Efficient and Divergent Synthesis of Cyclophosphamide Analogues from 2-Arylamino-3-acetyl-5,6-dihydro-4*H*-pyrans

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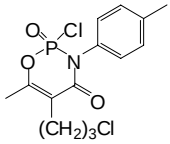
Supporting Information

I.General

All reagents were purchased from commercial sources and used without treatment, unless otherwise indicated. The products were purified by column chromatography over silica gel. ^{1}H NMR and ^{13}C NMR spectra were recorded at 25 °C at 500 MHz and 125 MHz, respectively, with TMS as internal standard. IR spectra (KBr) were recorded on FTIR-spectrophotometer in the range of 400-4000 cm^{-1}. Mass spectra were recorded on LCMsD mass spectrometer.

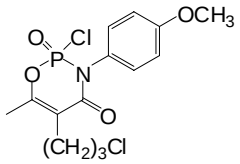
II. Analytical data of 3

3b



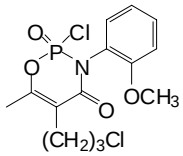
White solid: mp 75-76 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ = 1.94-2.04 (m, 2H), 2.32 (s, 3H), 2.39 (s, 3H), 2.51-2.65 (m, 2H), 3.54-3.62 (m, 2H), 7.197 (d, *J* = 8.0 Hz, 2H), 7.289 (d, *J* = 8.0 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ = 18.8, 21.4, 24.5, 31.4, 44.4, 114.3, 128.9, 129.0, 130.2, 130.6, 140.0, 156.9; IR (KBr) 1694.5, 1662.4, 1511.8, 1303.9, 1246.1, 1129.5, 1090.8, 990.2, 620.5, 562.3 cm^{-1}; Anal. Calcd for $C_{14}H_{16}Cl_2NO_3P$: C, 48.30; H, 4.63; N, 4.02. Found: C, 48.23; H, 4.58; N, 4.10.

3c



White solid: mp 92-93 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ = 1.95-2.00 (m, 2H), 2.31 (s, 3H), 2.53-2.61 (m, 2H), 3.55-3.59 (m, 2H), 3.83 (s, 3H), 6.985 (d, *J* = 9.0 Hz, 2H), 7.219-7.239 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ = 18.7, 24.5, 31.4, 44.6, 55.8, 114.2, 115.2, 124.1, 130.4, 157.1, 160.5, 163.3; Anal. Calcd for $C_{14}H_{16}Cl_2NO_4P$: C, 46.17; H, 4.43; N, 3.85. Found: C, 46.31; H, 4.38; N, 3.92.

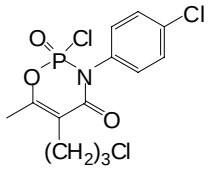
3d



White solid: mp 84-86 °C; ^{1}H NMR (500 MHz, CDCl3) δ = 1.96-2.02 (m, 2H), 2.30 (s, 3H), 2.53-2.66 (m, 2H), 3.56-3.60 (m, 2H), 3.82 (s, 3H), 7.01-7.06 (m, 2H), 7.33 (d, *J* = 7.0 Hz, 2H), 7.40-7.43(m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ = 18.8, 20.5, 31.4, 44.4, 56.1, 112.7, 114,5, 120.6,

121.4, 129.3, 131.4, 156.3, 157.1, 162.4; Anal. Calcd for $C_{14}H_{16}Cl_2NO_4P$: C, 46.17; H, 4.43; N, 3.85. Found: C, 46.05; H, 4.39; N, 3.79.

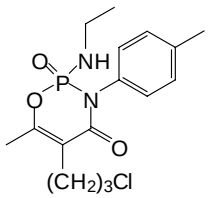
3e



White solid: mp 67-68 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ = 1.91-2.00 (m, 2H), 2.31 (s, 3H), 2.49-2.62 (m, 2H), 3.54-3.59 (m, 2H), 7.256 (d, *J* = 7.5Hz, 2H), 7.451 (d, *J* = 7.5 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ = 18.8, 24.6, 31.4, 44.5, 114.3, 130.2, 130.5, 130.7, 135.9, 157.4, 162.8; Anal. Calcd for $C_{13}H_{13}Cl_3NO_3P$: C, 42.36; H, 3.56; N, 3.80. Found: C, 42.54; H, 3.61; N, 3.67.

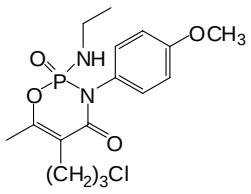
III. Analytical data of 4

4b-1



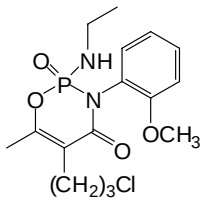
White solid: mp 121-122 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ = 0.99 (t, *J* = 7.0 Hz, 3H), 1.93-1.97 (m, 2H), 2.25 (s, 3H), 2.37 (s, 3H), 2.42-2.56 (m, 2H), 2.81-2.95 (m, 3H), 3.55 (t, *J* = 6.0 Hz, 2H), 7.22 (s, 4H); ^{13}C NMR (125 MHz, $CDCl_3$) δ = 16.7, 19.0, 21.3, 24.4, 31.7, 36.7, 44.9, 111.8, 128.6, 130.1, 131.6, 138.6, 157.1, 165.1; IR (KBr) 3207.4, 1673.3, 1439.6, 1390.2, 1253.0, 111.9, 1076.7, 987.3, 655.2, 567.7 cm^{-1}; Anal. Calcd for $C_{16}H_{22}ClN_2O_3P$: C, 53.86; H, 6.22; N, 7.85. Found: C, 53.98; H, 6.28; N, 7.79.

4c-1



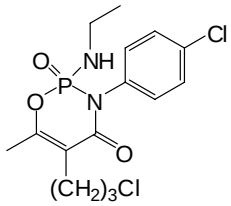
White solid: mp 122-123 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ = 0.99 (t, *J* = 7.0 Hz, 3H), 1.91-1.97 (m, 2H), 2.24 (s, 3H), 2.42-2.54 (m, 2H), 2.82-2.94 (m, 2H), 3.01-3.06 (m, 1H), 3.55 (t, *J* = 6.0 Hz, 2H), 3.82 (s, 3H), 6.94 (d, *J* = 8.5 Hz, 2H), 7.25 (d, *J* = 8.5 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ = 16.7, 19.0, 24.4, 31.7, 36.6, 44.9, 55.7, 111.7, 114.7, 126.8, 130.1, 157.2, 159.6, 165.3; Anal. Calcd for $C_{16}H_{22}ClN_2O_4P$: C, 51.55; H, 5.95; N, 7.51. Found: C, 51.69; H, 5.87; N, 7.44.

4d-1



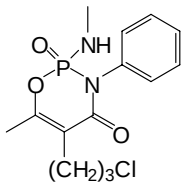
Semi-solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ = 0.86 (t, *J* = 6.5 Hz, 3H), 1.93-1.98 (m, 2H), 2.24 (s, 3H), 2.44-2.55 (m, 2H), 2.87-2.98 (m, 3H), 3.56 (t, *J* = 6.5 Hz, 3H), 3.82 (s, 3H), 6.98 (d, *J* = 8.0 Hz, 1H), 7.03 (t, *J* = 7.5 Hz, 1H), 7.37 (t, *J* = 7.5 Hz, 1H), 7.46 (d, *J* = 7.5 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ = 16.8, 18.7, 24.1, 31.5, 36.3, 44.7, 55.7, 111.3, 111.6, 121.2, 122.9, 130.0, 130.4, 155.4, 156.9, 163.7; Anal. Calcd for $C_{16}H_{22}ClN_2O_4P$: C, 51.55; H, 5.95; N, 7.51. Found: C, 51.68; H, 5.89; N, 7.46.

4e-1



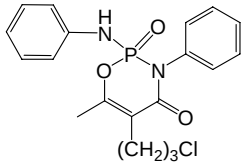
White solid: mp 128-129 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ = 1.00 (t, *J* = 7.0 Hz, 3H), 1.94-1.95 (m, 2H), 2.26 (s, 3H), 2.43-2.56 (m, 2H), 2.79-3.02 (m, 3H), 3.56 (t, *J* = 6.0 Hz, 2H), 7.29 (d, *J* = 8.5 Hz, 2H), 7.41 (d, *J* = 8.5 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ = 16.8, 19.0, 24.4, 31.7, 36.8, 44.9, 111.9, 129.7, 130.3, 133.0, 134.6, 157.5, 164.8; Anal. Calcd for $C_{15}H_{19}Cl_2N_2O_3P$: C, 47.76; H, 5.08; N, 7.43. Found: C, 47.69; H, 5.15; N, 7.35.

4a-2



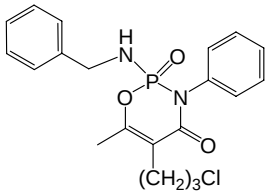
White solid: mp 131-132 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ = 1.96 (s, 2H), 2.27 (s, 3H), 2.46-2.55 (m, 5H), 2.98 (s, 1H), 3.55 (t, *J* = 6.0 Hz, 2H), 7.36 (d, *J* = 7.0 Hz, 2H), 7.40 (d, *J* = 7.0 Hz, 1H), 7.43 (d, *J* = 7.5 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ = 18.9, 24.4, 27.7, 31.7, 44.9, 111.9, 128.7, 128.9, 129.5, 134.4, 157.4, 165.0; Anal. Calcd for $C_{14}H_{18}ClN_2O_3P$: C, 51.15; H, 5.52; N, 8.52. Found: C, 51.27; H, 5.47; N, 8.59.

4a-3



White solid: mp 192-193 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ = 1.97-2.02 (m, 2H), 2.31 (s, 3H), 2.52-2.64 (m, 2H), 3.59 (t, *J* = 6.0 Hz, 2H), 6.30 (s, 1H), 6.71 (d, *J* = 7.5 Hz, 2H), 7.01 (d, *J* = 6.5 Hz, 2H), 7.07 (t, *J* = 7.5 Hz, 1H), 7.16 (t, *J* = 7.5 Hz, 1H), 7.25 (d, *J* = 8.0 Hz, 3H), 7.30 (d, *J* = 7.0 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ = 19.1, 24.5, 31.7, 44.9, 112.2, 118.5, 118.6, 123.4, 129.0, 129.3, 129.7, 133.3, 138.5, 157.2, 164.8; IR (KBr) 3181.6, 1677.1, 1501.1, 1418.7, 1389.7, 1266.9, 1133.4, 965.4, 748.7, 695.5 cm^{-1}; Anal. Calcd for $C_{19}H_{20}ClN_2O_3P$: C, 58.39; H, 5.16; N, 7.17. Found: C, 58.54; H, 5.21; N, 7.08.

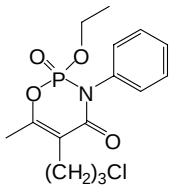
4a-4



White solid: mp 147-148 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ = 1.96 (s, 2H), 2.20 (s, 3H), 2.46-2.55 (m, 2H), 3.29 (s, 1H), 3.57 (t, *J* = 6.0 Hz, 2H), 4.00-4.07 (m, 2H), 7.03 (t, *J* = 7.5 Hz, 2H), 7.26 (s, 3H), 7.38 (d, *J* = 7.0 Hz, 2H), 7.44 (d, *J* = 7.5 Hz, 3H), ; ^{13}C NMR (125 MHz, $CDCl_3$) δ = 18.9, 24.4, 31.7, 44.9, 45.7, 111.9, 127.4, 127.9, 128.8, 128.9, 129.1, 129.7, 134.3, 138.0, 157.4, 164.9; Anal. Calcd for $C_{20}H_{22}ClN_2O_3P$: C, 59.34; H, 5.48; N, 6.92. Found: C, 59.46; H, 5.41; N, 6.87.

IV. Analytical data of 5

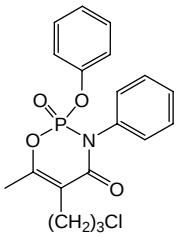
5a-2



White solid: mp 54-55 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ = 1.19-1.25 (m, 3H), 1.93-1.96 (m, 2H), 2.24 (s, 3H), 2.50 (t, *J* = 7.5 Hz, 2H), 3.53-3.56 (m, 2H), 4.11-4.18 (m, 2H), 7.31 (d, *J* = 7.5 Hz, 2H), 7.40 (d, *J* = 7.5 Hz, 1H), 7.44 (t, *J* = 7.5 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ = 16.3, 18.8, 31.6, 44.7, 66.2, 112.2, 129.1, 129.4, 129.7, 133.3, 156.9, 164.2; IR (KBr) 2924.6, 1665.7, 1459.6, 1346.6, 1143.0, 1031.7, 986.9, 695.7, 554.3 cm^{-1}; Anal. Calcd for $C_{15}H_{19}ClNO_4P$: C, 52.41; H, 5.57; N, 4.07.

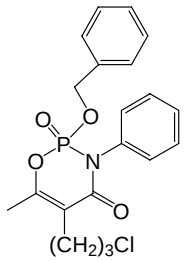
Found: C, 52.26; H, 5.63; N, 4.15.

