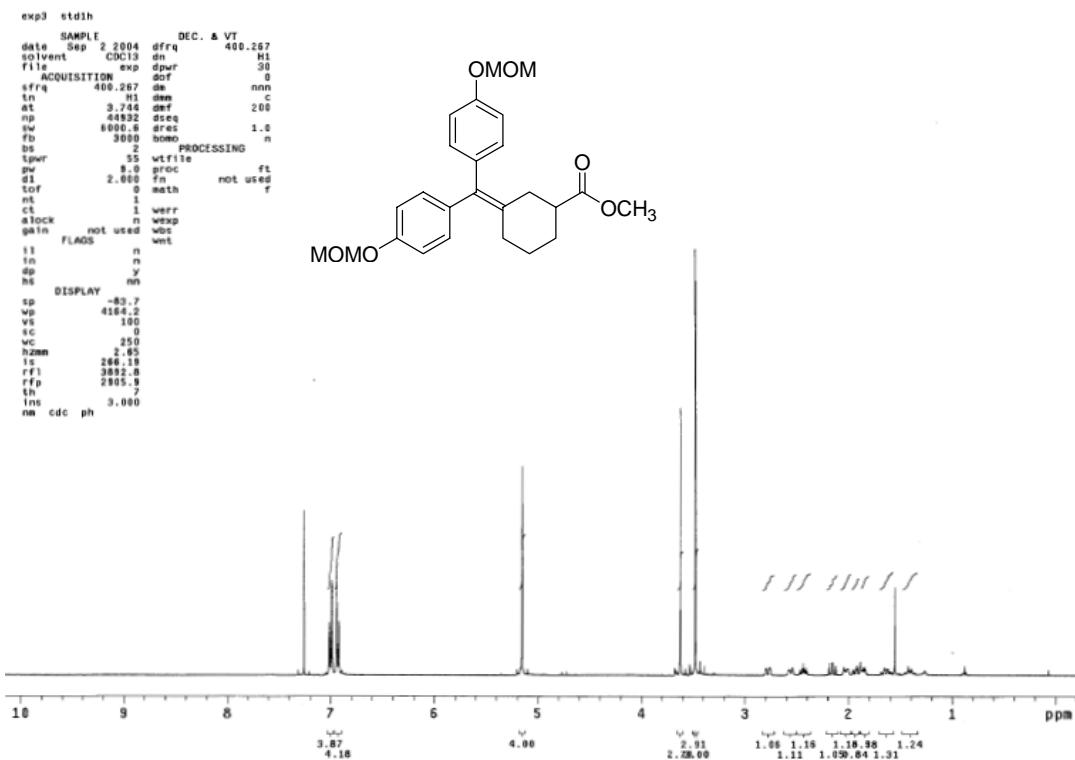
```
exp2 std1c
SAMPLE DEC. & VT
date Aug 31 2004 dfrq 400.269
solvent DMSO dn H1
file exp dpwrf 38
ACQUISITION dof 0
sfrq 100.657 dm nnn
tn C13 dmt 10692
at 1.199 dmt 10692
np 53988 dseq
pw 2500.0 dres 1.0
fb 14000 homo n
bs 3 ib PROCESSING
tpwr 4.0 wfile 1.00
tr 4.0
d1 1.000 proc ft
d1 1.000 fn not used
ct 2048 math
ct 2048 werr
alock not used wexp
gain 1.0 wts
FLAGS wmt
!l n
in n
dp y
hs mn
DISPLAY
sp -3845.2
wp 25000.0
vs 100
sc 0
wc 250
hzw 8.11
tr 0.00
rt 7020.8
rfa 3975.5
th 2
ins 100.000
nm ph
```



Methyl 3-[Bis(4-methoxymethoxyphenyl)methylene]cyclohexanecarboxylate (33a).

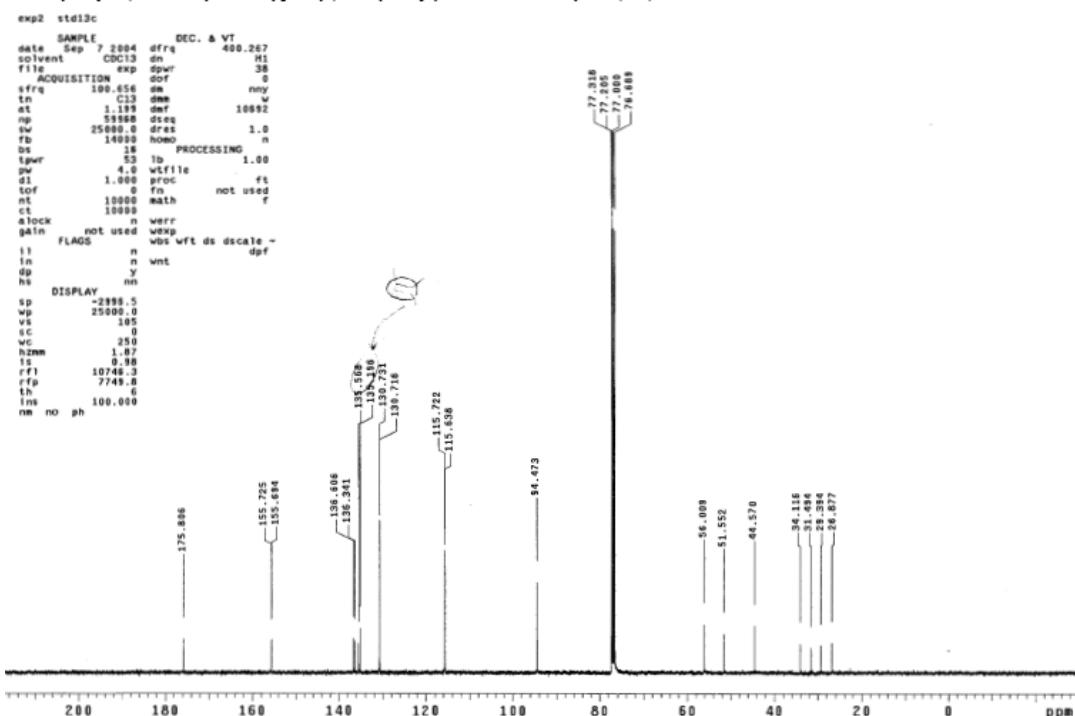
¹H NMR (400 MHz, CDCl₃)

Methyl 3-[Bis(4-methoxymethoxyphenyl)methylene]cyclohexanecarboxylate (33a)



¹³C NMR (100 MHz, CDCl₃)

Methyl 3-[Bis(4-methoxymethoxyphenyl)methylene]cyclohexanecarboxylate (33a)



3-Hydroxymethyl[bis(4-methoxymethoxyphenyl)methylene]cyclohexane (34a).

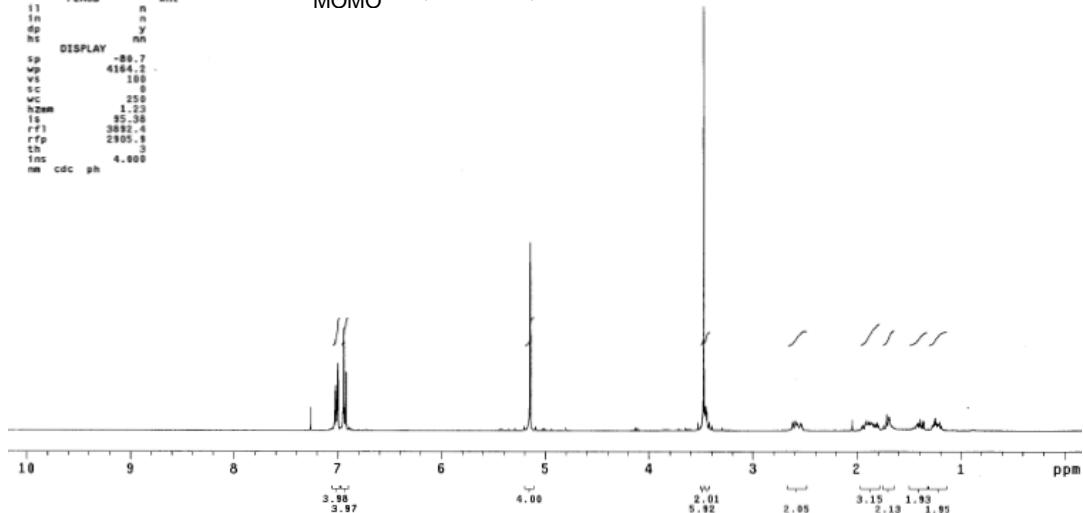
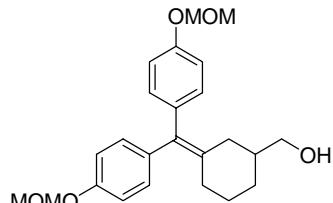
¹H NMR (400 MHz, CDCl₃)

3-Hydroxymethyl[bis(4-methoxymethoxyphenyl)methylene]cyclohexane (34a)

```

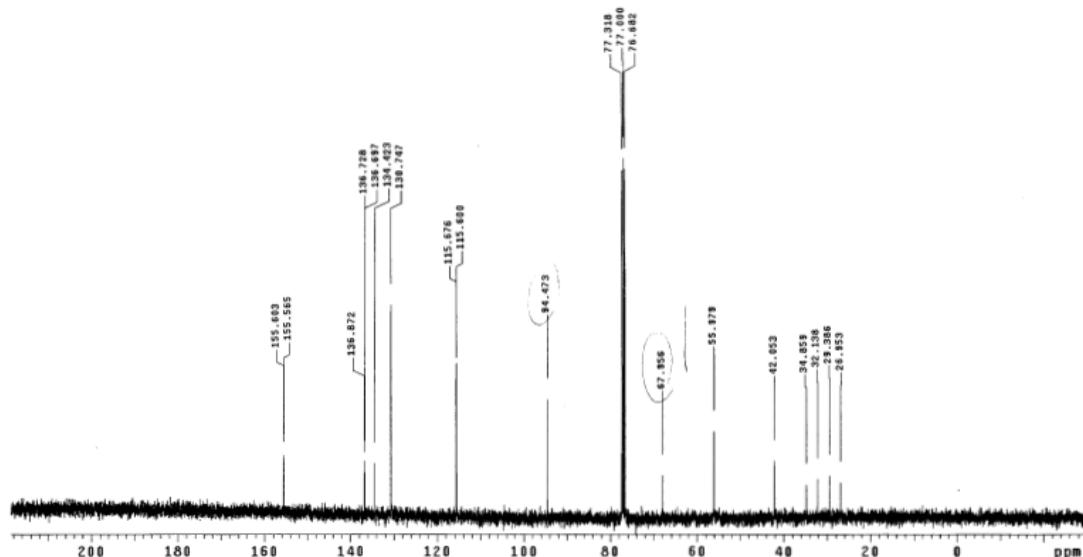
exp3 stdih
SAMPLE          DEC. & VT
date Sep 6 2005 dfrq 400.267
solvent   CDCl3      dt1
file      exp dpur 30
ACQUISITION dof 0
tfrq    400.267 def nmm
tn       H1      nme C
at       3.744      200
np       44532      dsw
nw       600        1.0
rb       30500     homo n
bs       2          PROCESSING
tpur    51 wfile
pw0     8.0         ft
d1      2.009     fm not used
tof      9         math f
nt      30
ct      12 warr
alock   n wexp
gain    not used wos
gain2   not used wos
FLAGS   n
in      n
dp      nn
hs      nn
DISPLAY
sp      -89.7
wp      416.2
v1      100
sc      0
uc      250
hcsm   1.23
is      95.38
r1f    3892.4
rfp    2900.8
th      3
ins    4.000
nm cdc ph

```



¹³C NMR (100 MHz, CDCl₃)

3-Hydroxymethyl[bis(4-methoxymethoxyphenyl)methylene]cyclohexane (34a)



3-Hydroxymethyl[bis(4-hydroxyphenyl)methylene]cyclohexane (35a).

¹H NMR (400 MHz, DMSO-*d*₆)

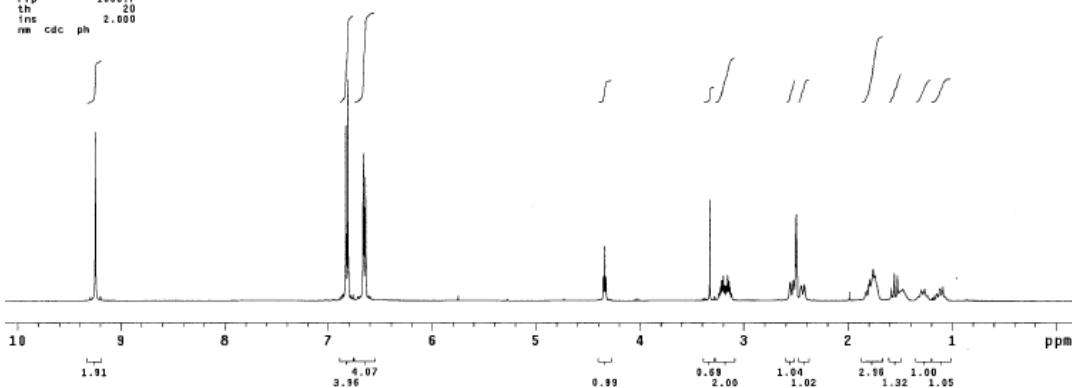
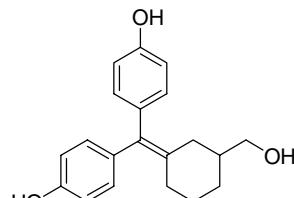
3-Hydroxymethyl[bis(4-hydroxyphenyl)methylene]cyclohexane (35a)

```

  expr stdin      SAMPLE          DEC. & VT
  data Sep 8 1977  sfrq        400.288
  solvent DMSO      dswd        His
  solvent   ssp       dswr        39.0
  ACQUISITION dof         0
  sfrq    409.289  nmr
  at      3.744  dec
  np     4952.3  dsep
  sw     6000.6  dres
  bw     300.0  home
  bs      2        n
  pwf      55        PROCESSING
  pw     9.0        pffile
  2.000      9.0        proc
  tof      0        fit
  n       0        not used
  nt      16
  ct      16        math
  atck     16        werr
  gain    not used  wbs
  gain      16        wnt
  flags      nn
  ii      n
  in      n
  dp      n
  hs      nn

  DISPLAY
  sp      -89.7
  wp     4154.2
  vs      50
  sc      250
  hc      250
  hcawm  2.41
  ts     273.44
  ff     186.7
  rfp    1800.7
  th      20
  ins    2.000

```



¹³C NMR (100 MHz, DMSO-*d*₆)

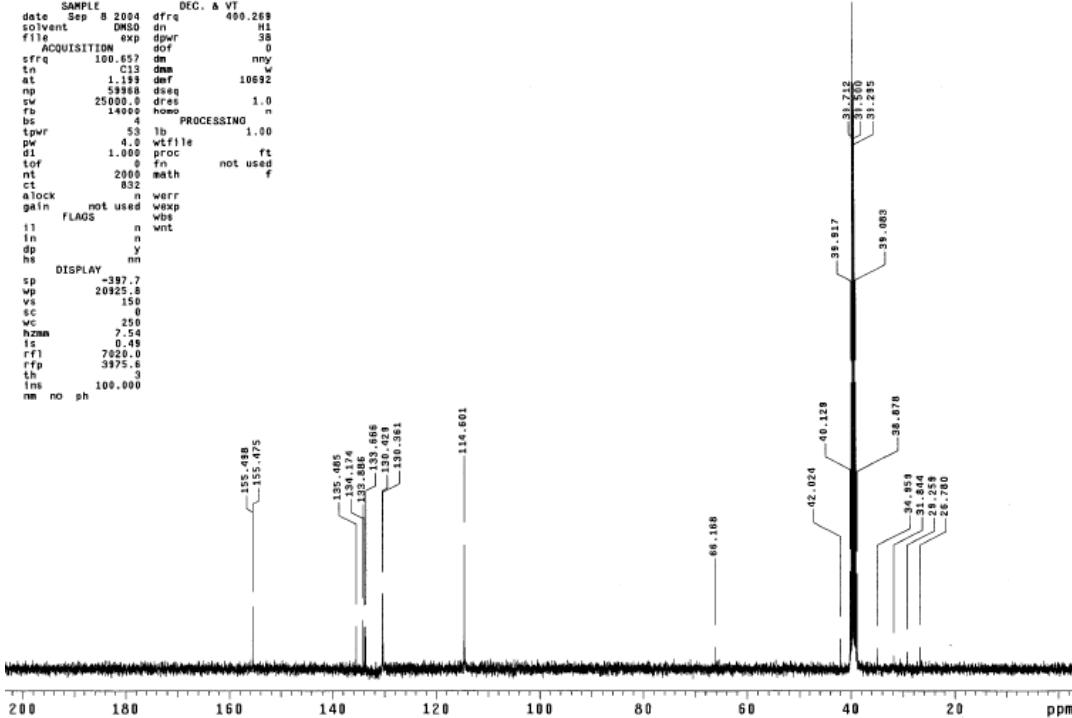
3-Hydroxymethyl[bis(4-hydroxyphenyl)methylene]cyclohexane (35a)

```

exp3 std13c
      SAMPLE          DEC. & VT
date Sep 8 2004 dfrg 450.26
solvent        DMSO H
coagent        DMSO S
      ACQUISITION dof
      sfrq 100.657 dm
      ats 1.433 dms
      ats 1.433 dmr 10691
      np 53988 dseq
      sw 25000.0 dres 1.
      bs 14000.0 dres
      bs      4 PROCESSING
      tprw 53 lb 1.0
      pw 4.0 wtfle
      dl 1.000 wfcoc f
      tof 2000 math
      nt 2000 math
      ct 852
      alock 4 warr
      gain not used wexp
      flags      wbs
      t1      n wbt
      t1      n wbt
      dp      nm
      hs      nm

      DISPLAY
      sp      -397.7
      wp      20325.8
      vs      150
      ws      0
      wcr      250
      hzmn      7.54
      ts      0.49
      rfl 7620.0
      rfp 3375.6
      th      3
      ins 100.000

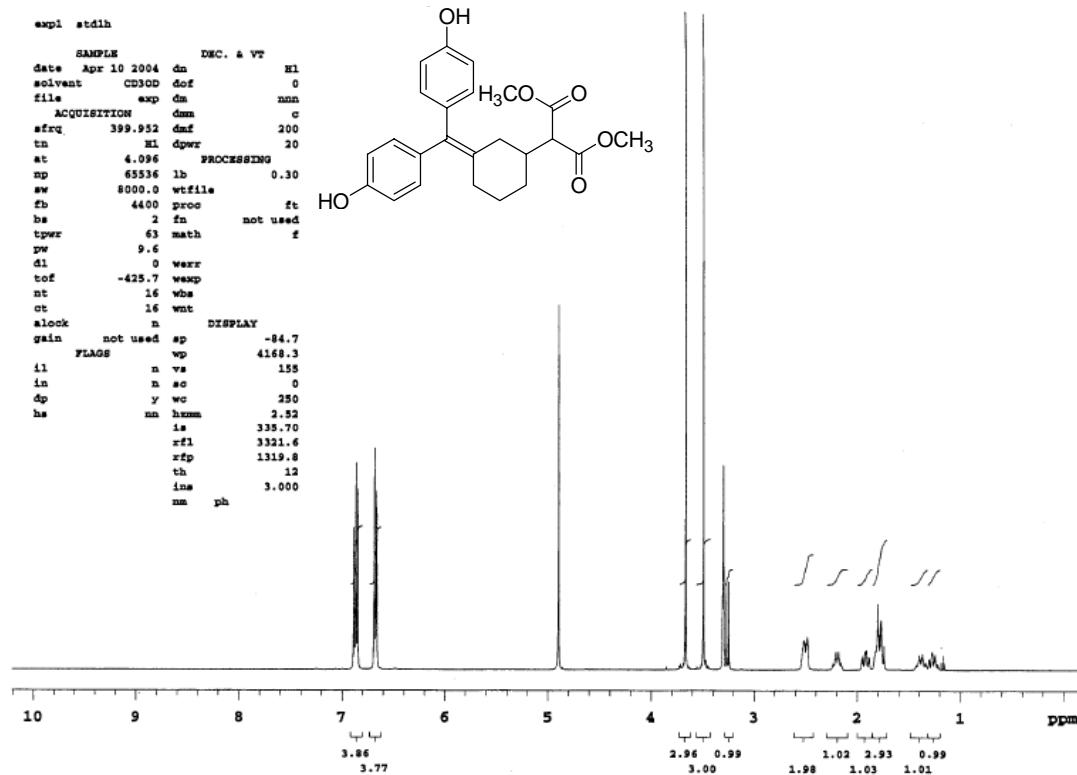
```



Dimethyl 2-{3-[Bis(4-hydroxyphenyl)methylene]cyclohexyl}malonate (30b).

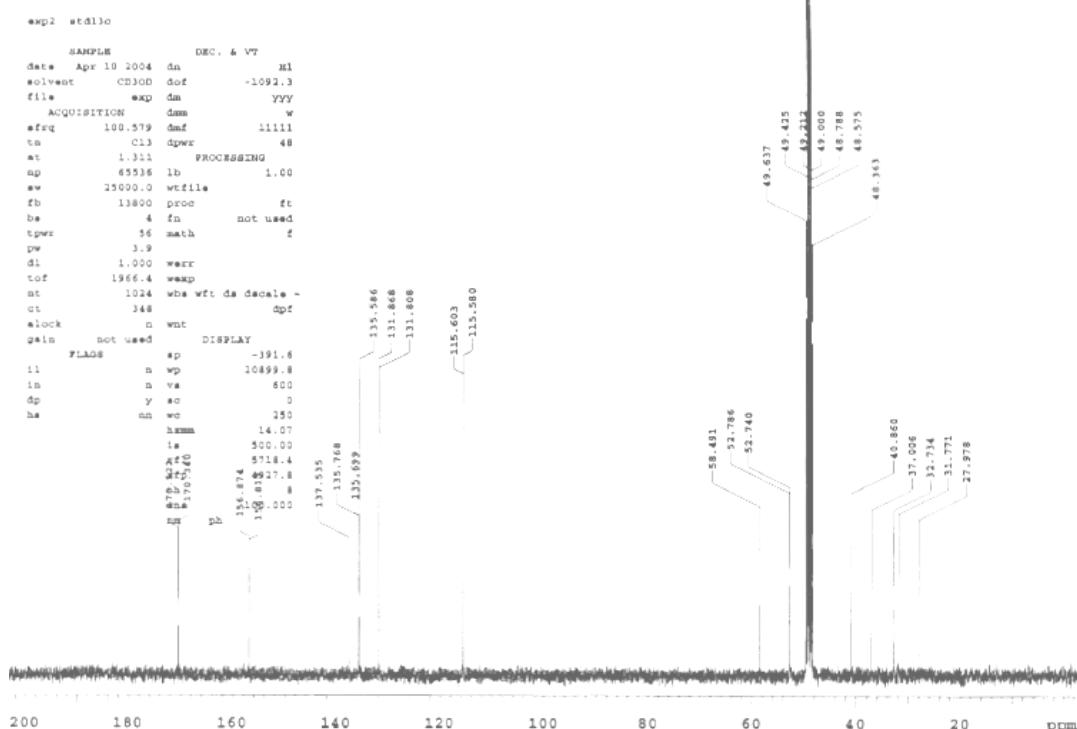
¹H NMR (400 MHz, methanol-*d*₄)

Dimethyl 2-[3-[Bis(4-hydroxyphenyl)methylene]cyclohexyl]malonate (30b)



¹³C NMR (100 MHz, methanol-*d*₄)

Dimethyl 2-(3-[Bis(4-hydroxyphenyl)methylene]cyclohexyl)malonate (30b)



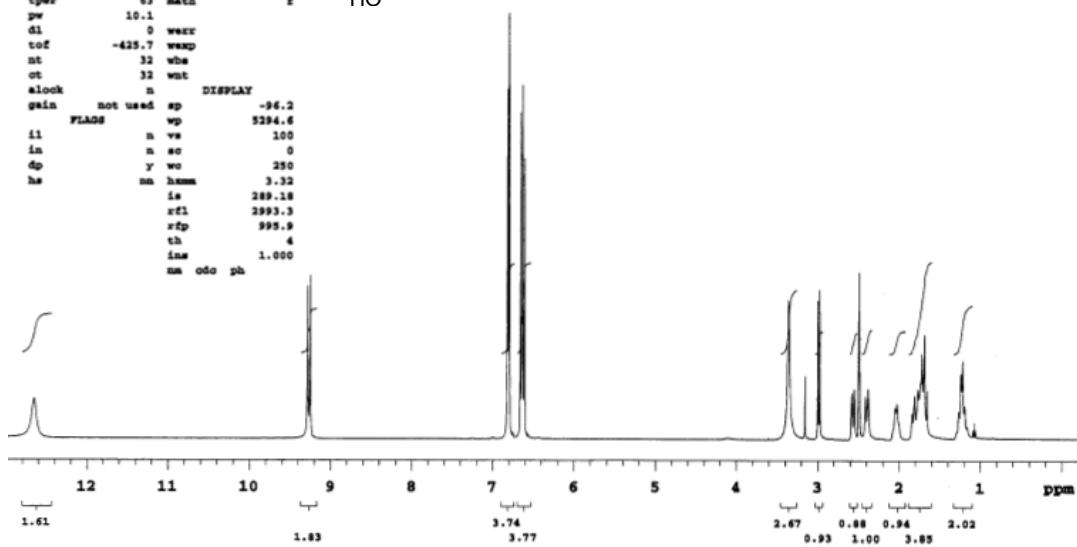
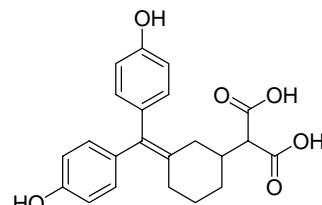
2-[3-[Bis(4-hydroxyphenyl)methylene]cyclohexyl]malonic acid (31).