5a-3



White solid: mp 117-118 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ = 1.93-1.99 (m, 2H), 2.27 (s, 3H), 2.50-2.62 (m, 2H), 3.53-3.61 (m, 2H), 7.04 (d, *J* = 7.5 Hz, 2H), 7.21 (t, *J* = 7.5 Hz, 1H), 7.31-7.34 (m, 4H), 7.41-7.48 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ = 18.8, 24.5, 31.6, 44.7, 113.0, 120.3, 126.3, 129.4, 129.5, 129.8, 130.3, 133.1, 150.2, 157.2, 164.1; IR (KBr) 1687.6, 1590.5, 1487.2, 1389.2, 1299.2, 1129.5, 1070.4, 955.4, 693.5, 529.1 cm^{-1}; Anal. Calcd for $C_{19}H_{19}ClNO_4P$: C, 58.25; H, 4.89; N, 3.58. Found: C, 58.37; H, 4.96; N, 3.51.

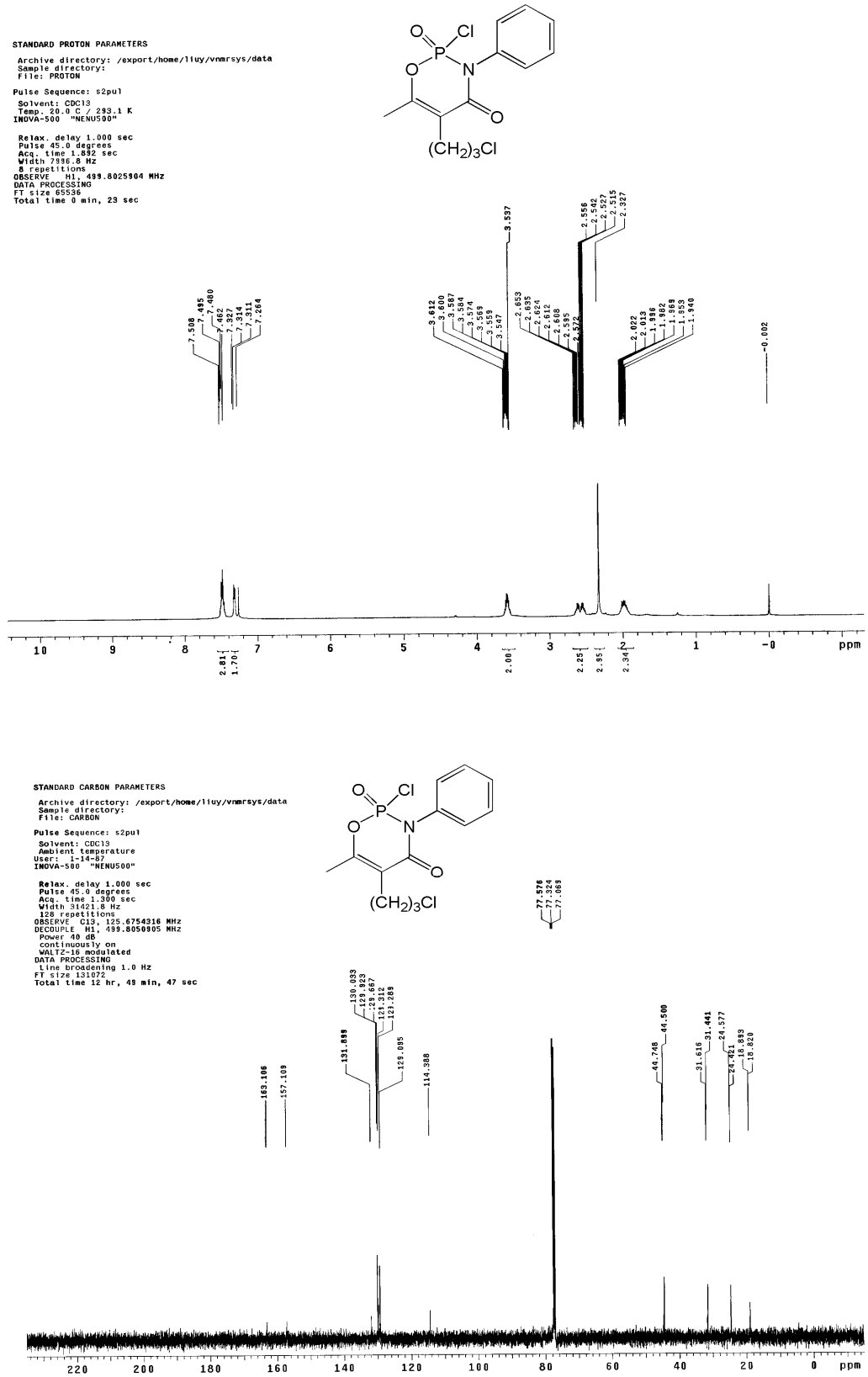
5a-4



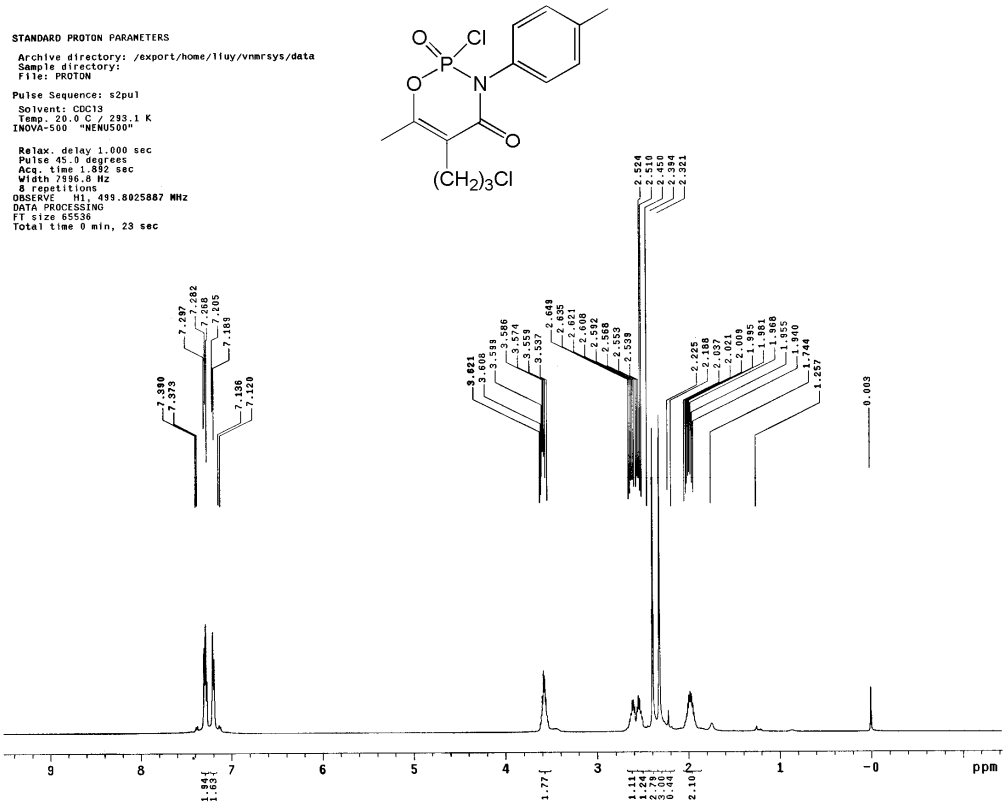
White solid: mp 75-76 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ = 1.91-1.96 (m, 2H), 2.06 (s, 3H), 2.44-2.54 (m, 2H), 3.49-3.56 (m, 2H), 5.02-5.17 (m, 2H), 7.26-7.27 (m, 4H), 7.34-7.35 (m, 3H), 7.40-7.41 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ = 18.2, 24.0, 31.2, 44.2, 70.9, 112.0, 128.2, 128.6, 128.7, 128.8, 129.0, 129.3, 132.7, 134.7, 156.5, 163.8; Anal. Calcd for $C_{20}H_{21}ClNO_4P$: C, 59.19; H, 5.22; N, 3.45. Found: C, 59.26; H, 5.17; N, 3.52.

V. Copies of NMR spectra for compounds 3-5

3a



3b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Sample directory:
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Pulse Sequence: s2pul
Solvent: CDCl3
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Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7996.8 Hz
8 repetitions
OBSERVE H1, 499.8025887 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
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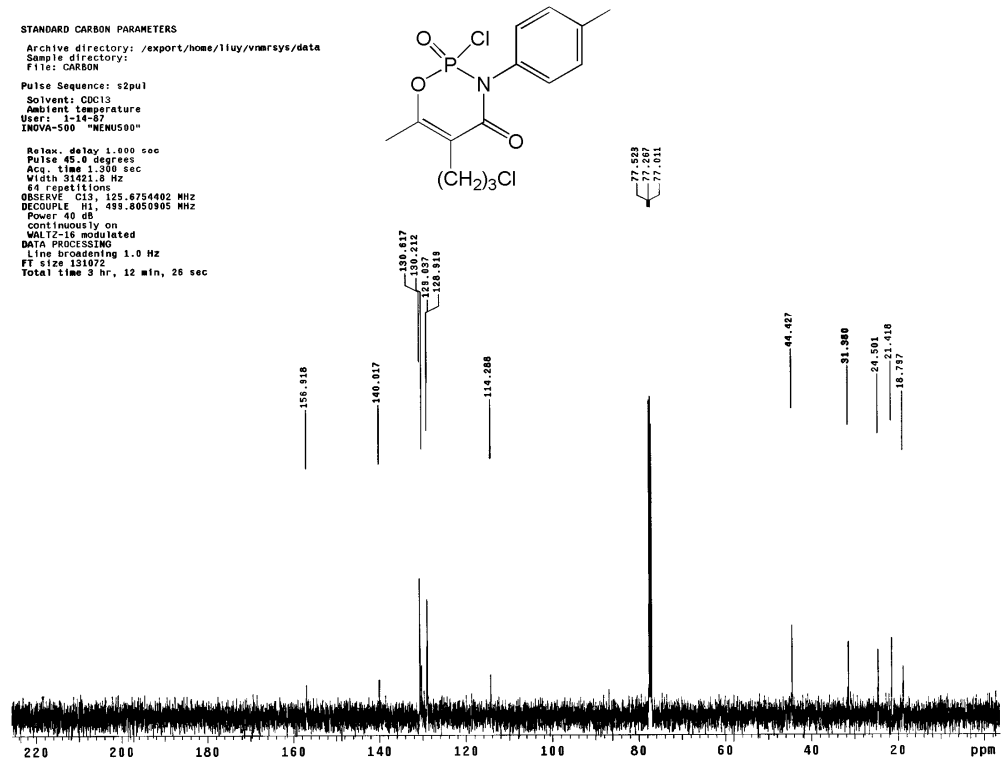
O
P
N
O
O
(CH2)3Cl
ppm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Sample directory:
File: CARBON
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Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
64 repetitions
OBSERVE C13, 125.6754402 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 3 hr, 12 min, 26 sec
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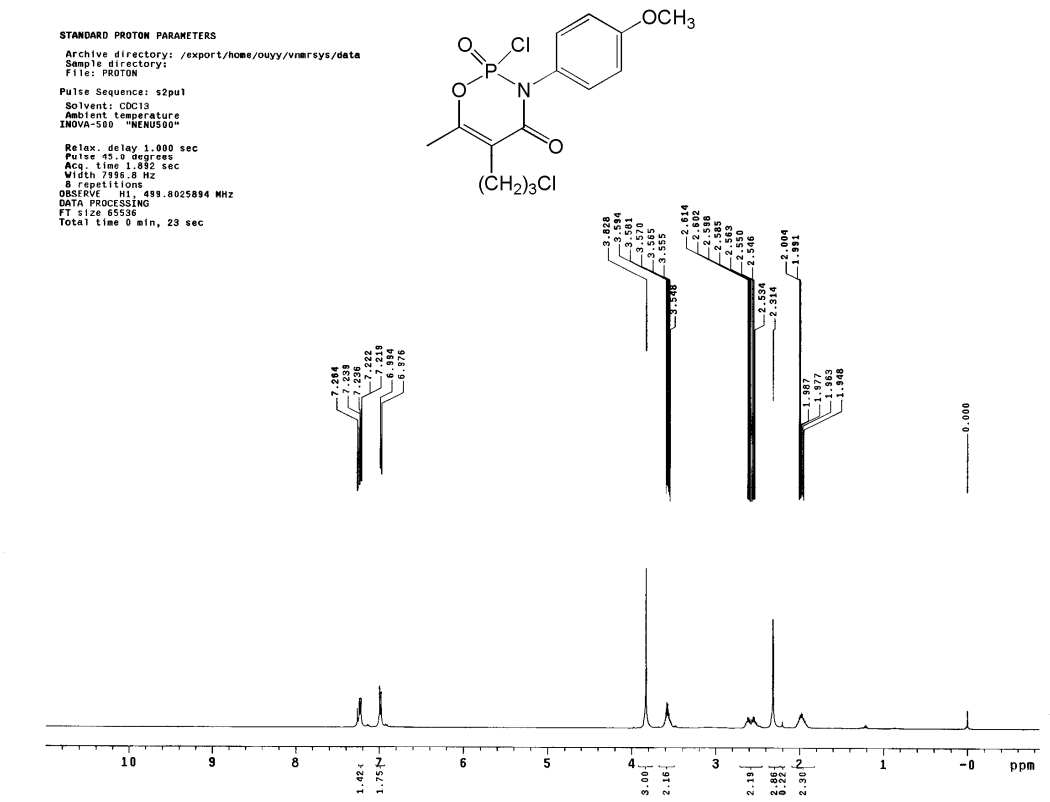
O
P
N
O
O
(CH2)3Cl
ppm

3c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Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7996.8 Hz
8 repetitions
OBSERVE H1, 499.8025894 MHz
DATA PROCESSING
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Total time 0 min, 23 sec
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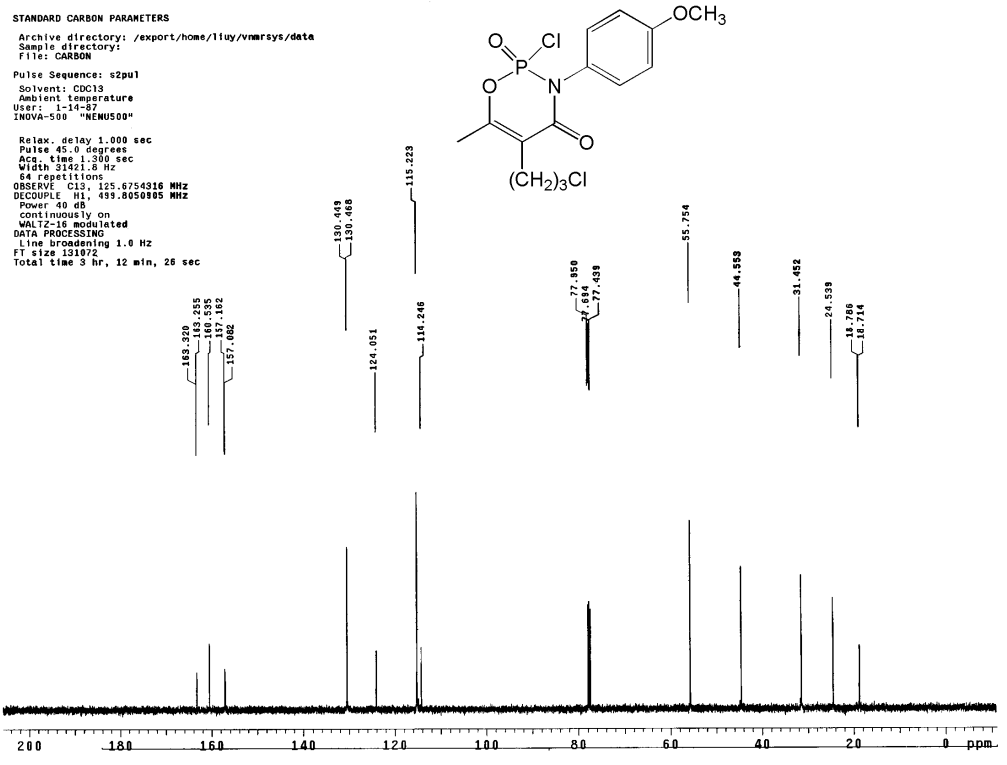
Cl
O
P
N
(CH2)3Cl
ppm


STANDARD CARBON PARAMETERS
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Sample directory:
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Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
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Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
64 repetitions
OBSERVE C13, 125.6754316 MHz
DECOUPLE H1, 499.8050805 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
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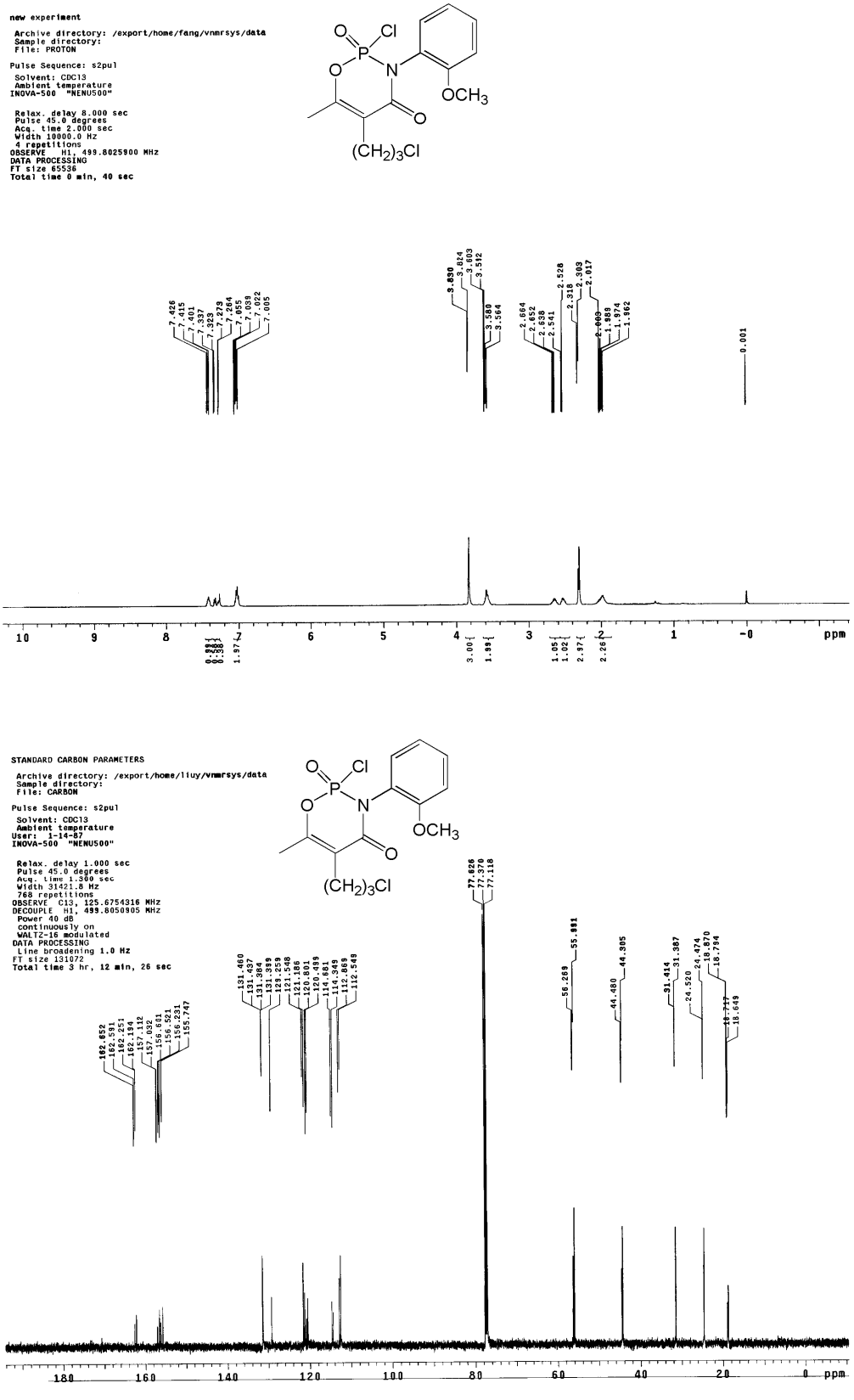
Cl
(CH2)3Cl
ppm

3d


new experiment
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Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "NENU500"
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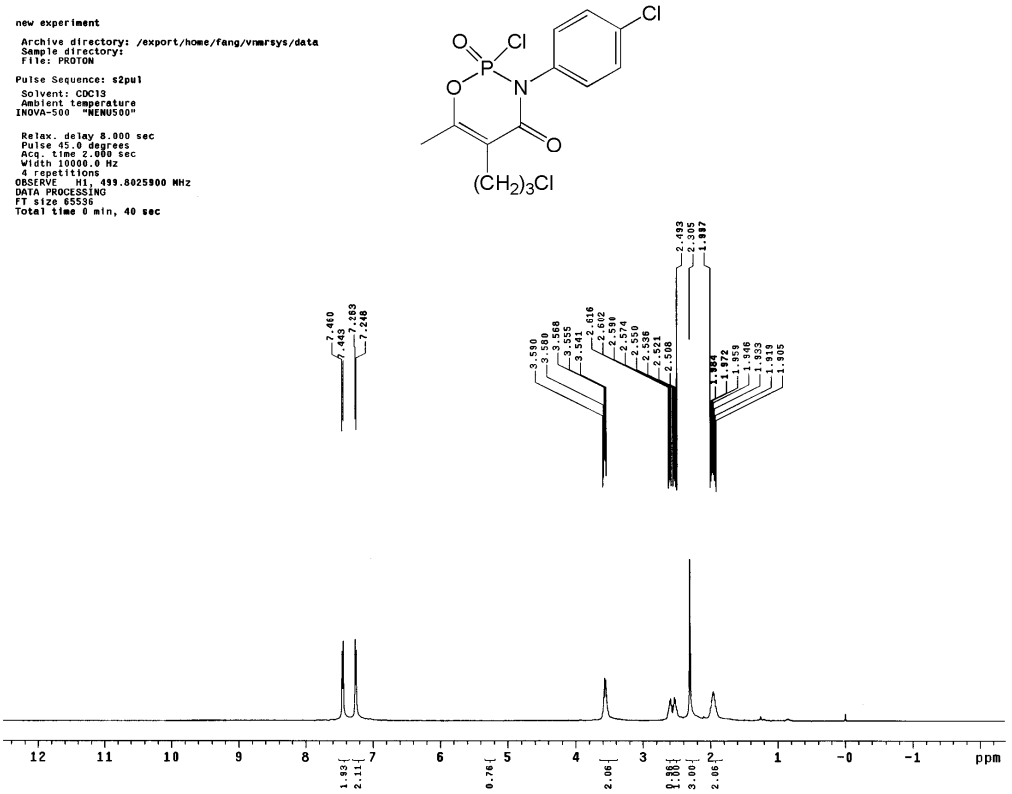
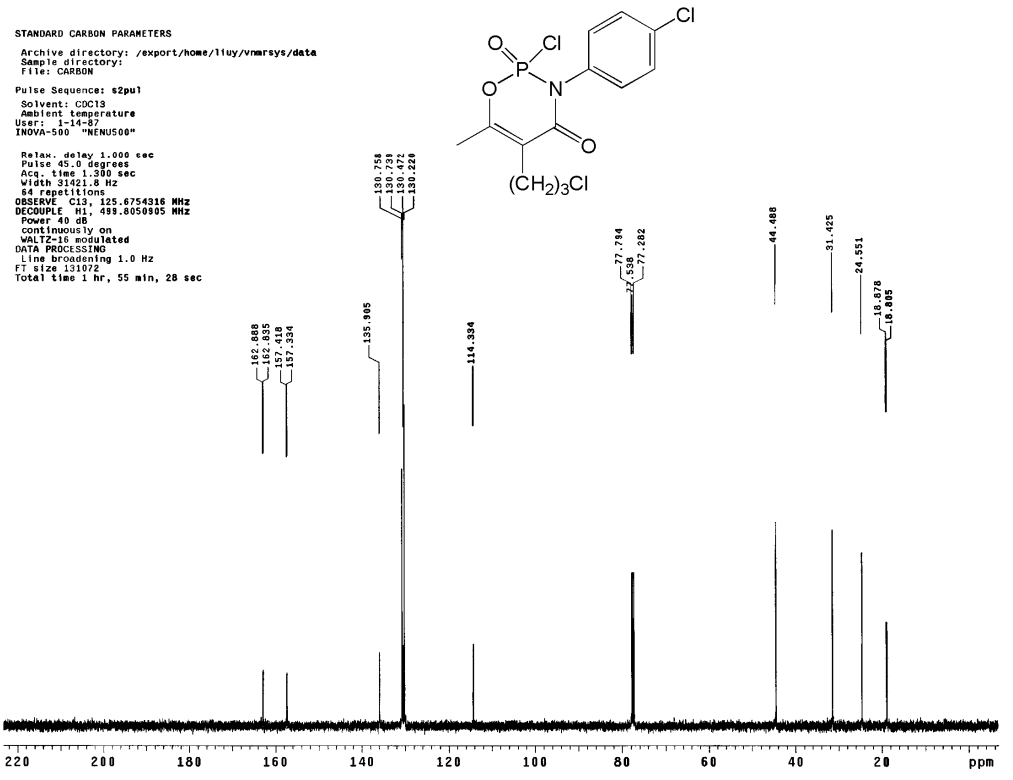
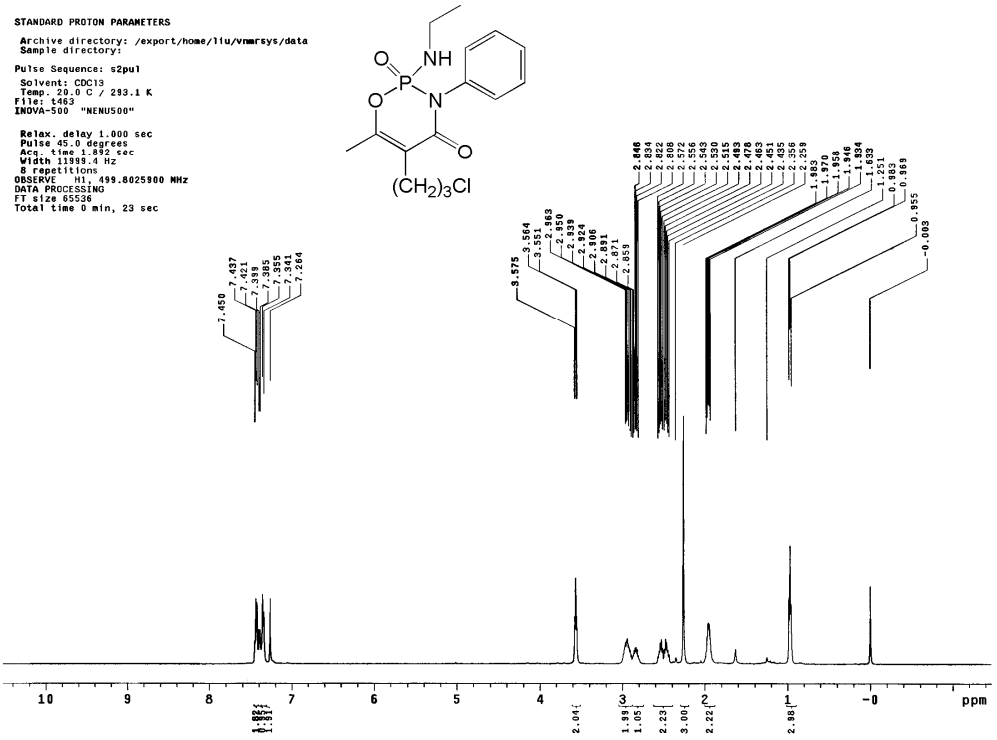
Acq. time 2.000 sec
Width 10000.0 Hz
4 repetitions
OBSERVE H1, 499.8025900 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 40 sec
STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
768 repetitions
OBSERVE C13, 125.6754316 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 3 hr, 12 min, 26 sec
ppm