¹H NMR (400 MHz, DMSO-*d*₆)

2-[3-[Bis(4-hydroxyphenyl)methylene]cyclohexyl]malonic acid (31)

exp1 std1h

```
SAMPLE      DSC. & VT
date Jun 26 2004 dn   H1
solvent DMSO dof    0
file   exp dn   nnn
ACQUISITION dnm   c
sfrq  399.953 dmf  200
tn    H1 dppr  20
at    4.096   PROCESSING
np    65536 1b   0.30
sw   8000.0 wtfil
fb    4400 proc   ft
bs    2 fn    not used
tppw  63 math   f
pw    10.1
dl    0 wetc
t0f   -425.7 wexp
nt    32 wha
ot    32 wnt
alock n
DISPLAY
gain  not used ap   -96.2
PLADS  wp   5294.6
ii    n vs   100
in    n sc   0
dp    y wo   250
hs    nn hexm  3.32
           is   289.18
           rrf1 2993.3
           rfp 995.9
           th   4
           ins  1.000
           na odo ph
```

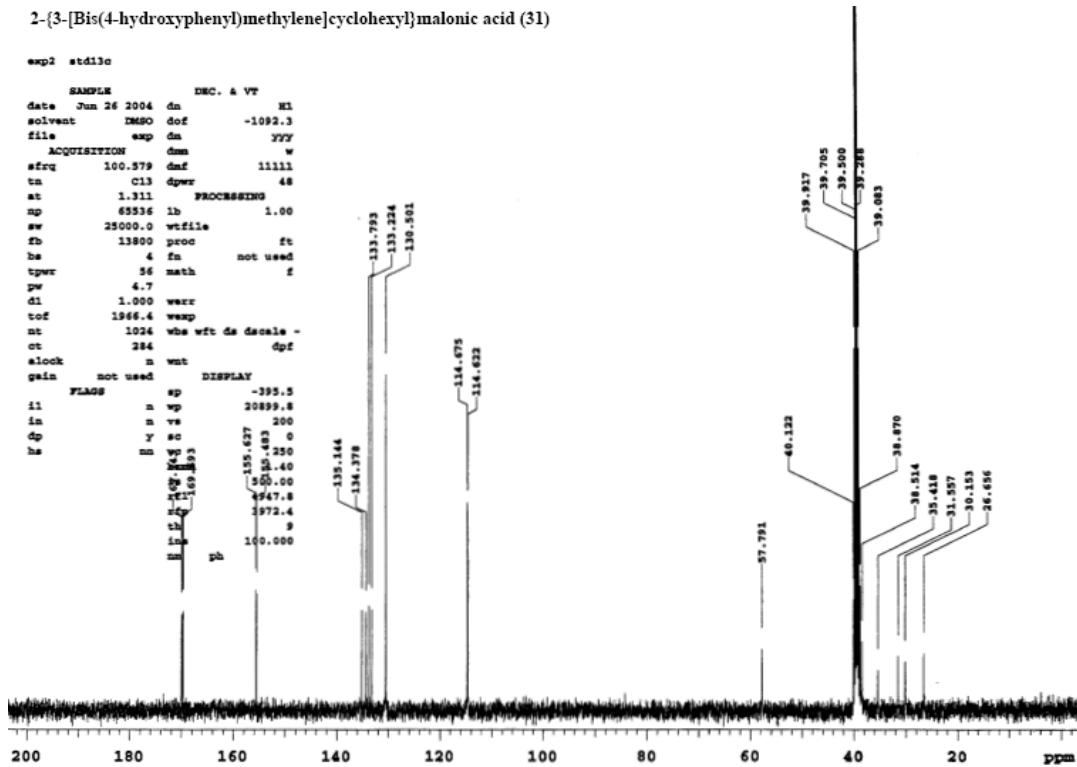


¹³C NMR (100 MHz, DMSO-*d*₆)

2-[3-[Bis(4-hydroxyphenyl)methylene]cyclohexyl]malonic acid (31)

exp2 std13c

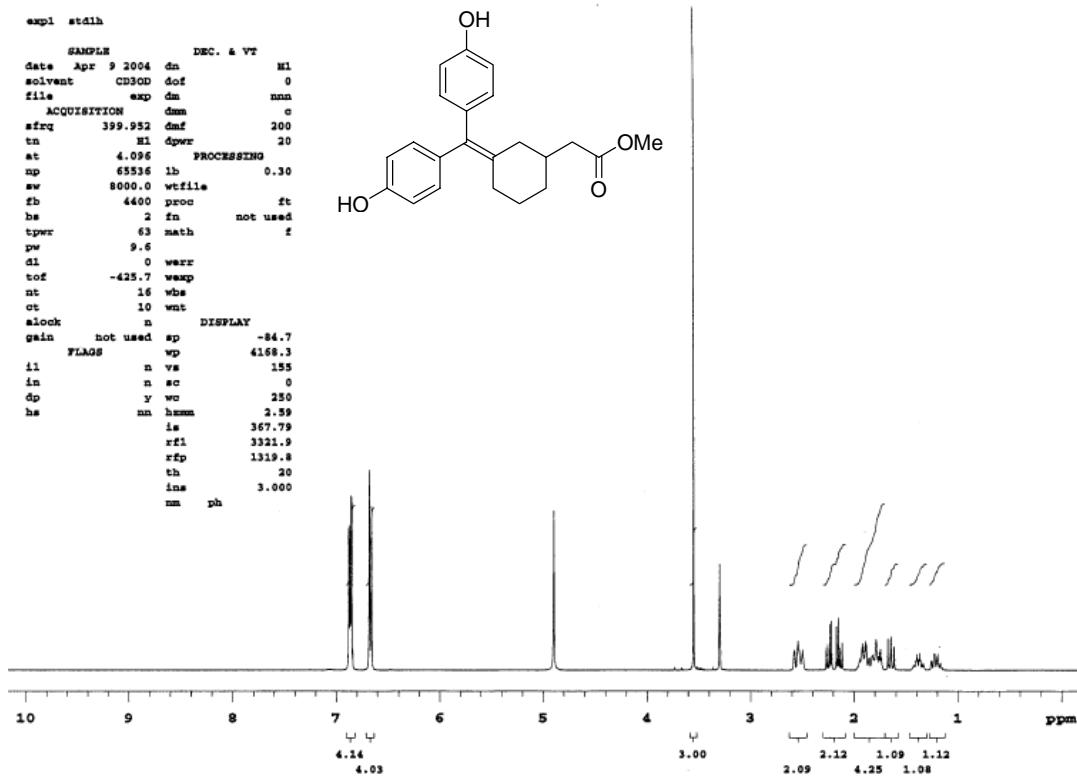
```
SAMPLE      DSC. & VT
date Jun 26 2004 dn   H1
solvent DMSO dof   -1092.3
file   exp dn   vvv
ACQUISITION dnm   w
sfrq  100.579 dmf  11111
tn    C13 dppr  48
at    1.311   PROCESSING
np    65536 1b   1.00
sw   25000.0 wtfil
fb    138000 proc   ft
bs    4 fn    not used
tppw  56 math   f
pw    4.7
dl    1.000 wetc
t0f   1946.4 wexp
nt    1024 wha wft ds decsle -
ot    284      dpf
alock n wut
gain  not used DISPLAY
PLADS  ap   -395.5
ii    n wp   20899.8
in    n vs   200
dp    y sc   0
hs    nn wp  250
           is   135.144
           rrf1 135.178
           rfp 135.144
           th   9
           ins  100.000
           na odo ph
```



Methyl 2-{3-[Bis(4-hydroxyphenyl)methylene]cyclohexyl}acetate (32).

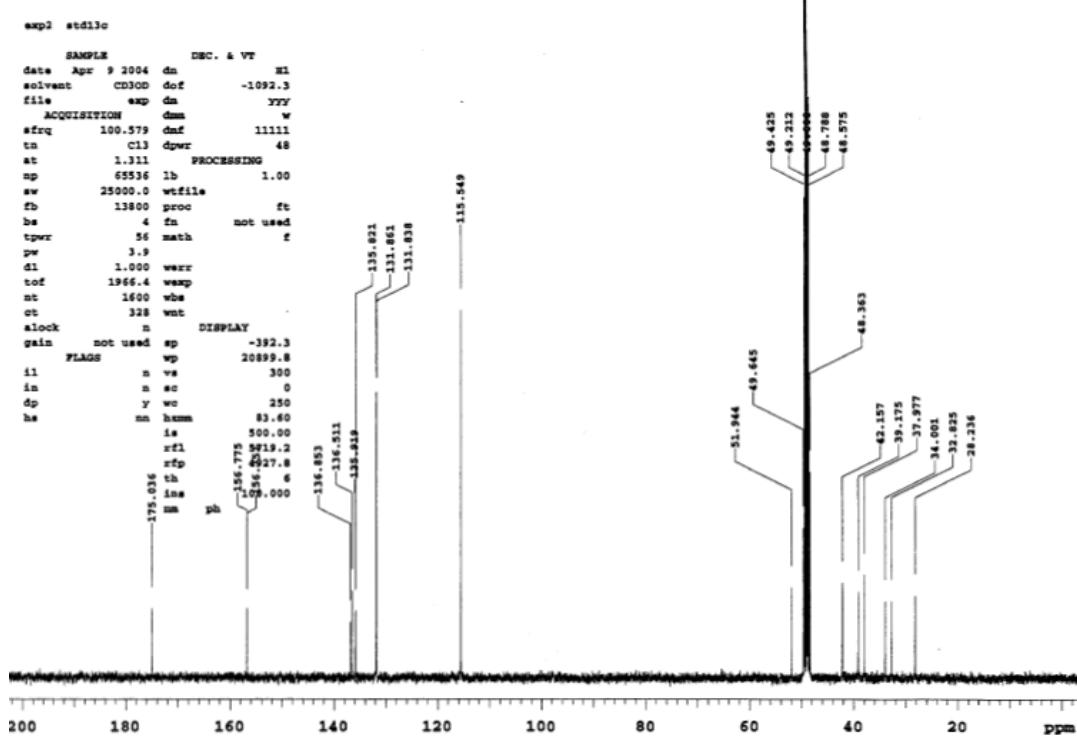
¹H NMR (400 MHz, methanol-d₄)

Methyl 2-{3-[Bis(4-hydroxyphenyl)methylene]cyclohexyl}acetate (32)



¹³C NMR (100 MHz, methanol-d₄)

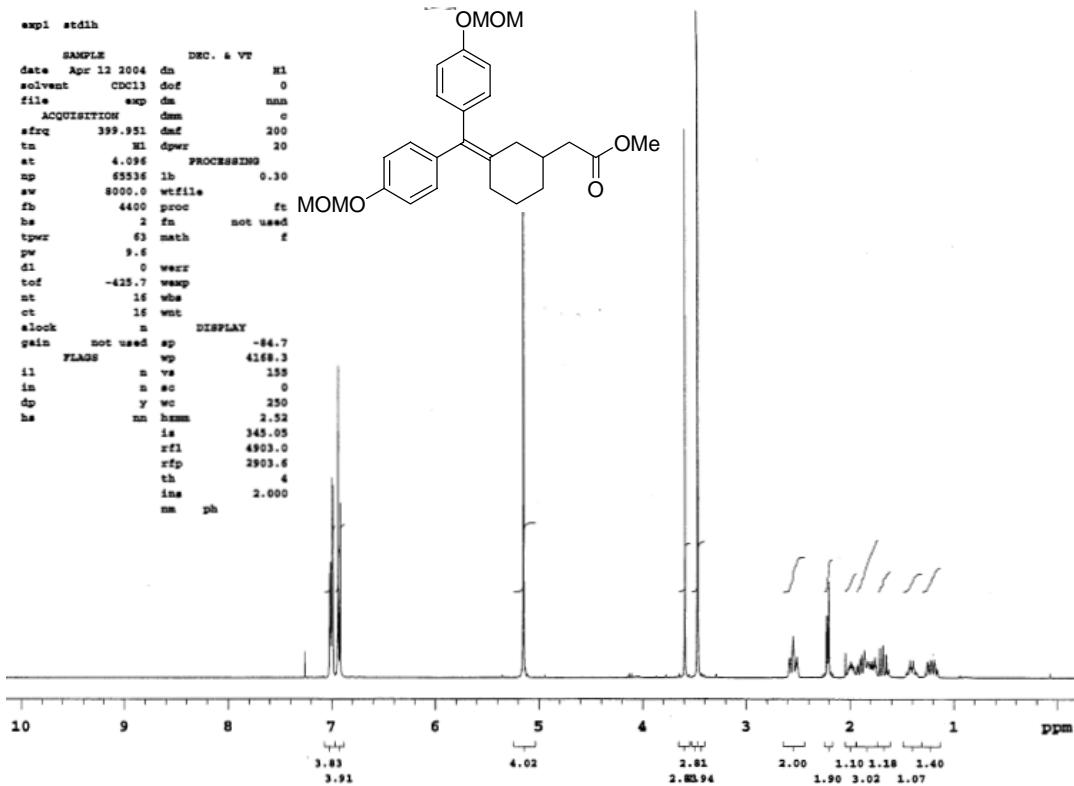
Methyl 2-{3-[Bis(4-hydroxyphenyl)methylene]cyclohexyl}acetate (32)



Methyl 2-{3-[Bis(4-methoxymethoxyphenyl)methylene]cyclohexyl}acetate (33b).