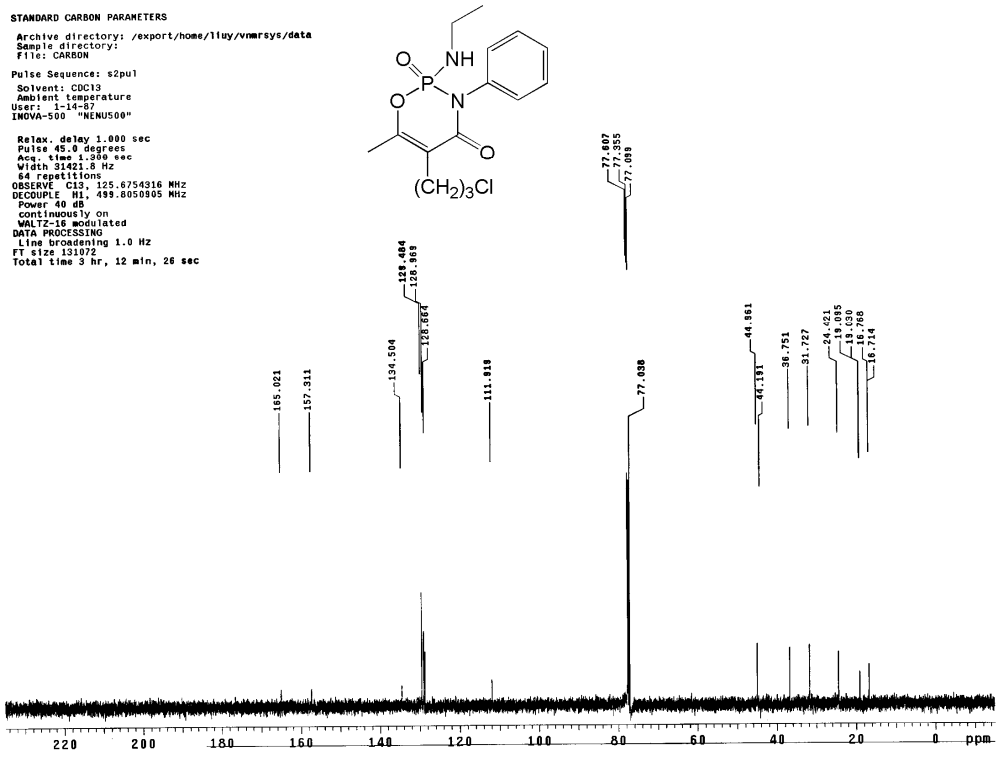
3e


new experiment
Archive directory: /export/home/fang/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "NENU500"
Relax. delay 8.000 sec
Pulse 45.0 degrees
Acq. time 2.000 sec
Width 10000.0 Hz
4 repetitions
OBSERVE H1, 499.8025900 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 40 sec
Cl
O
Cl
P
O
N
O
(CH2)3Cl
7.460
7.443
7.289
7.248
3.590
3.580
3.568
3.555
3.541
2.616
2.602
2.590
2.574
2.559
2.536
2.521
2.508
2.493
2.305
1.997
1.984
1.972
1.959
1.946
1.933
1.919
1.905
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm
1.93
2.11
0.76
2.06
1.00
3.00
2.05

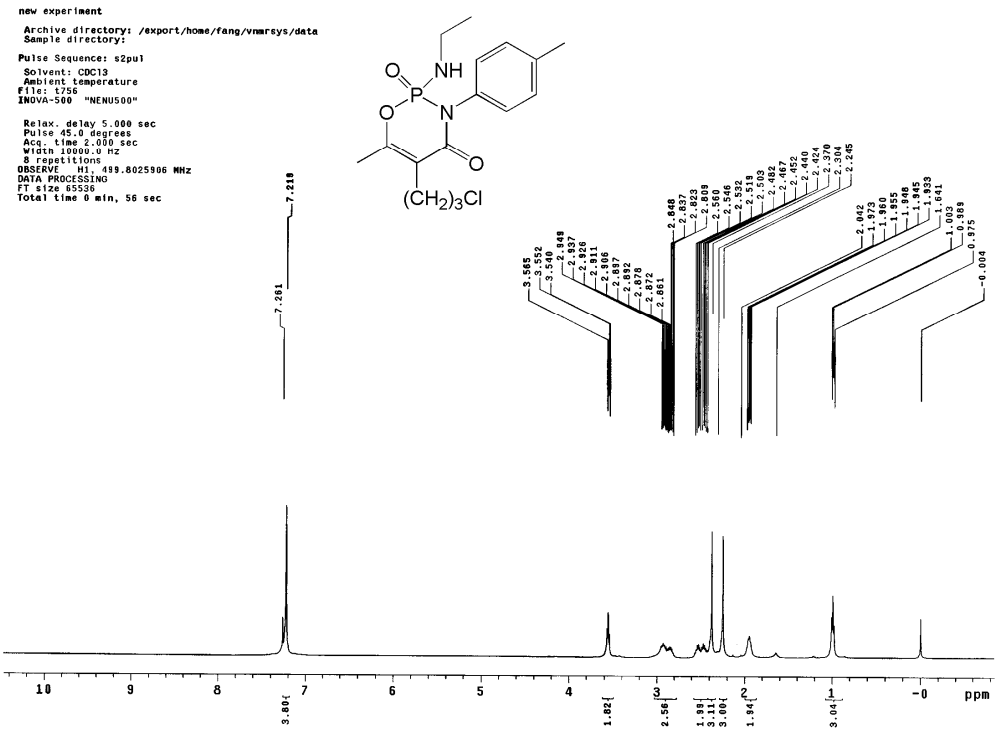

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
64 repetitions
OBSERVE C13, 125.6754316 MHz
DECOUPLE H1, 499.8050305 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 hr, 55 min, 28 sec
Cl
O
Cl
P
O
N
O
(CH2)3Cl
162.888
162.835
157.418
157.334
135.905
130.758
130.731
130.472
130.220
114.334
77.784
77.538
77.282
44.488
31.425
24.551
18.878
18.805
220
200
180
160
140
120
100
80
60
40
20
ppm

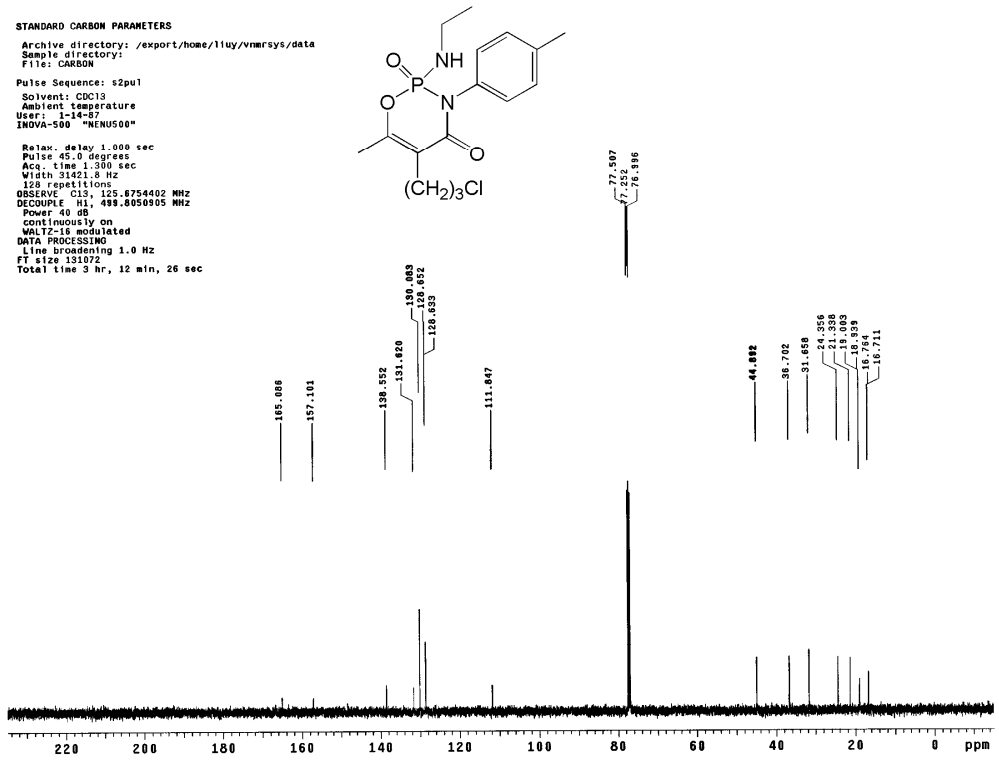
4a-1


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liu/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 20.0 C / 293.1 K
File: t463
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 11999.4 Hz
8 repetitions
OBSERVE H1, 499.8025900 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
NH
O
P
N
O
O
(CH2)3Cl
ppm

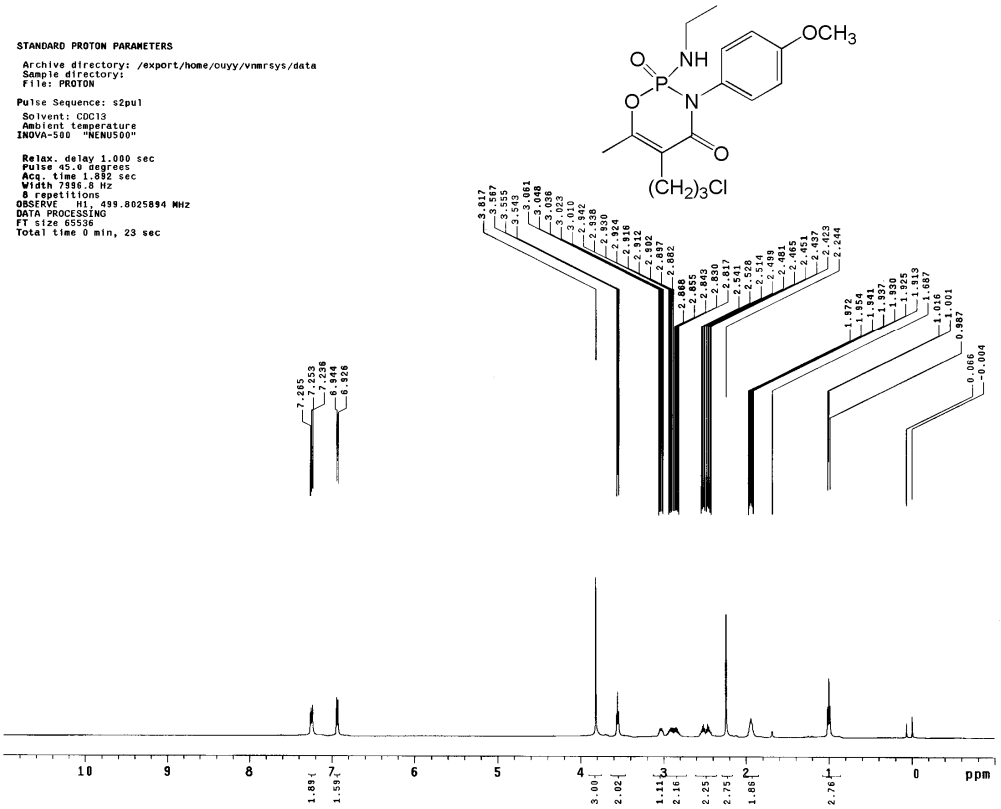

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
64 repetitions
OBSERVE C13, 125.6754316 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 3 hr, 12 min, 26 sec
NH
O
P
N
O
O
(CH2)3Cl
ppm

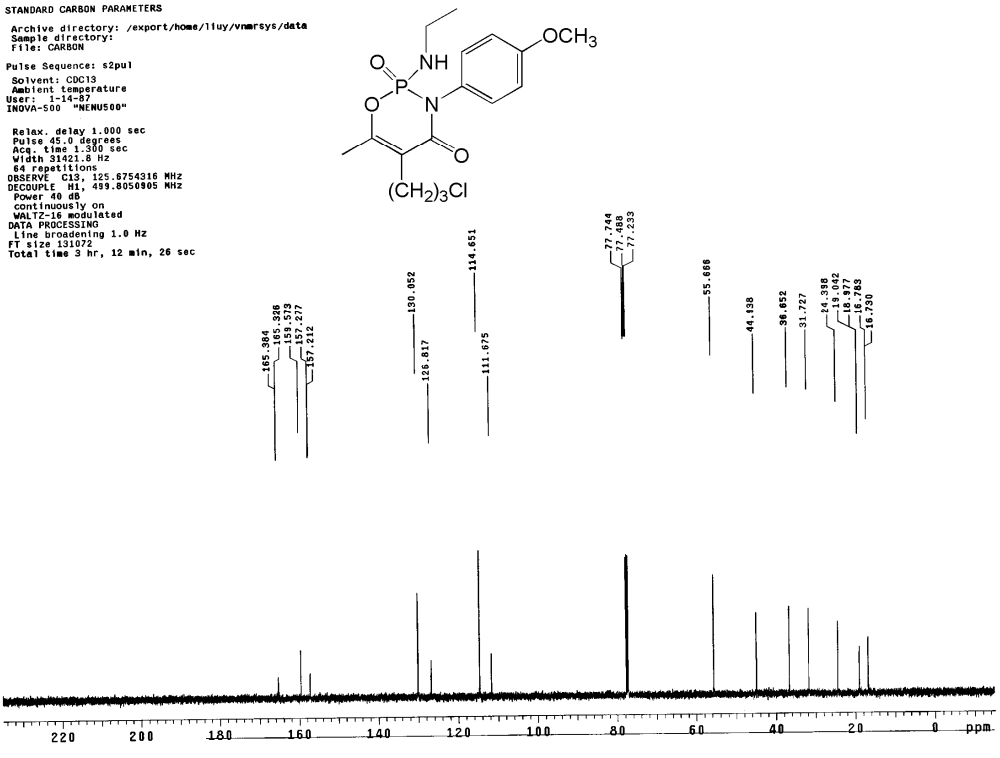
4b-1


new experiment
Archive directory: /export/home/fang/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: t756
INOVA-500 "NENU500"
Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 2.000 sec
Width 10000.0 Hz
8 repetitions
OBSERVE H1, 499.8025906 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 56 sec
NH
O
P
N
O
O
(CH2)3Cl
7.261
7.219
ppm

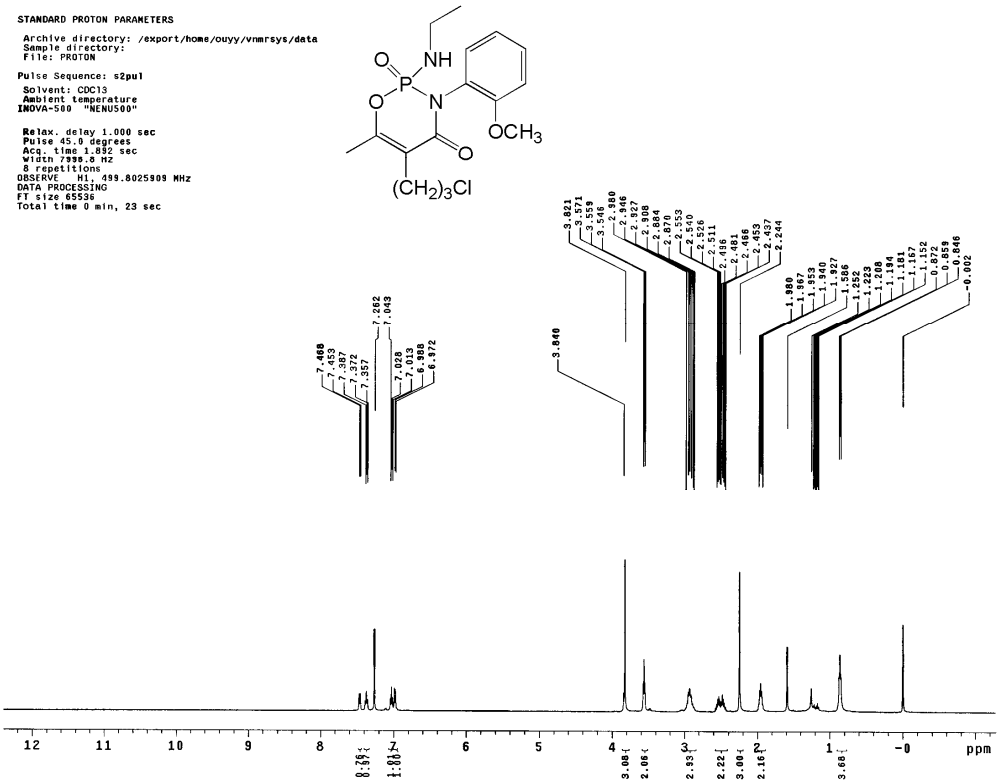

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-07
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13, 125.6754402 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 3 hr, 12 min, 26 sec
NH
O
P
N
O
O
(CH2)3Cl
77.507
77.192
76.876
165.086
157.101
138.552
131.620
130.083
128.652
128.633
111.847
44.882
36.702
31.658
24.356
24.338
19.083
18.939
16.784
16.711
ppm

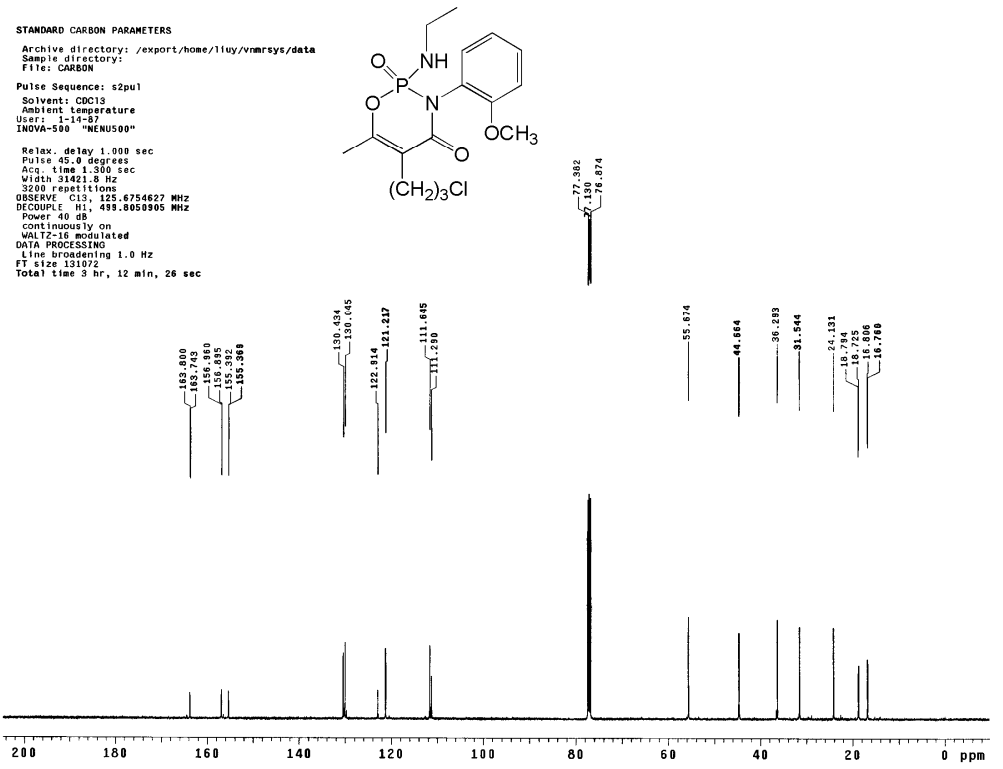
4c-1


STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.882 sec
Width 7996.8 Hz
8 repetitions
OBSERVE H1, 499.8025894 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
OCH3
NH
O
P
N
(CH2)3Cl
ppm

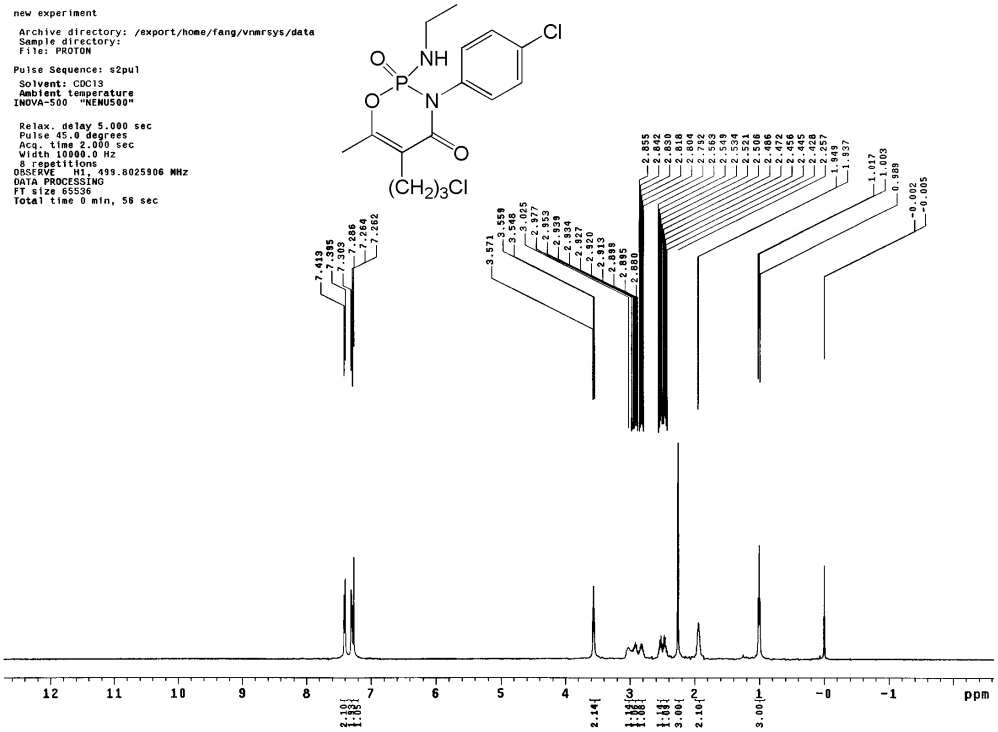

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
64 repetitions
OBSERVE C13, 125.8754316 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 3 hr, 12 min, 26 sec
OCH3
NH
O
P
N
(CH2)3Cl
ppm

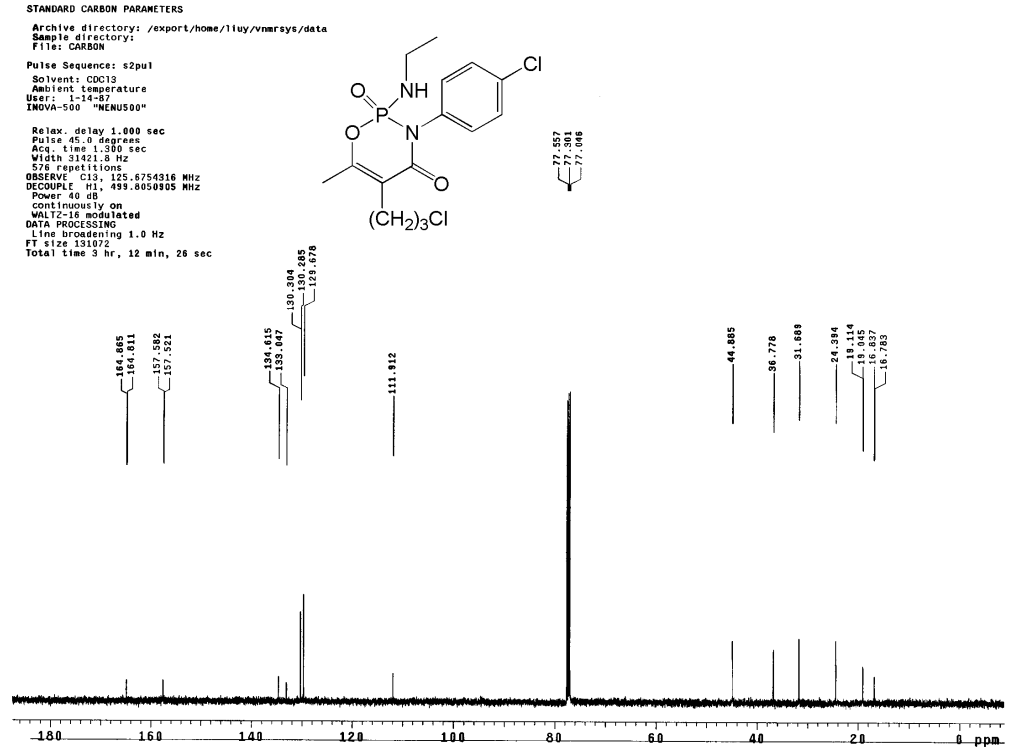
4d-1


STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7998.8 Hz
8 repetitions
OBSERVE H1, 499.8025909 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
NH
O
P
O
N
OCH3
O
(CH2)3Cl
ppm