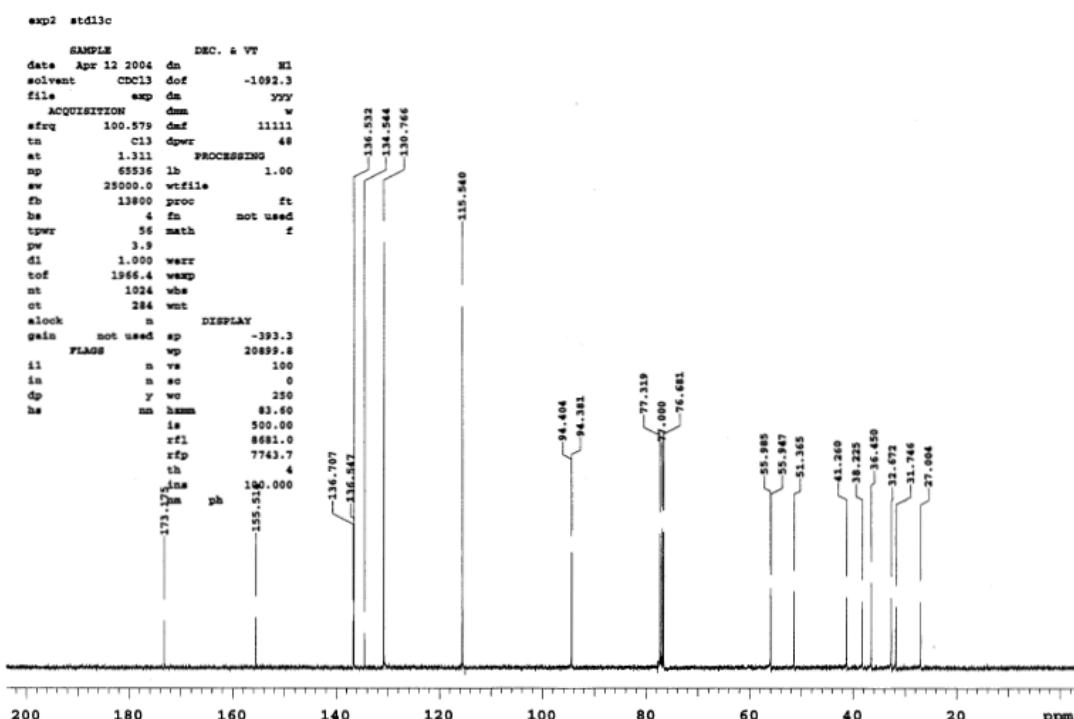
¹H NMR (400 MHz, CDCl₃)

Methyl 2-{3-[Bis(4-methoxymethoxyphenyl)methylene]cyclohexyl}acetate (33b)



¹³C NMR (100 MHz, CDCl₃)

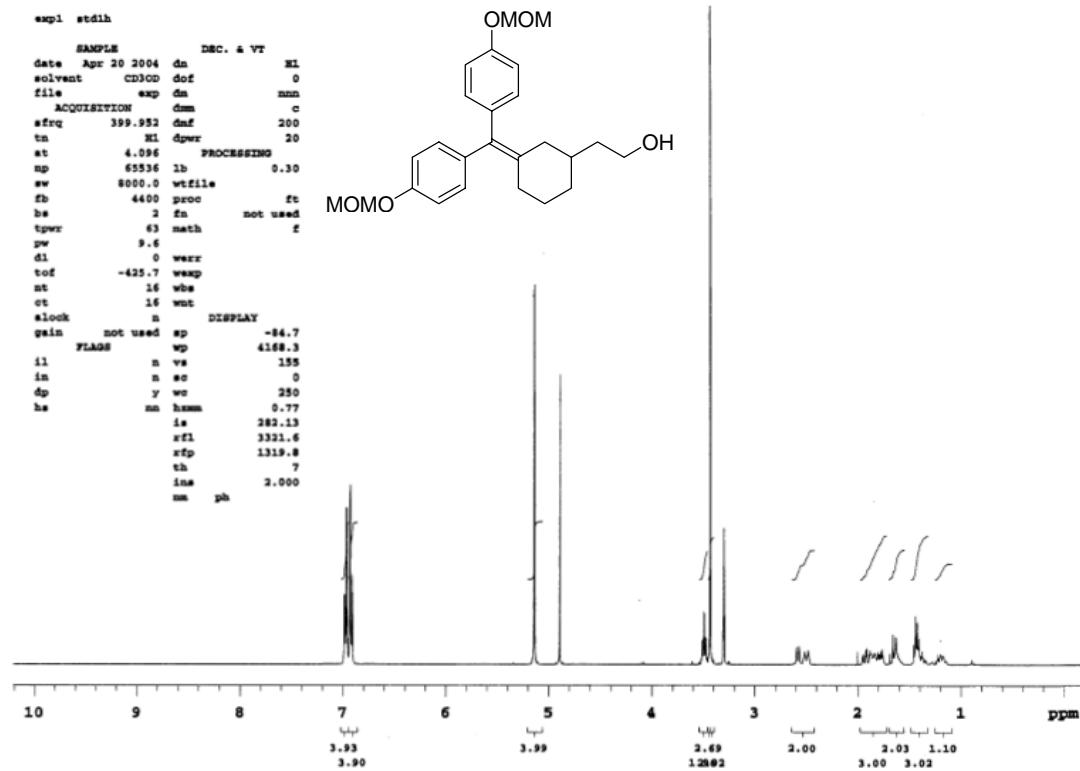
Methyl 2-{3-[Bis(4-methoxymethoxyphenyl)methylene]cyclohexyl}acetate (33b)



3-(2-Hydroxyethyl)[bis(4-methoxymethoxyphenyl)methylene]cyclohexane (34b).

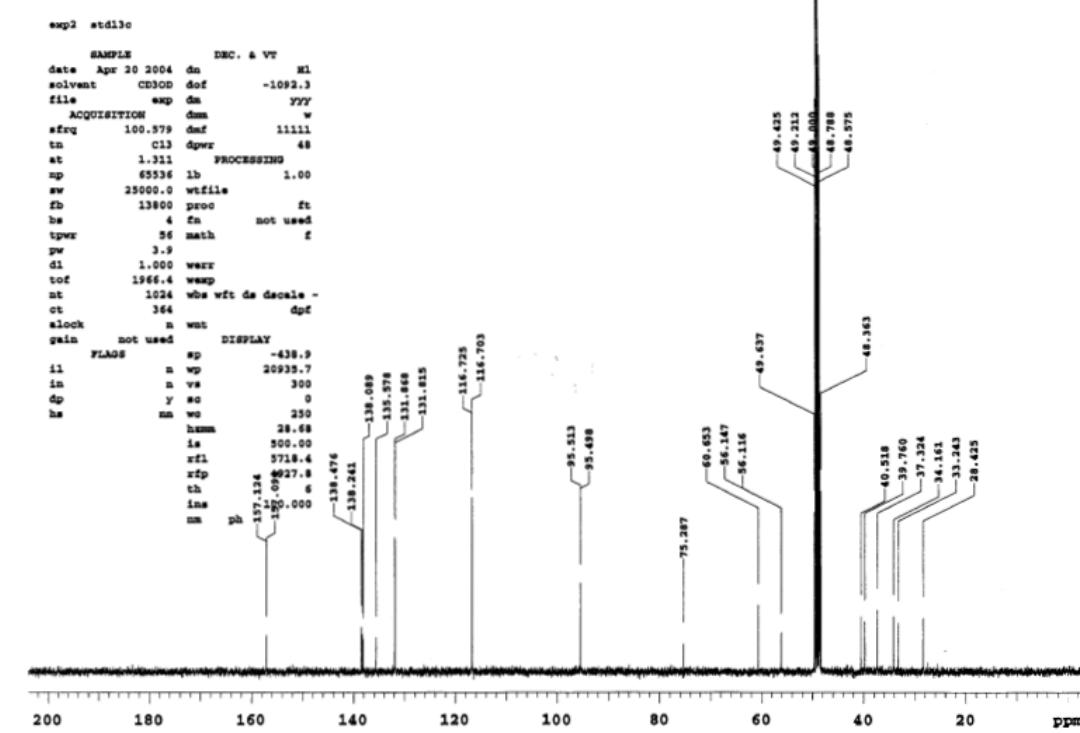
¹H NMR (400 MHz, methanol-*d*₄)

3-(2-Hydroxyethyl)[bis(4-methoxymethoxyphenyl)methylene]cyclohexane (34b)



¹³C NMR (100 MHz, methanol-*d*₄)

3-(2-Hydroxyethyl)[bis(4-methoxymethoxyphenyl)methylene]cyclohexane (34b)

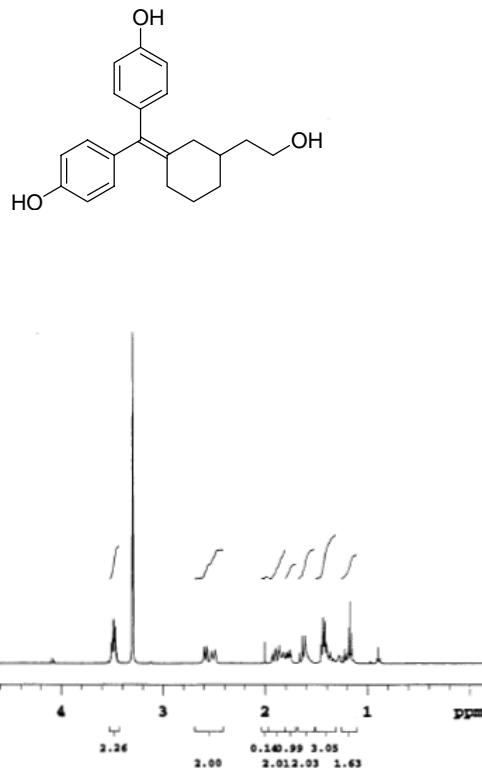


3-(2-Hydroxyethyl)[bis(4-hydroxyphenyl)methylene]cyclohexane (35b).

¹H NMR (400 MHz, methanol-d₄)

3-(2-Hydroxyethyl)[bis(4-hydroxyphenyl)methylene]cyclohexane (35b)

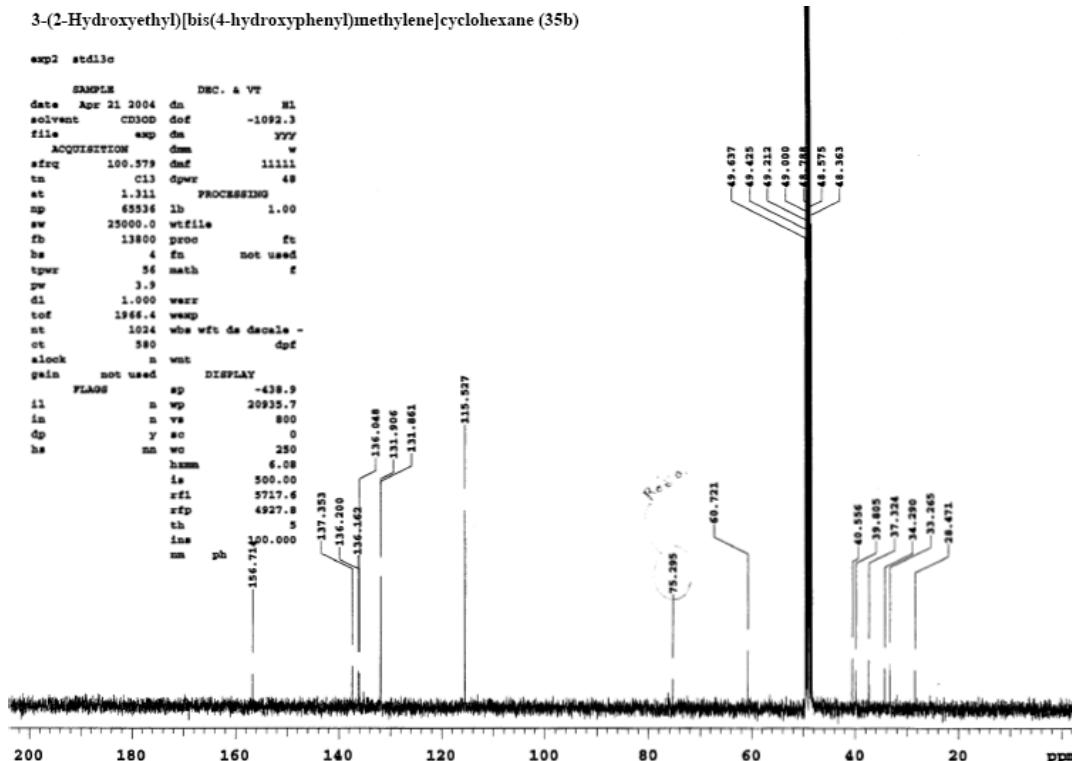
```
exp1 std1h
SAMPLE      DSC. & VT
date  Apr 21 2004 dn   H1
solvent  CD3OD dof   0
file    exp dm   nnn
ACQUISITION dnm   c
sfrq   399.952 dmf  200
tn     H1 dpwr   20
at     4.096   PROCESSING
np    65536  lb   0.30
sw    8000.0 wtf file
fb    4400  proc   ft
bs    2 fn   not used
tpwr   64 math   f
pw    9.6
d1    0 warr
tof   -425.7 wexp
nt    16 wbs
ct    16 wnt
alock  n
gain  not used sp   -84.7
FLAGS   n vs   155
il    n sc   0
in    n wc   250
dp    y wc
hs    nn hznm  4.40
is    308.54
rf1   3321.9
rf2p  1319.8
th    20
ins   2.000
nm   ph
```



¹³C NMR (100 MHz, methanol-d₄)