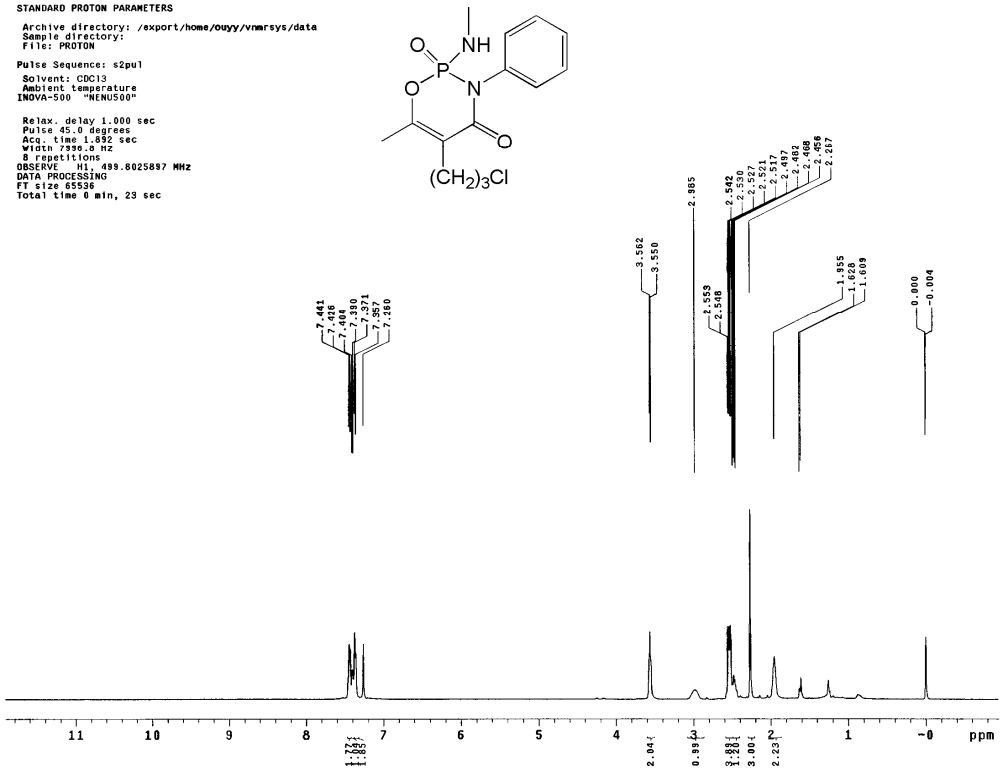

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
3200 repetitions
OBSERVE C13, 125.6754627 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 3 hr, 12 min, 26 sec
NH
O
P
O
N
OCH3
O
(CH2)3Cl
ppm

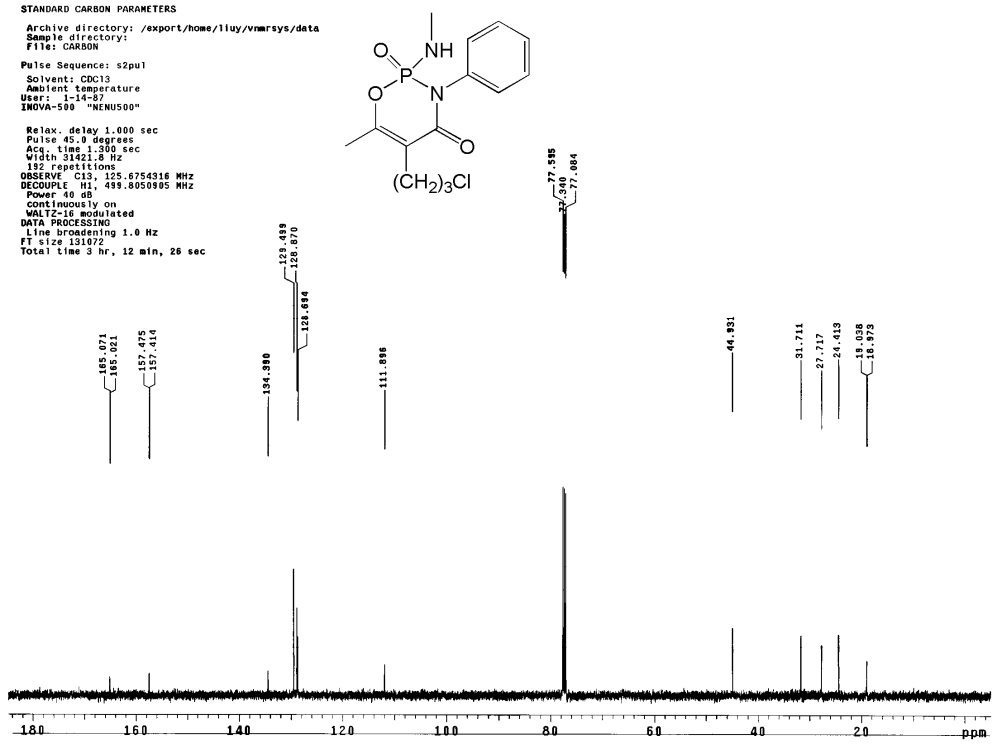
4e-1


new experiment
Archive directory: /export/home/fang/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "NENU500"
Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 2.000 sec
Width 10000.0 Hz
8 repetitions
OBSERVE H1, 499.8025906 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 58 sec
NH
P
O
N
Cl
(CH2)3Cl
ppm

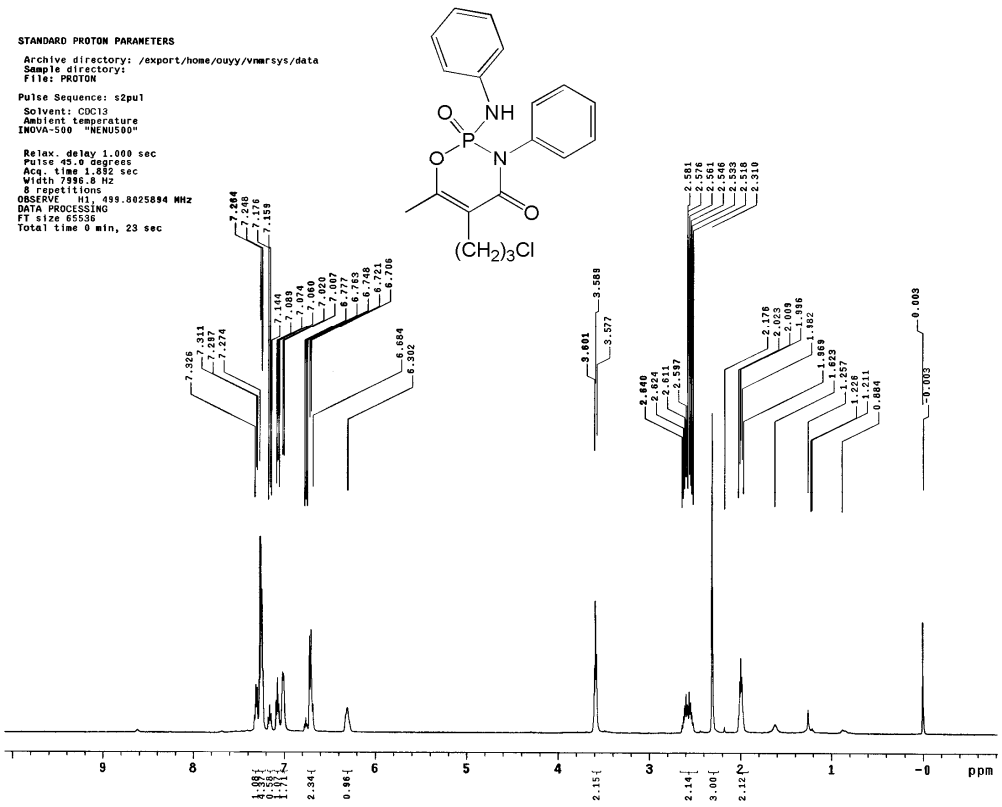

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
570 repetitions
OBSERVE C13, 125.6754316 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 3 hr, 12 min, 26 sec
NH
P
O
N
Cl
(CH2)3Cl
ppm

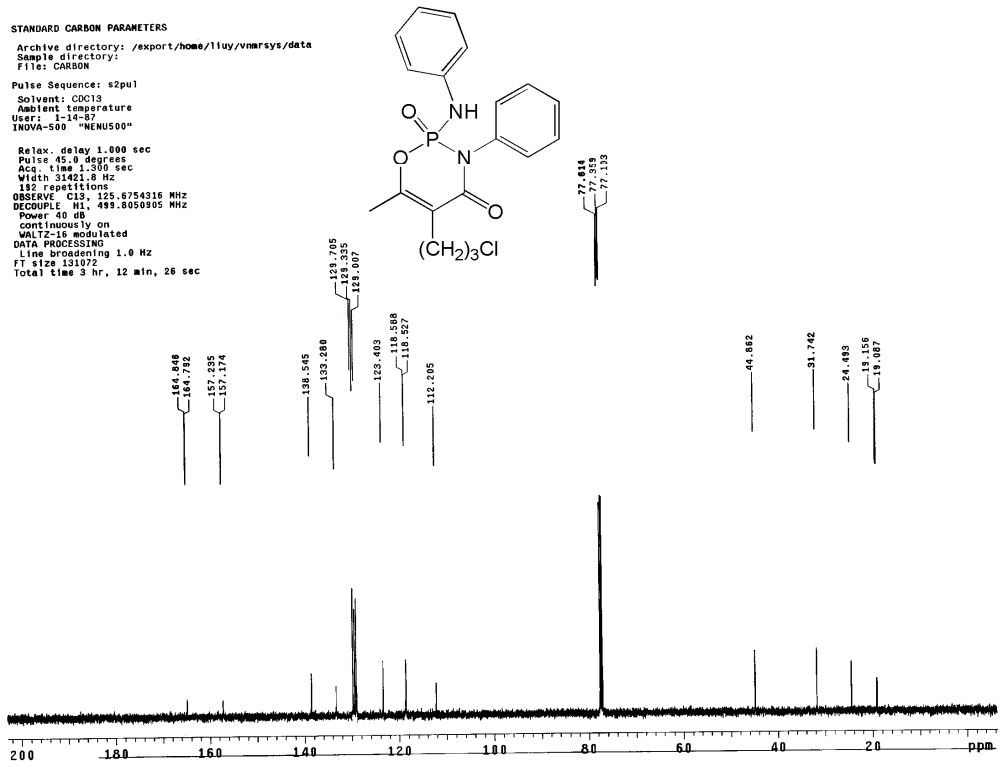
4a-2


STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7330.8 Hz
8 repetitions
OBSERVE H1, 499.8025897 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
NH
O
P
N
O
O
(CH2)3Cl
ppm

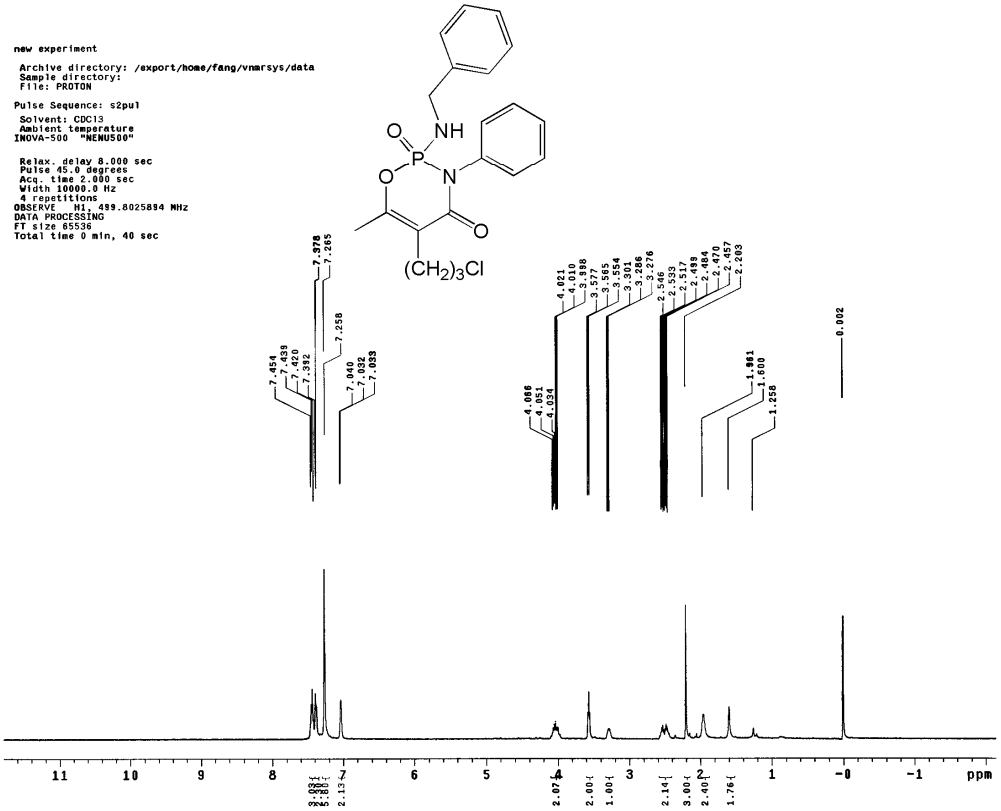

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
192 repetitions
OBSERVE C13, 125.6754316 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 3 hr, 12 min, 26 sec
NH
O
P
N
O
O
(CH2)3Cl
ppm

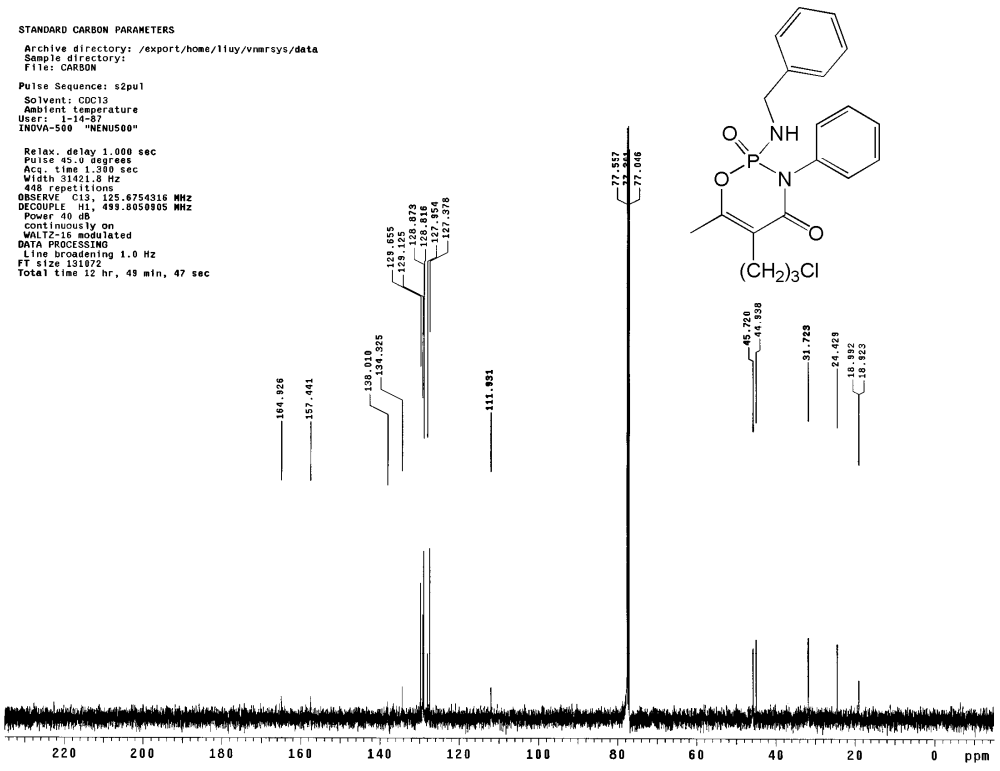
4a-3


STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.882 sec
Width 7996.8 Hz
8 repetitions
OBSERVE H1, 499.8025894 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
NH
O
P
N
O
O
(CH2)3Cl
ppm

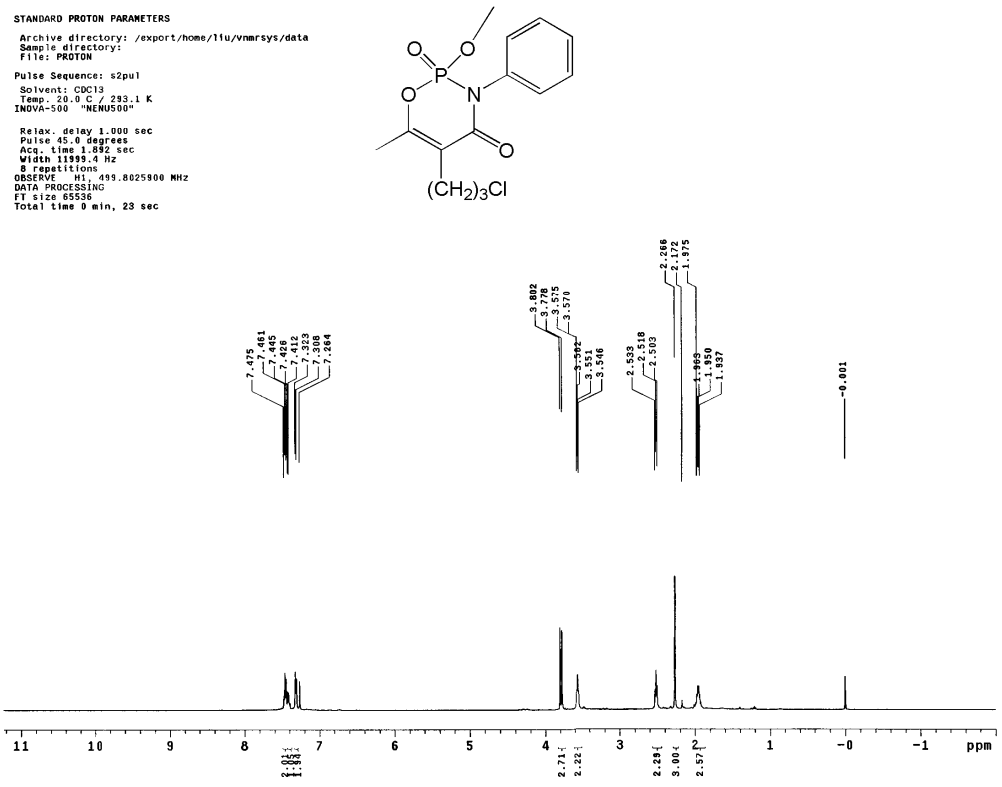

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
192 repetitions
OBSERVE C13, 125.6754316 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 3 hr, 12 min, 26 sec
NH
O
P
N
O
O
(CH2)3Cl
ppm

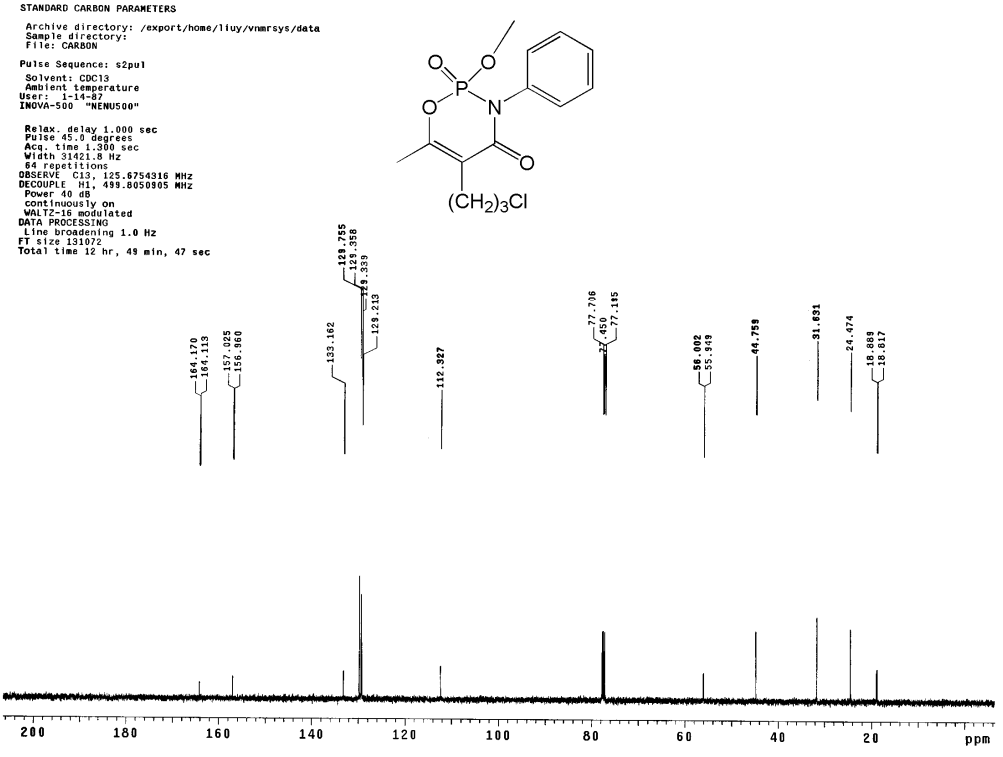
4a-4


new experiment
Archive directory: /export/home/fang/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "NENU500"
Relax. delay 8.000 sec
Pulse 45.0 degrees
Acq. time 2.000 sec
Width 10000.0 Hz
4 repetitions
OBSERVE H1, 499.8025894 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 40 sec
NH
O
P
N
(CH2)3Cl
ppm

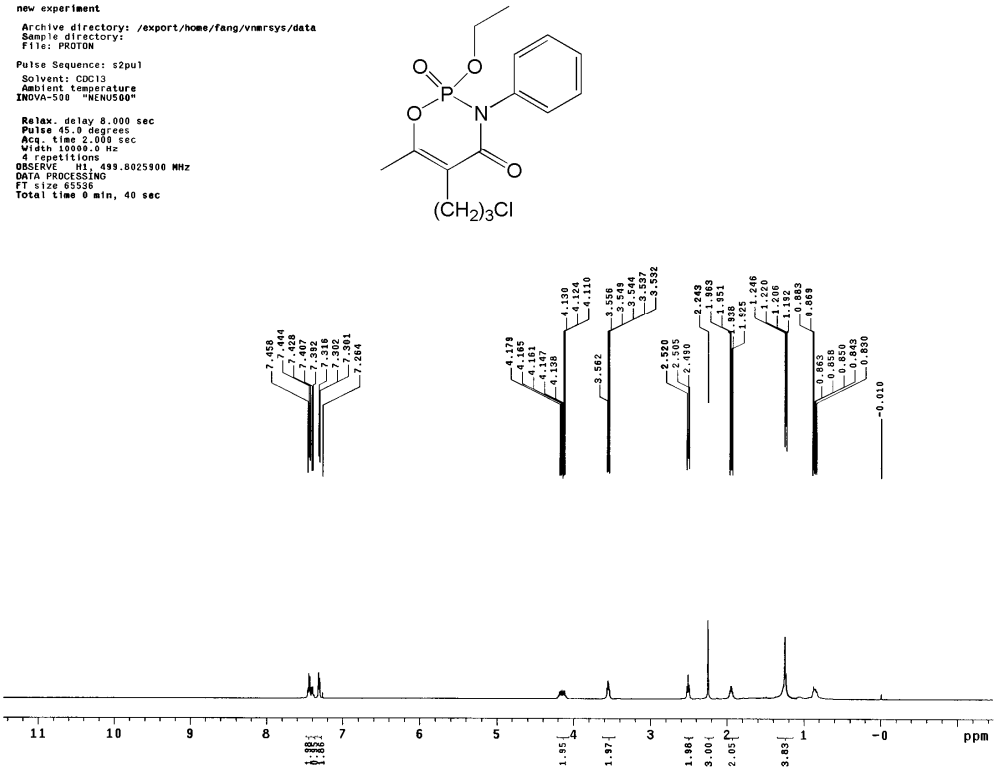

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: i-14-87
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.380 sec
Width 31421.8 Hz
448 repetitions
OBSERVE C13, 125.6754316 MHz
DECOUPLE H1, 499.8059805 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 12 hr, 49 min, 47 sec
NH
O
P
N
(CH2)3Cl
ppm

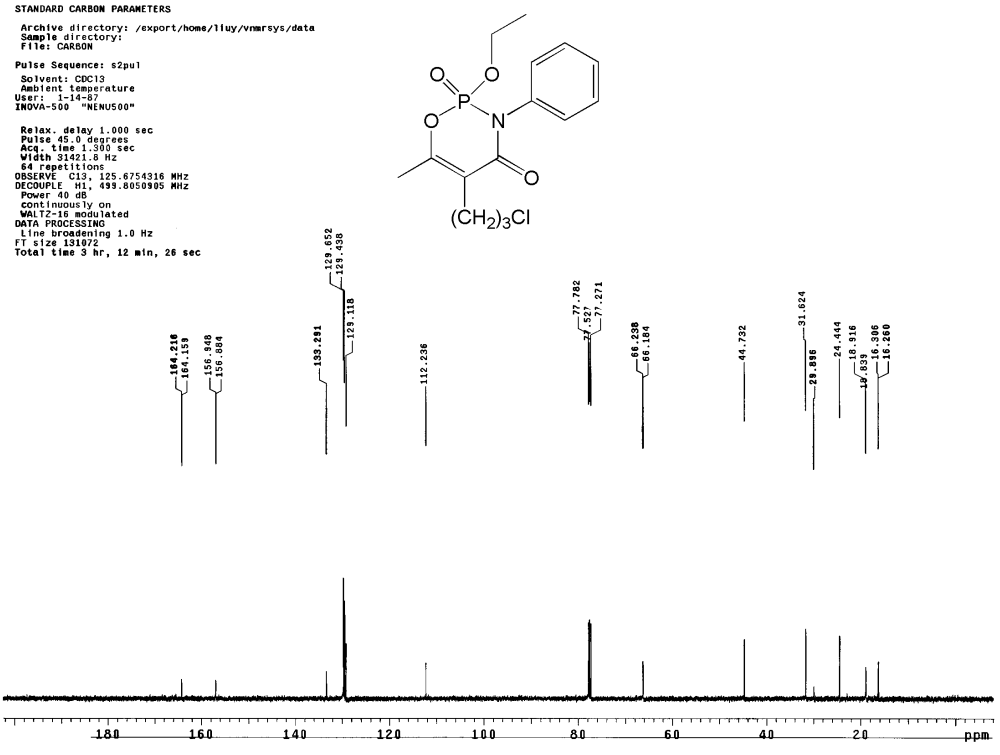
5a-1


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liu/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 20.0 C / 293.1 K
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 11999.4 Hz
8 repetitions
OBSERVE H1, 499.8025800 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
(CH2)3Cl
7.475
7.461
7.445
7.426
7.412
7.323
7.308
7.284
3.802
3.778
3.575
3.570
3.551
3.546
2.533
2.518
2.503
2.266
2.172
1.975
1.968
1.837
-0.001
ppm