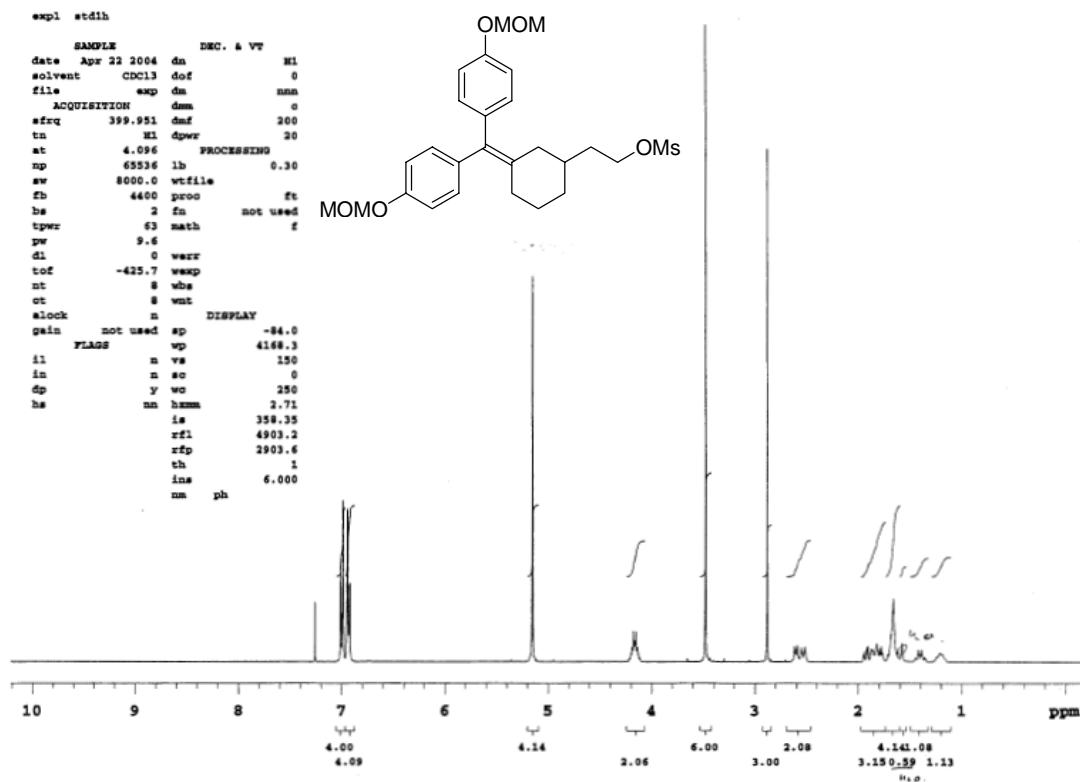
3-(2-Hydroxyethyl)[bis(4-hydroxyphenyl)methylene]cyclohexane (35b)

```
exp2 std13c
SAMPLE      DSC. & VT
date  Apr 21 2004 dn   H1
solvent  CD3OD dof  -1092.3
file    exp dm   3VV
ACQUISITION dnm   w
sfrq   100.779 dmf  11111
tn     C13 dpwr   48
at     1.111   PROCESSING
np    65536  lb   1.00
sw    25000.0 wtf file
fb    13800  proc   ft
bs    4 fn   not used
tpwr   56 math   f
pw    3.9
d1    1.000 warr
tof   1866.4 wexp
nt    1024 wbs wft da dascale -
ct    380 dmf
alock  n wnt
gain  not used DISPLAY
FLAGS   sp   -438.9
il    n wp   20935.7
in    n vs   800
dp    y sc   0
hs    nn wc   250
nm   ph
```



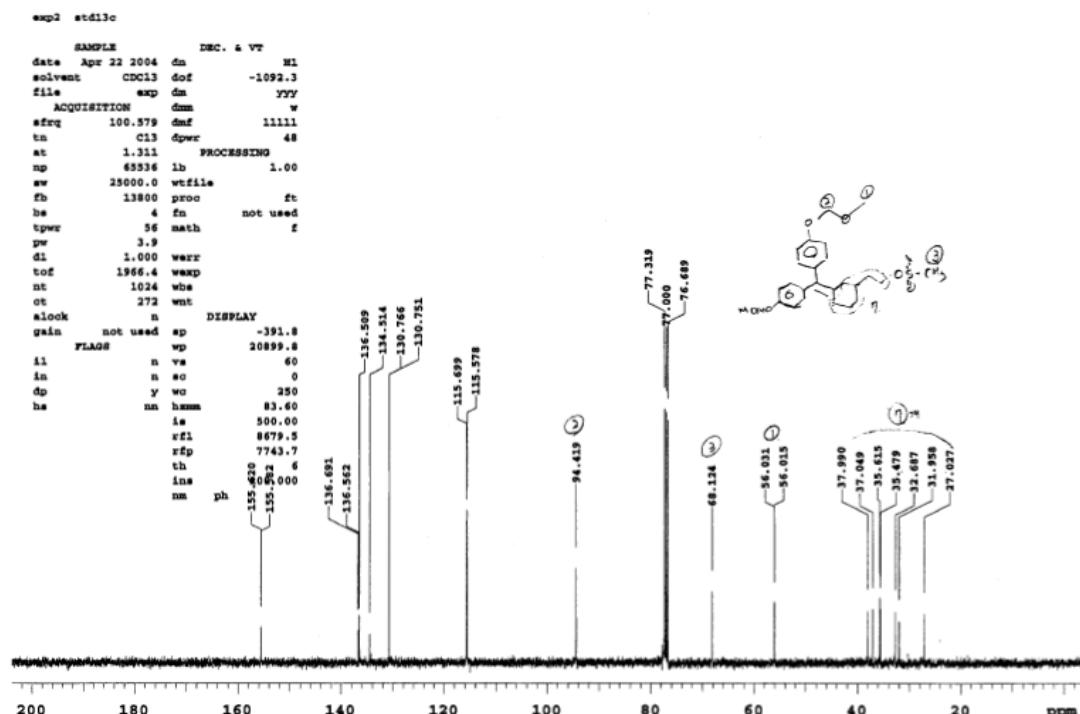
**3-(2-Methanesulfonyloxyethyl)[bis(4-methoxymethoxyphenyl)methylene]cyclohexane
(36b). ^1H NMR (400 MHz, CDCl_3)**

3-(2-Methanesulfonyloxyethyl)[bis(4-methoxymethoxyphenyl)methylene]cyclohexane (36b)



^{13}C NMR (100 MHz, CDCl_3)

3-(2-Methanesulfonyloxyethyl)[bis(4-methoxymethoxyphenyl)methylene]cyclohexane (36b)

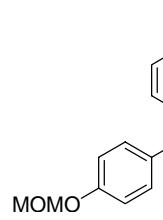


3-(2-Fluoroethyl)[bis(4-methoxymethoxyphenyl)methylene]cyclohexane.

¹H NMR (400 MHz, CDCl₃)

3-(3-Fluoropropyl)[bis(4-methoxymethoxyphenyl)methylene]cyclohexane.

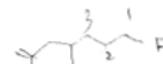
```
SAMPLE DEC. & VT
date Dec 8 2004 dfrq 400.267
solvent CDCl3 dn H1
file exp dpwv 30
ACQUISITION dof 0
sfrq 400.267 dn mnny
tn 113 dn c
at 3.741 dmf 200
np 44932 dseq 1.0
sw 8000.0 tics 1.0
fb not used homo n
bs not used 2 PROCESSING
tpwr 55 wtflic
pw 5.0 proc
dt 2.000 fn not used ft
tcf 0 meth f
nt 16
ct 1 werr
clock 1 wexp
gain not used wbs wnt
FLAGS
(i) n
(in) n
(dp) y
(hs) nn
DISPLAY
sp -407.8
wp 4167.0
vs 150
ic 0
wc 250
hzw 0.65
is 455.04
rf1 3984.9
rf2 2905.9
th 12
lms 6.000
nm ph
```



¹³C NMR (100 MHz, CDCl₃)

3-(3-Fluoropropyl)[bis(4-methoxymethoxyphenyl)methylene]cyclohexane.

```
SAMPLE DEC. & VT
date Dec 8 2004 dfrq 400.267
solvent CDCl3 dn H1
file exp dpwv 30
ACQUISITION dof 0
sfrq 100.656 dn mnny
tn 113 dn c
at 1.191 10692
np 25000.0 dres 1.0
sw 25000.0 dres 1.0
fb not used homo n
bs not used PROCESSING
tpwr 53 lb 1.00
pw 4.0 wtflic
dt 1.000 proc
tcf 0 fn not used f
nt 2000 meth f
ct 200
clock 1 werr
gain not used wexp
FLAGS
(i) n
(in) n
(dp) y
(hs) nn
DISPLAY
sp -407.8
wp 20925.8
vs 80
ic 0
wc 250
hzw 83.70
is 589.00
rf1 19751.4
rf2 7749.8
th 12
lms 100.000
nm no ph
```

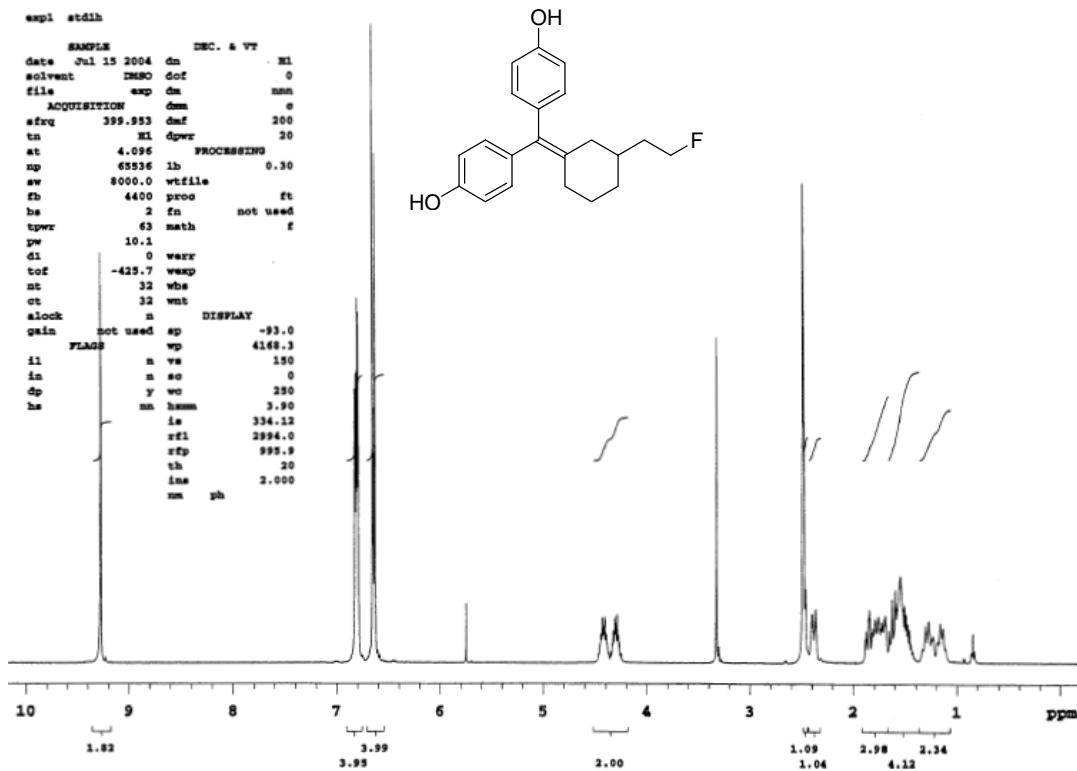


ppm

3-(2-Fluoroethyl)[bis(4-hydroxyphenyl)methylene]cyclohexane (37b).

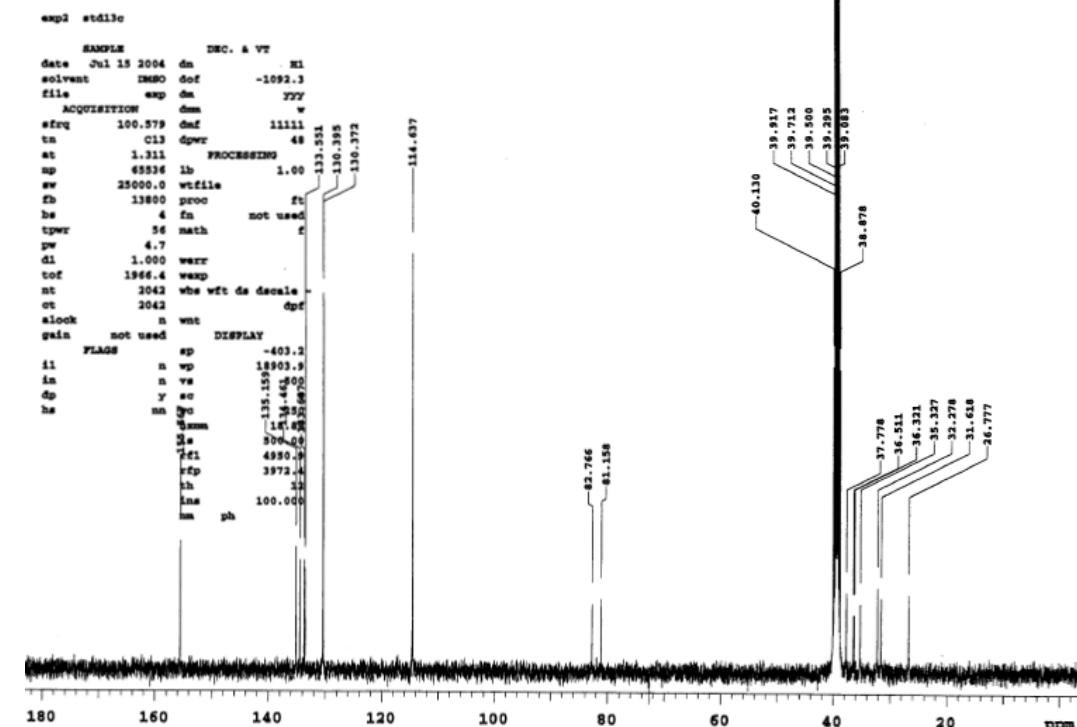
¹H NMR (400 MHz, methanol-*d*₄)

3-(2-Fluoroethyl)[bis(4-hydroxyphenyl)methylene]cyclohexane (37b)



¹³C NMR (100 MHz, methanol-*d*₄)

3-(2-Fluoroethyl)[bis(4-hydroxyphenyl)methylene]cyclohexane (37b)

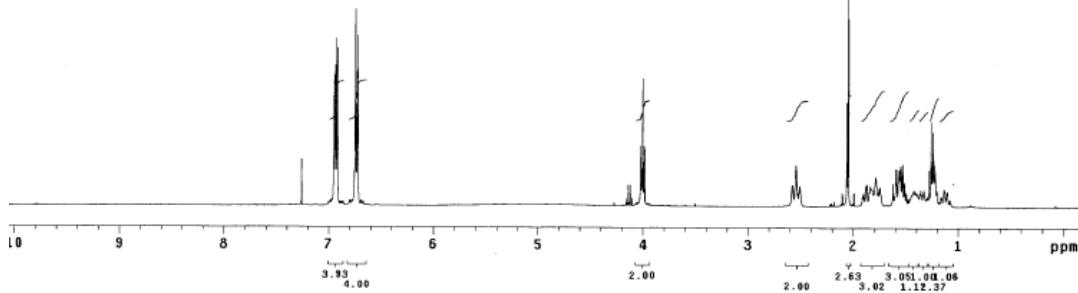
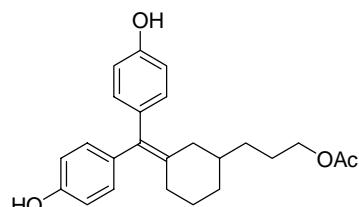


3-(3-Acetoxy-*n*-propyl)-1-[bis(4-hydroxyphenyl)methylene]cyclohexane (30c).

¹H NMR (400 MHz, CDCl₃)

3-(3-Acetoxy-*n*-propyl)-1-[bis(4-hydroxyphenyl)methylene]cyclohexane (30c)

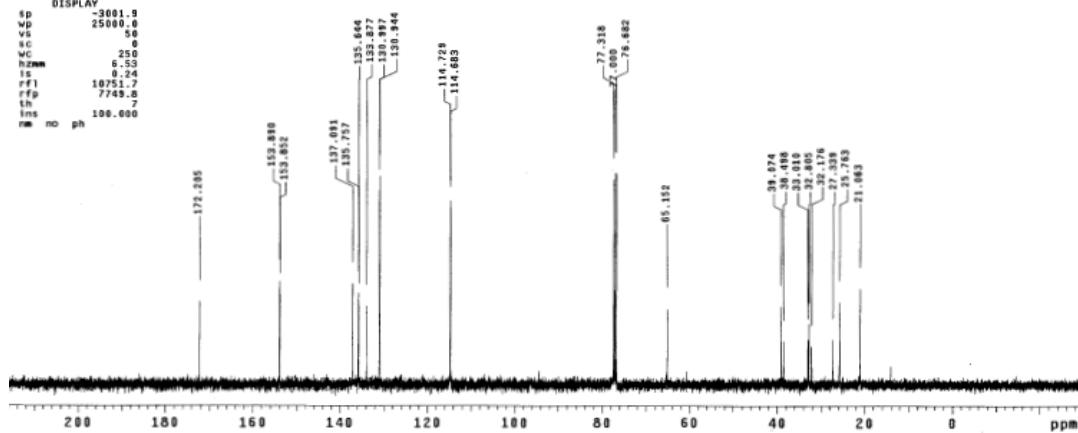
```
exp2 std1h
SAMPLE DEC. & VT
date Jan 19 2005 dfrq 400.267
solvent CDCl3 dn H1
file exp dpwr 30
ACQUISITION dof 0
sfrq 400.267 dn nnn
tn 3.741 dmf 200
nt 64 dsw 1.0
sw 5000.0 dss 1.0
fb not used homo n
pw 3.0 wtfle 1.0
dpw 2.000 fn not used
d1 0 math f
t0f 0
ct 12 werr n
clock 12 wexp n
gain not used wbs wmt
FLAGS n
ii n
in y
dp nn
hs nn
DISPLAY
sp -75.2
wp 410.5
vs 150
sc 0
nc 256
hzmw 3.12
ts 238.47
r1f 3885.6
rfp 2905.9
th 7
lms 2.000
nm ph
```



¹³C NMR (100 MHz, CDCl₃)

3-(3-Acetoxy-*n*-propyl)-1-[bis(4-hydroxyphenyl)methylene]cyclohexane (30c)

```
exp1 std13c
SAMPLE DEC. & VT
date Jan 19 2005 dfrq 400.267
solvent CDCl3 dn H1
file exp dpwr 38
ACQUISITION dof 0
sfrq 100.656 dn nny
tn C13 dmw w
at 1.000 dmw 10000
dp 55500.0 dsw 1.0
sw 25000.0 dss 1.0
fb not used homo n
pw 53 lb 1.00
tpw 4.0 wtfle
dt 1.000 proc f
dof 0 fn not used
nt 1024 math f
ct 300
clock 12 werr n
gain not used wexp
FLAGS wbs wft bc ds dsca-
ii n le dpf
in y
dp wmt
hs nn
DISPLAY
sp 13001.9
wp 25000.0
vs 56
sc 0
wc 250
hzmw 6.53
ts 254
r1f 10751.7
rfp 7745.8
th 7
lms 100.000
nm no ph
```



3-(3-Acetoxy-*n*-propyl)-1-[bis(4-methoxymethoxyphenyl)methylene]cyclohexane (33c).

¹H NMR (400 MHz, CDCl₃)

3-(3-Acetoxy-*n*-propyl)-1-[bis(4-methoxymethoxyphenyl)methylene]cyclohexane (33c)

exp6 stdh

SAMPLE DEC. & VT

date Dec 2, 2004 dfrg 400.267

solvent CDCl₃ dfrg n1

rf1 55 dprw 38

ACQUISITION dof 0

sfreq 400.267 dm nnn

td 1.000 sec c

et 3.741 dcf 2.09

np 44932 dseq 1.0

sw 8000.0 dres n

tb not used n

bs 2 wtfinc

tpw1 55 winc

dt 2.000 fn not used ft

tof 0 math r

nt 16

cl 16 werr

clock n wexp

gain not used n whs

FLAGS wnt

11 n

1n n

dp y

hs nn

DISPLAY

sp -88.0

wp 4183.8

vs 150

sc 0

wc 250

zmax 8.2

ls 288.12

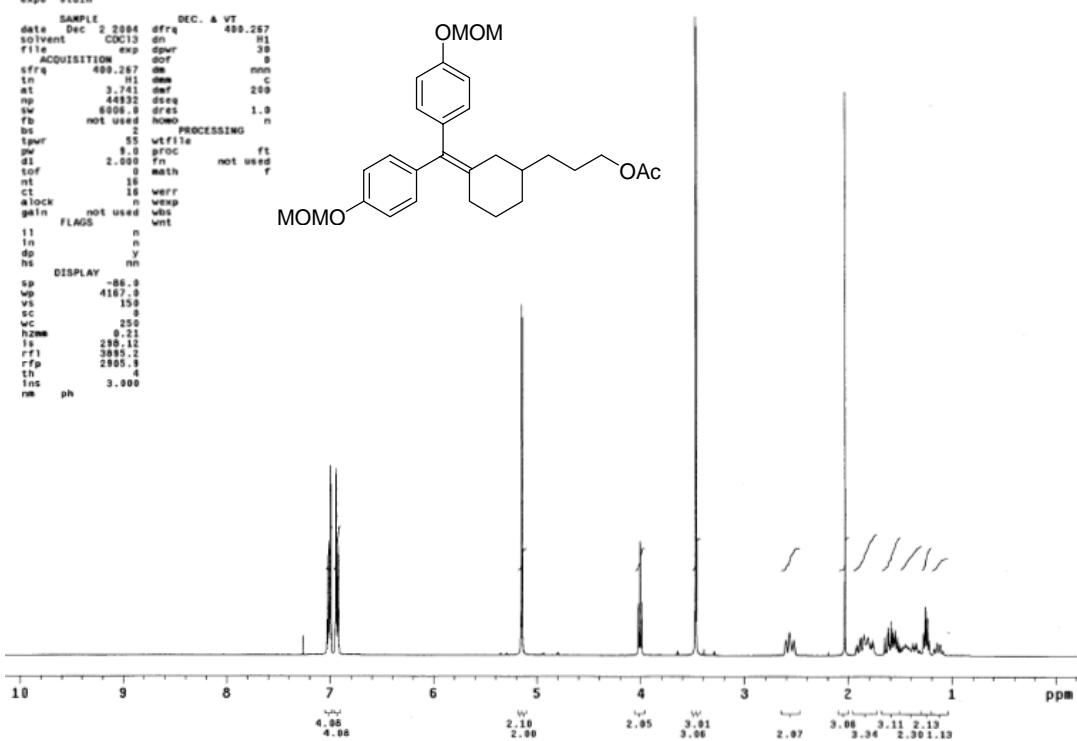
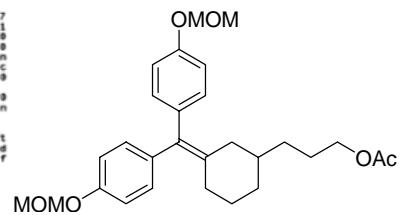
rfl1 3885.2

rfp 2885.8

th 4

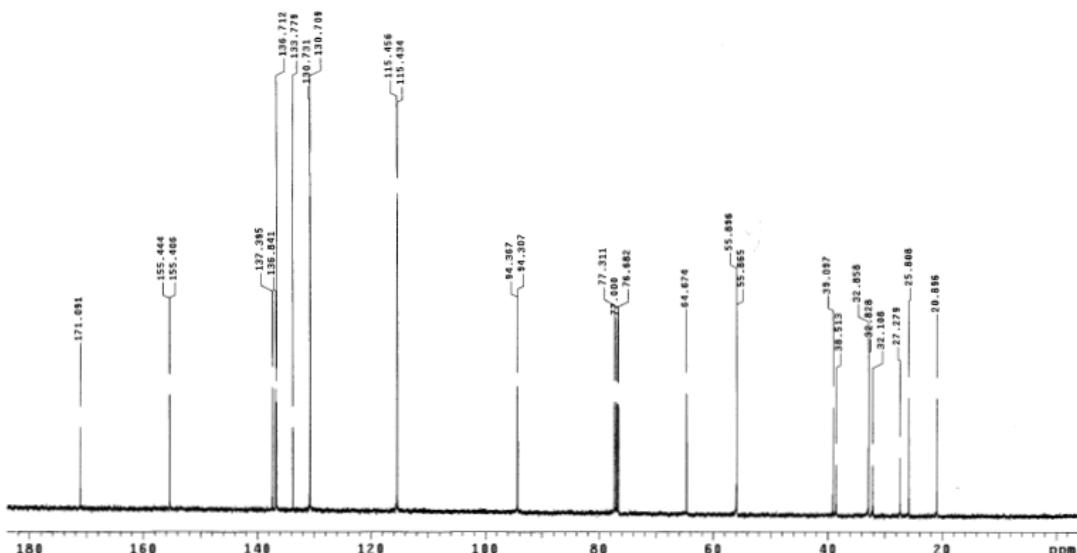
tns 3.000

nm ph



¹³C NMR (100 MHz, CDCl₃)

3-(3-Acetoxy-*n*-propyl)-1-[bis(4-methoxymethoxyphenyl)methylene]cyclohexane (33c)



3-(3-Hydroxypropyl)[bis(4-methoxymethoxyphenyl)methylene]cyclohexane (34c).

¹H NMR (400 MHz, CDCl₃)

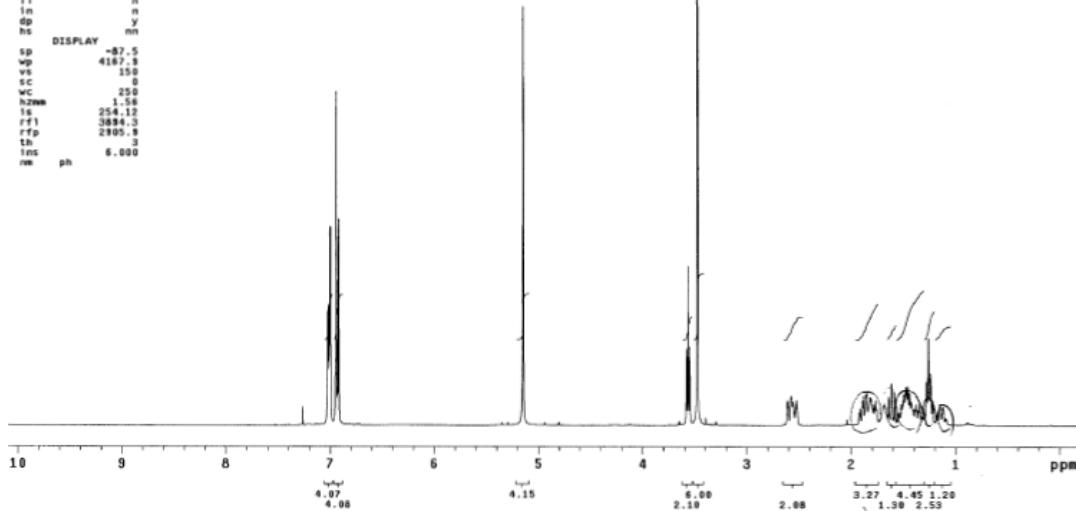
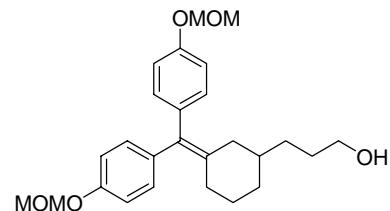
3-(3-Hydroxypropyl)[bis(4-methoxymethoxyphenyl)methylene]cyclohexane (34c)

exp6 std13c

```

SAMPLE          DEC. & VT
date Dec 6 2004 dfrq   400.267
solvent   CDCl3 dn      H1
file      exp dprw   38
          acq dof      8
sfreq    400.267 dm      nnn
tn       3.741 dmt     200
np      44532 dseq    1.0
sw      8000.0 dppr   1.0
rb       not used homn  n
bs        2 PROCESSING
tpwr      53 wtfille
pw       4.000 pwdec
dt       2.000 fn      not used
tof      0 math
nt       0
ct       12 vverr
alock     n wexp
gain     not used wbs
FLAGS
ii       n
in       n
dp       y
hs       nn
DISPLAY
sp      -400.5
wp      410.1
ve      150
sc       0
wc      250
ncnm
ts      1.58
is      254.12
rfl     3894.3
rfp     2905.1
tb      1.3
ins     6.000
nm no ph

```



¹³C NMR (100 MHz, CDCl₃)