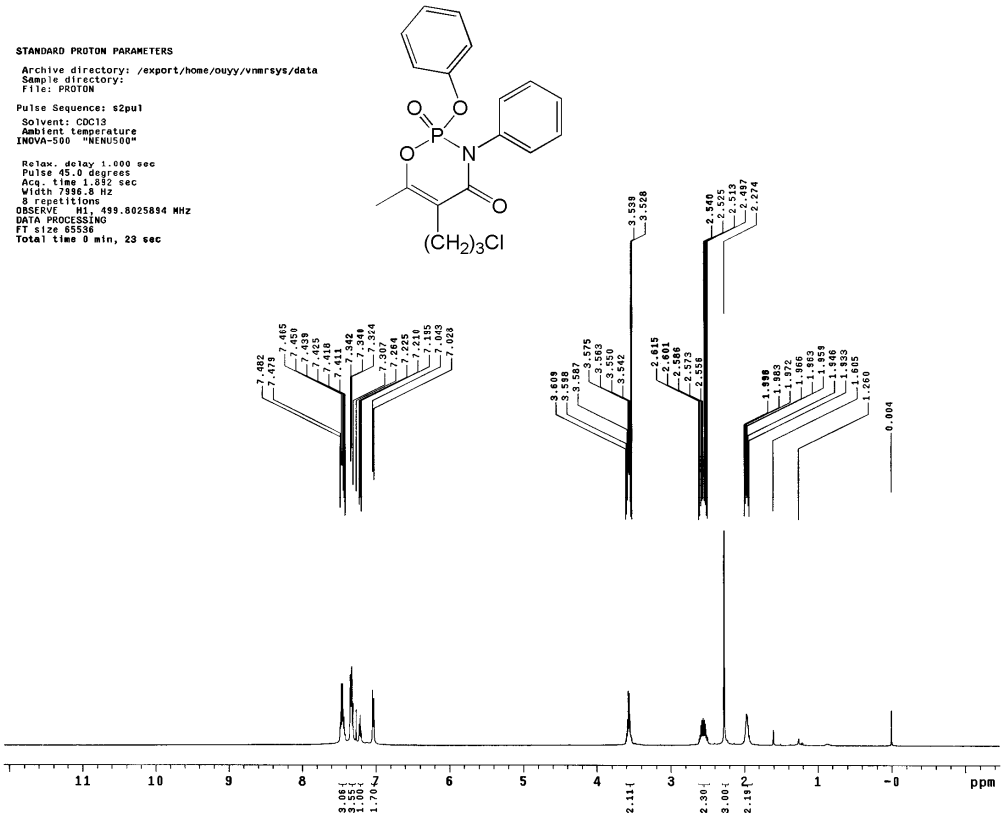

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
64 repetitions
OBSERVE C13, 125.6754316 MHz
DECOUPLE H1, 499.8050805 MHz
Power 40 dB
continuously On
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 12 hr, 49 min, 47 sec
(CH2)3Cl
164.170
164.113
157.025
156.960
133.162
129.755
129.358
129.339
129.213
112.927
77.706
77.450
77.195
56.002
55.949
44.758
31.631
24.474
18.868
18.817
ppm

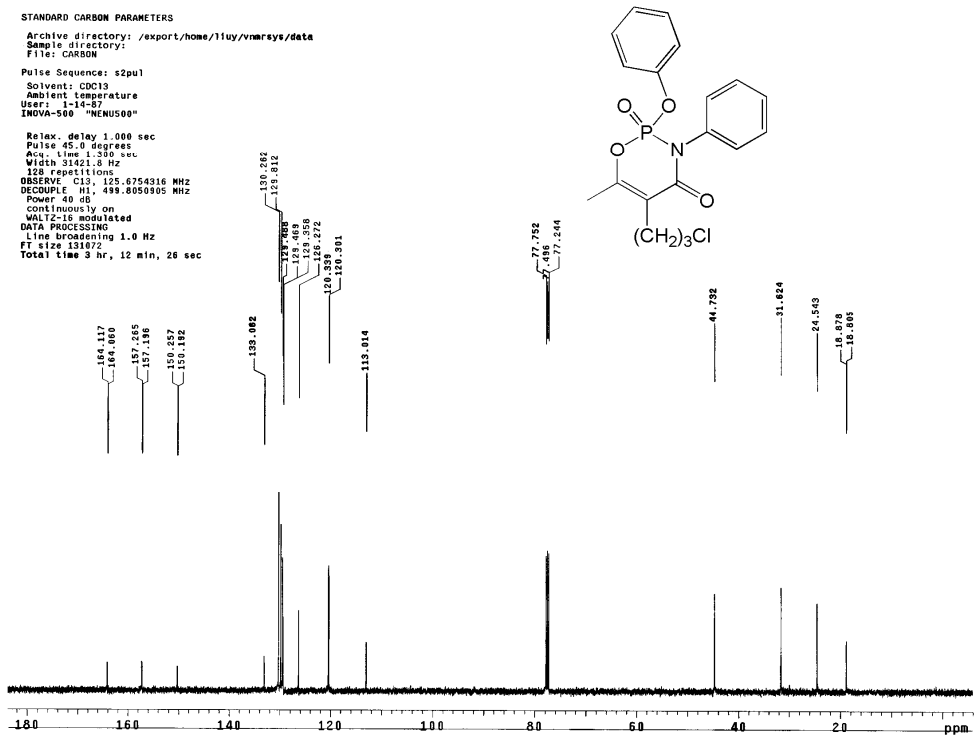
5a-2


new experiment
Archive directory: /export/home/fang/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "NENU500"
Relax. delay 8.000 sec
Pulse 45.0 degrees
Acq. time 2.000 sec
Width 10000.0 Hz
4 repetitions
OBSERVE H1, 499.8025900 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 40 sec
(CH2)3Cl
ppm

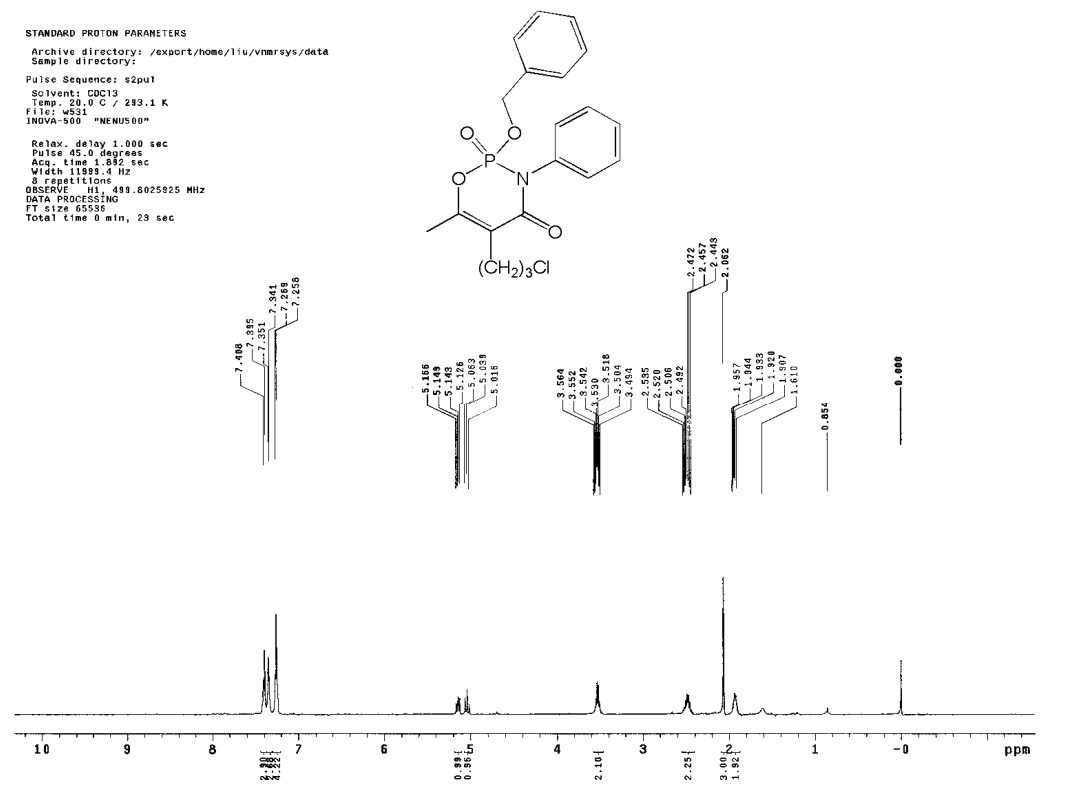

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
64 repetitions
OBSERVE C13, 125.6754316 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
line broadening 1.0 Hz
FT size 131072
Total time 3 hr, 12 min, 26 sec
(CH2)3Cl
ppm

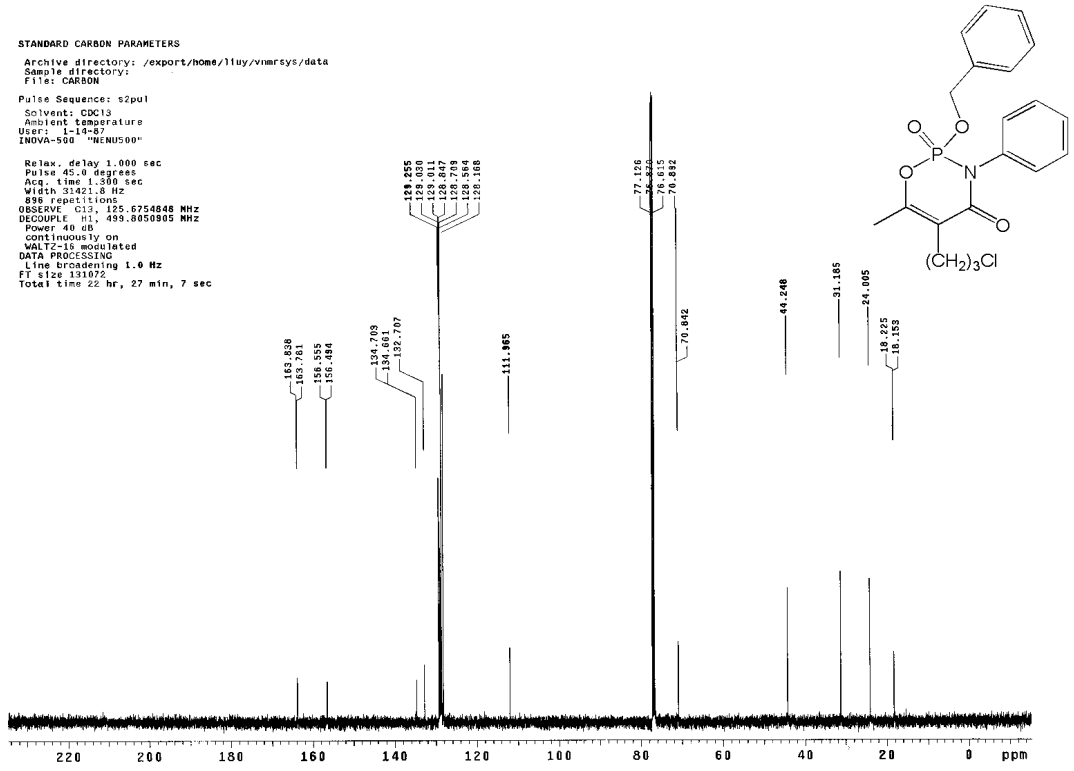
5a-3


STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.882 sec
Width 7996.8 Hz
8 repetitions
OBSERVE H1, 499.8025894 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
(CH2)3Cl
ppm


STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13, 125.6754316 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 3 hr, 12 min, 26 sec
(CH2)3Cl
ppm

5a-4


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liu/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 20.0 C / 293.1 K
File: w531
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.882 sec
Width 11988.4 Hz
8 repetitions
OBSERVE H1, 499.8025825 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
(CH2)3Cl
ppm


STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-07
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
896 repetitions
OBSERVE C13, 125.6754848 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 22 hr, 27 min, 7 sec
(CH2)3Cl
ppm