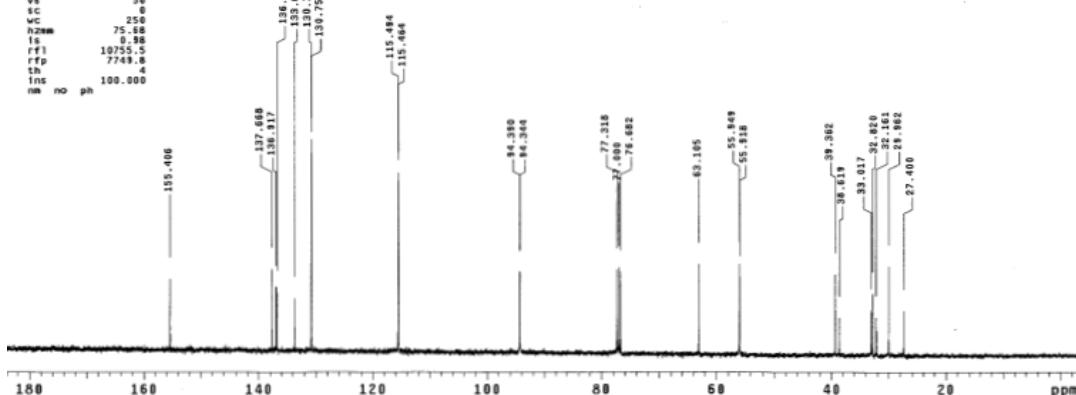
3-(3-Hydroxypropyl)[bis(4-methoxymethoxyphenyl)methylene]cyclohexane (34c)

exp6 std13c

```

SAMPLE          DEC. & VT
date Dec 6 2004 dfrs   400.267
solvent   CDCl3 dn      H1
file      exp dprw   38
          acq dof      8
sfreq    100.656 dm      nny
tn       1.193 dmt     10002
np      53958 dses    1.0
sw      25000.0 dres
rb       not used homn  n
bs        4 PROCESSING
tpwr      53 1b      1.00
pw       4.000 wtfille
dt       1.000 fn      not used
tof      0 math
nt       1000 meth
ct       150
alock     n vverr
gain     not used wexp
FLAGS
ii       n
in       n
dp       y
hs       nn
DISPLAY
sp      -400.5
wp      18913.9
ve      50
sc       0
wc      250
ncnm
ts      0.98
is      19755.5
rfl     7749.8
rfp     4
ins     100.000
nm no ph

```



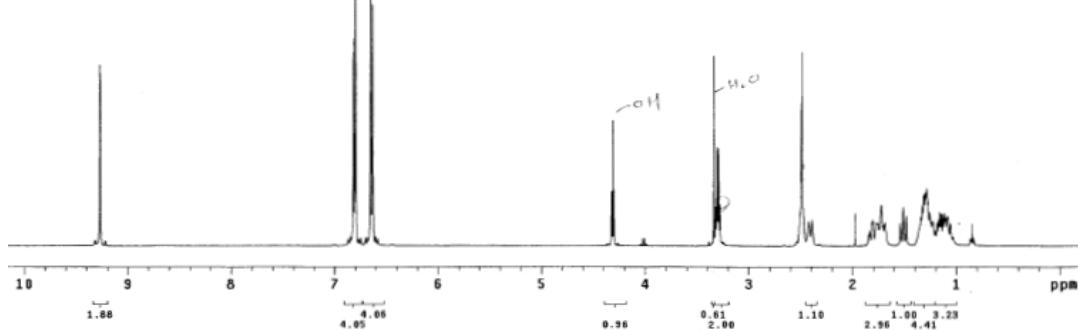
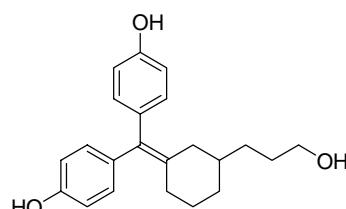
3-(3-Hydroxy-*n*-propyl)-1-[bis(4-hydroxyphenyl)methylene]cyclohexane (35c).

¹H NMR (400 MHz, DMSO-*d*₆)

3-(3-Hydroxy-*n*-propyl)-1-[bis(4-hydroxyphenyl)methylene]cyclohexane (35c)

exp5 std1h

```
SAMPLE           DEC. & VT
date Dec 1 2004 dfrq   400.249
solvent      DMSO dn    H1
file        exp dpwr   30
ACQUISITION dof    0
sfrq       400.263 dm   nnn
tn         3.741 def   200
np        44332 dcs   1.0
sw        6000.0 Hz
fb        8000.0 homo
bs          2
PROCESSING
tpwr      55 wtf file
pw        50 proc
d1     2.000 fn   not used f
tof      0 math
nt       16 werr
ct       16 wexp
alock     n wexp
gain      not used was
FLAGS      n wmt
ii        n
in        n
dp        y
ns        nn
DISPLAY
sp      -185.0
vp      416.0
vt      79
sc      0
nc      100
hzw   0.84
ls      303.21
rfi    1982.5
r1p   991.7
th     2.0
ins   2.000
nm   ph
```

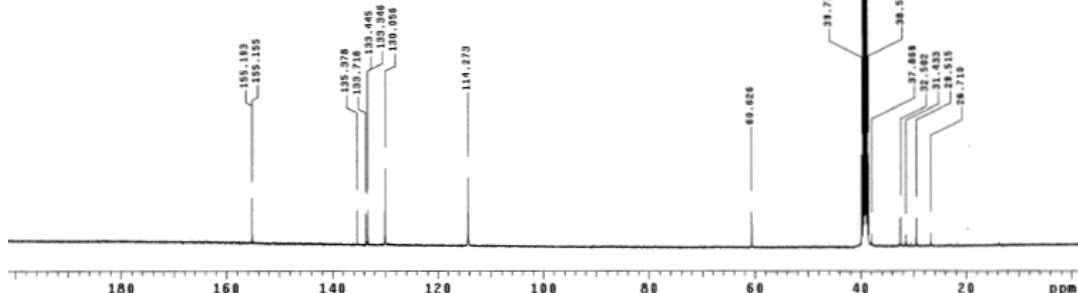


¹³C NMR (100 MHz, DMSO-*d*₆)

3-(3-Hydroxy-*n*-propyl)-1-[bis(4-hydroxyphenyl)methylene]cyclohexane (35c)

exp5 std13c

```
SAMPLE           DEC. & VT
date Dec 1 2004 dfrq   400.269
solvent      DMSO dn    H1
file        exp dpwr   38
ACQUISITION dof    0
sfrq       100.057 dm   nny
tn         1.193 def   10692 .
np        15000.0 dres  1.0
sw        2500.0 dres
fb        not used homo
bs          2
PROCESSING
tpwr      53 lb wtf file
pw        4.00 proc
d1     1.000 fn   not used f
tof      0 math
nt       15000 math
ct       15000
alock     n werr
gain      not used wexp
FLAGS      n wmt ds dscale
ii        n
in        n
dp        y
ns        nn
DISPLAY
sp      -185.0
vp      20553.5
vs      150
sc      0
nc      100
hzw   1.56
ls      500.00
rfi    7816.1
r1p   3946.3
th     3
ins   100.000
nm   no ph
```



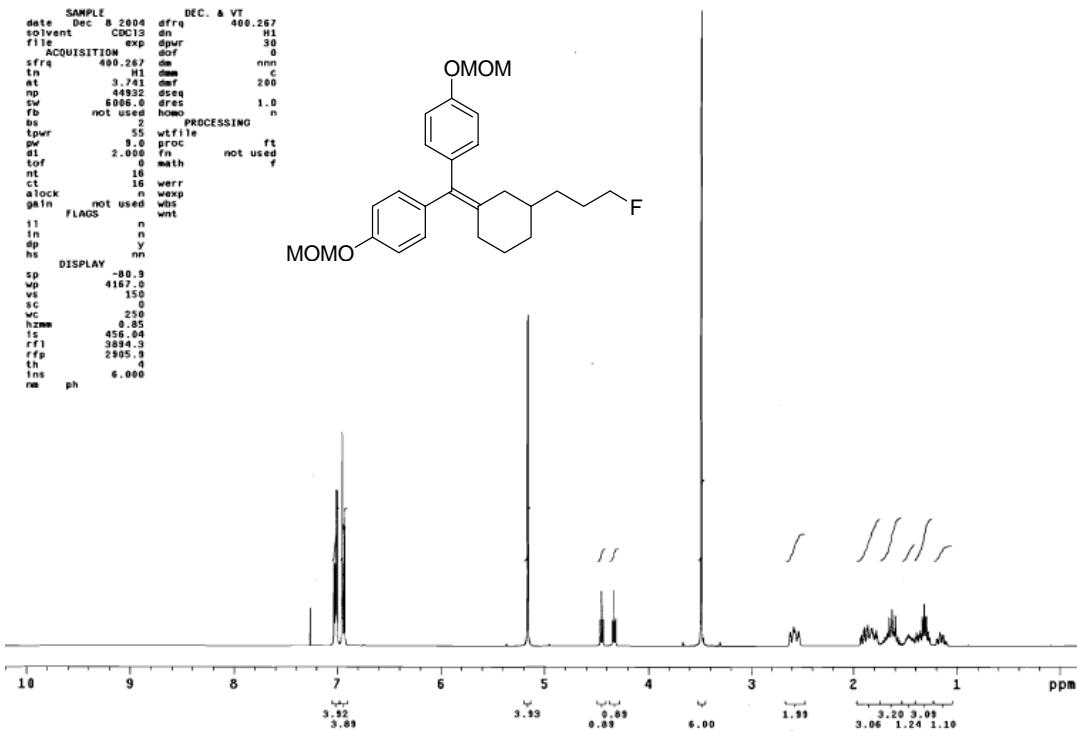
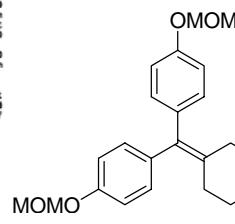
3-(3-Fluoropropyl)[bis(4-methoxymethoxyphenyl)methylene]cyclohexane.

¹H NMR (400 MHz, CDCl₃)

3-(3-Fluoropropyl)[bis(4-methoxymethoxyphenyl)methylene]cyclohexane

```
SAMPLE DEC. & VT
date Dec 8 2004 dfrq 400.267
solvent CDCl3 dn H1
file exp dpur 38
ACQUISITION dof 0
sfrq 400.267 dm nny
tn 3.741 dm c
at 3.741 dmf 260
np 44932 dfrq
sw 600.0 dres 1.0
fb not used homo n
bs 512 PROCESSING
lpwr 50 wfile
pw 9.0 proc ft
d1 2.000 fn not used
tof 16 math f
nt 16
ct 16 werr
alock 16 wexp
gain not used ws
gln 1.0 wnt
FLAGS
ii n
in Y
dp Y
hs nn
```

```
DISPLAY -80.3
sp 4167.0
vs 150
sc 0
nc 250
hzmw 0.85
ts 456.84
rf1 598.5
rf2 2905.9
th 4
ins 6.000
nm ph
```

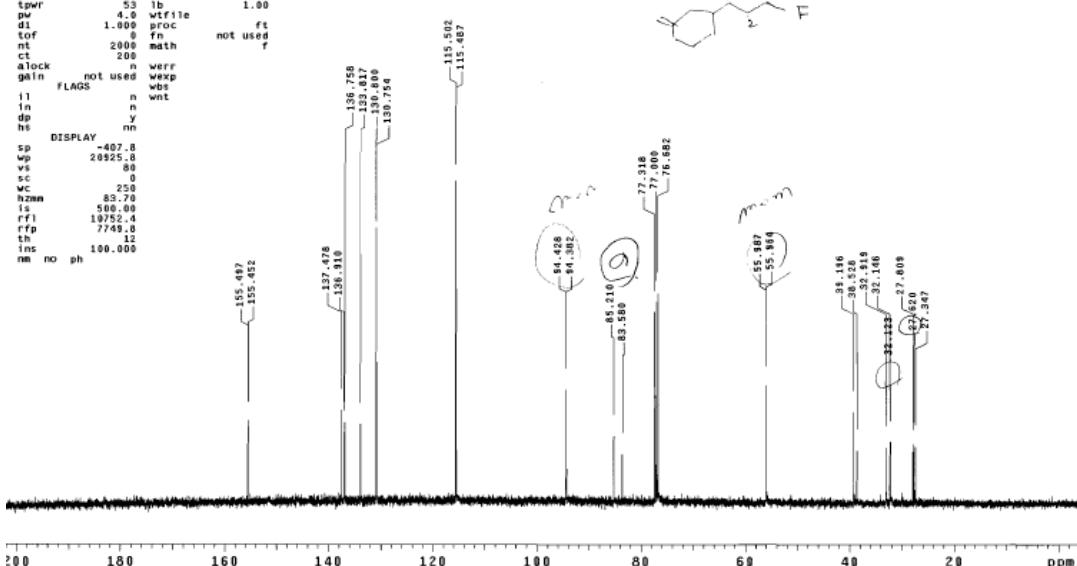
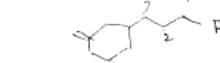


¹³C NMR (100 MHz, CDCl₃)

3-(3-Fluoropropyl)[bis(4-methoxymethoxyphenyl)methylene]cyclohexane

```
SAMPLE DEC. & VT
date Dec 8 2004 dfrq 400.267
solvent CDCl3 dn H1
file exp dpur 38
ACQUISITION dof 0
sfrq 100.656 dm nny
tn 1.193 dm v
at 1.193 dmf 10692
np 32720 dres
sw 2500.0 dres 1.0
fb not used homo n
bs 4 PROCESSING
lpwr 4.00 wfile
dw 1.000 proc ft
d1 1.000 fn not used
tof 2000 math f
nt 200
ct 200 werr
alock 1000 wexp
gain not used ws
gln 1.0 wnt
FLAGS
ii n
in n
dp Y
hs nn
```

```
DISPLAY -407.8
sp 20525.8
vs 80
sc 0
nc 250
hzmw 83.79
ts 500.00
rf1 19752.4
rf2 7749.0
th 12
ins 100.000
nm no ph
```



3-(3-Fluoro-*n*-propyl)-1-[bis(4-hydroxyphenyl)methylene]cyclohexane (37c).

¹H NMR (400 MHz, DMSO-*d*₆)

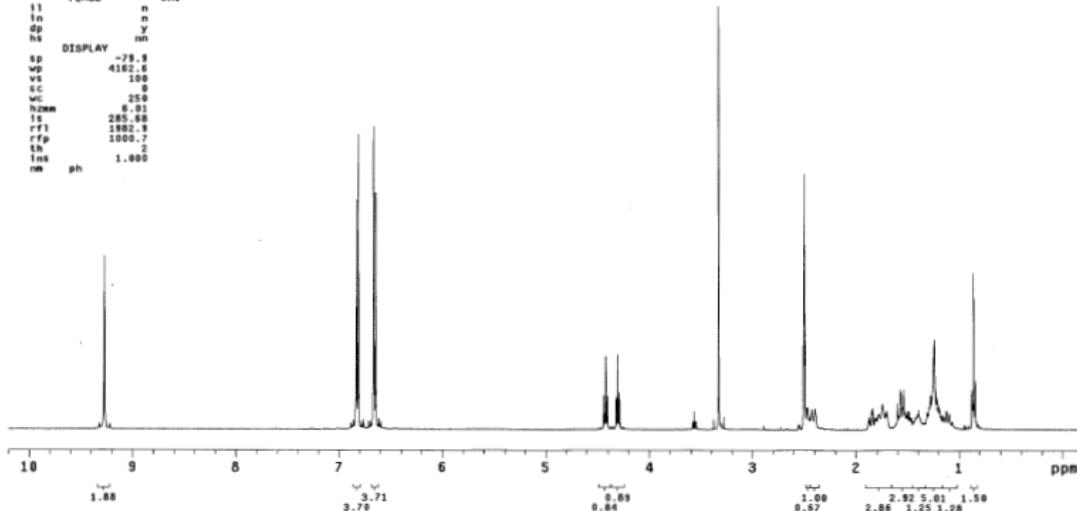
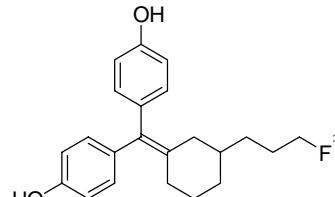
3-(3-Fluoro-*n*-propyl)-1-[bis(4-hydroxyphenyl)methylene]cyclohexane (37c)

```

exp4 std1h      SAMPLE          DEC. & VT
date Jul 13 2005 dfrq        400.2
solvent DMSO      dfrq
file      exp      dpwr
ACQUISITION dof
sfreq     400.263 def
psinc    90.0      psinc
t1       1.0       t1
at       3.743    def
np       44332   dsqeq
nd      6000.0    dres
sb      not used  sb
bs      2         homo
t1w      55       PROCESSING
pw      1.0       pw
ps      2.0000   proc
dt      0.000000 not us
tof      0.0       tof
nt      36        nt
ts      18        ts
slock   0.0       slock
gain    not used  gain
FLAGS    n         FLAGS
        n         n
        n         n
        y         hs
        nm        hs

DISPLAY
sp      -29.8
wp      4162.6
vs      198
c       100
wc      250
h2mz   6.91
t1      285.88
r1      1000.9
rfp     1000.7
th      1.0002
ts      1.000

```



¹³C NMR (100 MHz, acetone-*d*₆)

3-(3-Fluoro-*n*-propyl)-1-[bis(4-hydroxyphenyl)methylene]cyclohexane (37c)

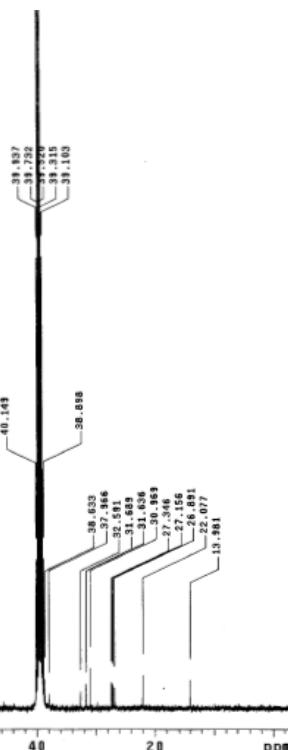
```

 3stns std13c

      SAMPLE           DEC. & VT
  date   Jul 13 2005 dftrs    400.168
  solvent   DMSO   dmsr     111
      exp   dgprw    38
      ACQUISITION dof      0
      sfrq    100.657
      at      C13    ame
      at     1.193    ame
      np      53636    dsqz
      wv     25000.0    dsw
      fb    not used    homo
      bs        4    PROCESSING
      tprw      53    ts
      prf     4.1    wf1f
      al     1.000    froc
      tof      20000    fn
      ct      38400    math
      clock      100    werr
      gain    not used    wexp
      FLAGS      n    wbs wft bc ds dsca-
      II       n    ts dpf
      ln       n    wnt
      dp       y
      hs       nn

      DISPLAY
      sp      -393.4
      wp     18911.6
      vs      258
      tc      9
      wc      258
      hzmn    11.42
      ts      0.12
      tr      701.5
      rfp     3977.6
      th      2
      ins     100.800
      ins     100.800

```



3 . HPLC purity data for compounds (37b and 37c)

37b was detected at 250 nm wavelength and **37c** at 254 nm wavelength.

Compd.	Column and Condition	Pressure	Flow rate	Room temperature	
				Retention time (min)	Purity (%)
37b	Alltech silica 5μ (250X4.6 mm)	540 psi	1 ml/min	9.95	>95%
	97% EtOAc/3% IPA				
37c	Phenomenex LUNA C8(2) 5μ (150X4.6 mm)	1168 psi	1 ml/min	11.8	>95%
	50% CH ₃ CN/50% H ₂ O				
	Alltech Econosil silica 10μ (250X10 mm)	1.9 MPa	3 ml/min	7.25	>95%
	100% EtOAc				
	RS Tech OP C18-51002546 (250X4.6 mm)	9.2 MPa	1 ml/min	9.55	>95%
	70% CH ₃ CN/30% H ₂ O				

HPLC trace of 37b

Normal Phase

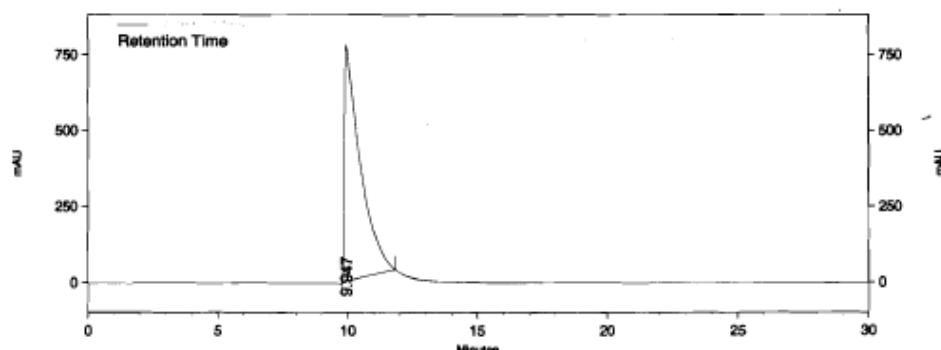
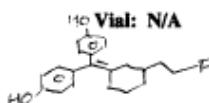
Alltech Alltima Silica 5 μ
Lot No AT0167
Part No 88111 Length 250mm

Area Percent Report Serial No. 01021121.1 ID 4.6mm Page 1 of 1
Data File: D:\Jay\DATA\cyclofenil\Jay_3-FEC_after_dry_normal_97%EA_3%IPA_1.dat
Acquired: 7/17/2004 11:33:18 AM Printed: 07/17/2004
02:00:48 PM

540PSI

Analyst: System

Sample ID: Jay_3-FEC_after_dry_normal_97%EA_3%IPA
Injection Volume: 0 20 μ L



Reverse Phase

P/N 007-4249-EO
phenomenex LUNA 5 μ C8(2)
150 x 4.6 mm
S/N 303709

Area Percent Report

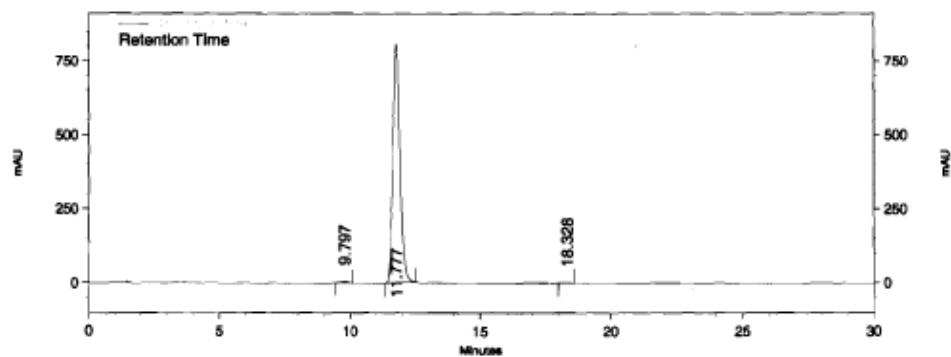
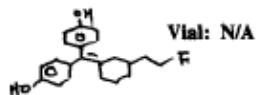
Data File: D:\Jay\DATA\cyclofenil\Jay_3-FEC_after_dry_reverse_50%ACN_50%H2O_1.dat
Acquired: 7/16/2004 1:16:19 AM Printed: 07/16/2004
01:56:05 AM

Page 1 of 1

1168PSI

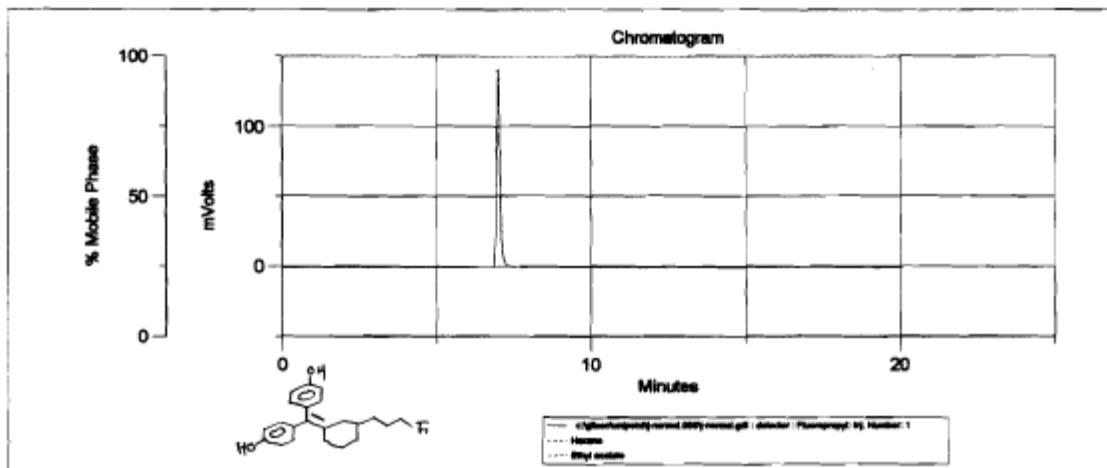
Analyst: System

Sample ID: Jay_3-FEC_after_dry_reverse_50%ACN_50%H2O
Injection Volume: 0 30 μ L 20 μ L



HPLC trace of 37c

Normal Phase

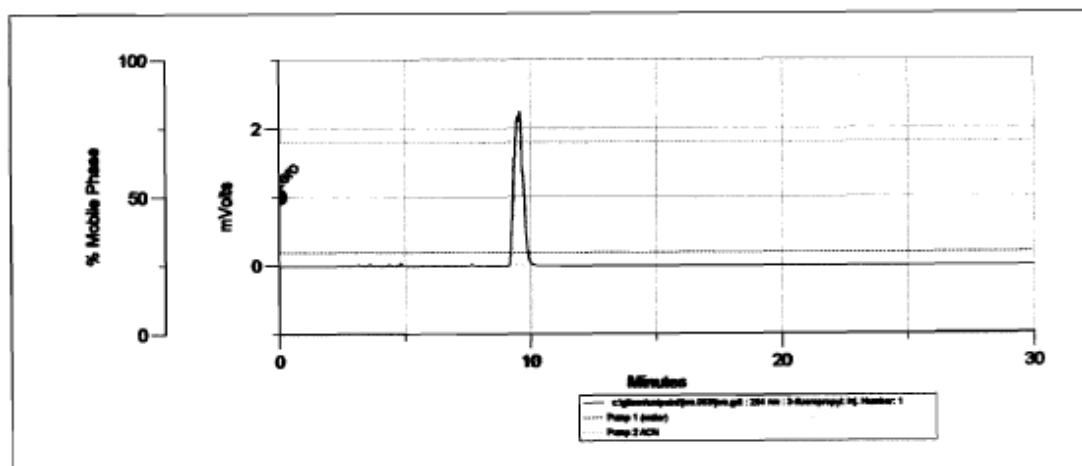


Column statue

Alltech
Normal phase
Cat. No.: Econosil Silica 10μ
250mm x 10 mm

1.9 Mpa.
100% EA
3 mL/min.

Reverse Phase



Column statue

RS tech
reverse phase
Cat. No. OP C18-51002546
250 mm X 4.6 mm

70% CH₃CN

9.2 Mpa.

30% H₂O

flow 1mL/min

70% CH₃CN 30% H₂O

flow rate: 1mL/min

