

Supplementary Material

Studies Towards a Concise, Enantioselective Synthesis of the Roseophilins

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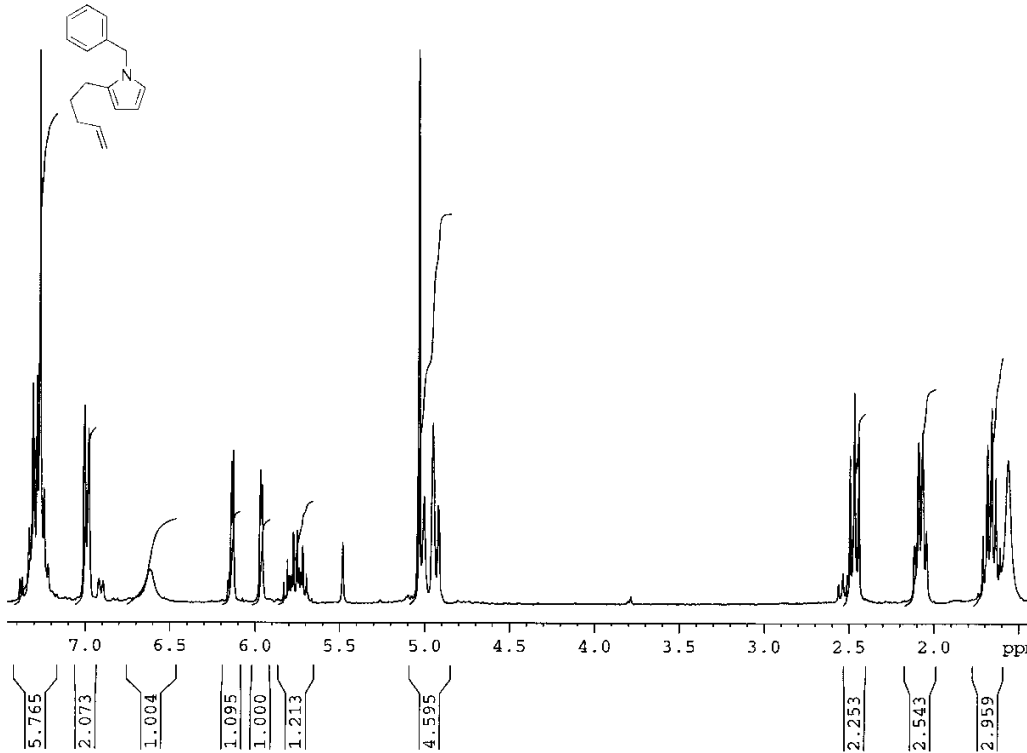
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^1H and ^{13}C NMR Spectra of Synthesised Compounds

1-Benzyl-2-(pent-4-enyl)-1H-pyrrole (15)

¹H NMR



Current Data Parameters
 NAME dksedk
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040823
 Time 11.03
 INSTRUM spect
 PROBHD 5 mm Multinucl
 PULPROG zg
 TD 16384
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 4496.403 Hz
 FIDRES 0.274439 Hz
 AQ 1.8219508 sec
 RG 456.1
 DW 111.200 usec
 DE 6.00 usec
 TE 303.2 K
 D1 3.0000000 sec
 MCREST 0.0000000 sec
 MCWRK 0.01500000 sec

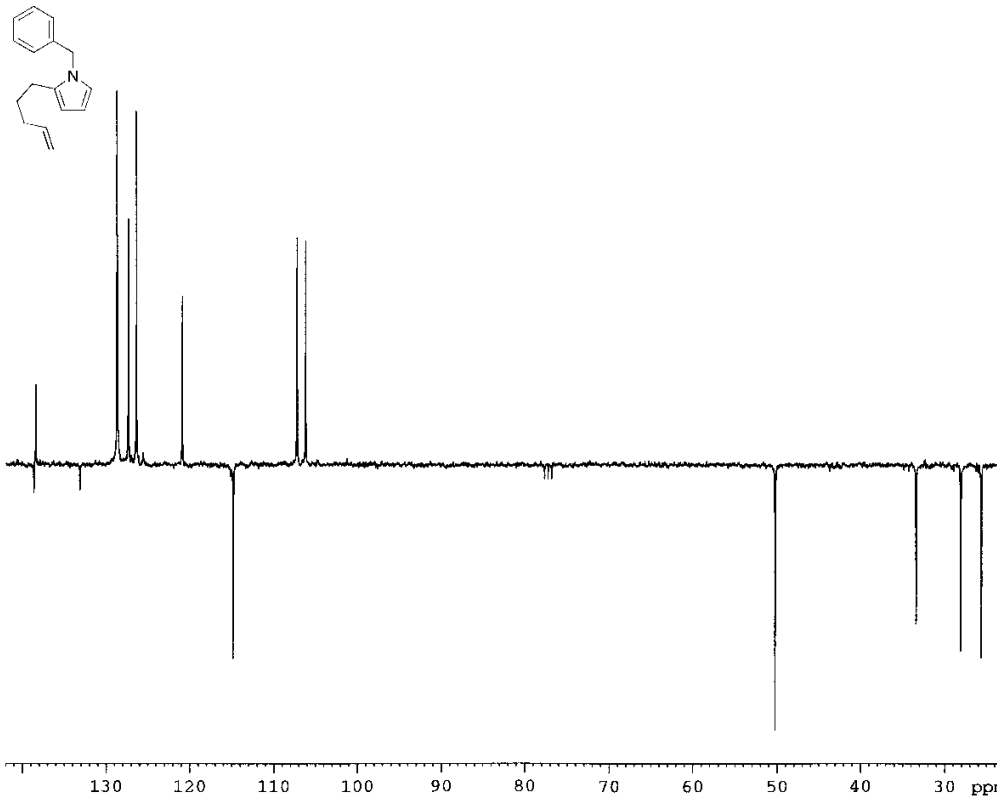
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 P1 3.00 usec
 PL1 -3.00 dB
 SFO1 300.1318320 MHz

F1 - Acquisition parameters
 NDO 2
 TD 512
 SFO1 300.1314 MHz
 FIDRES 7.01535 Hz
 SW 11.968 ppm
 FaMODE undefined

F2 - Processing parameters
 SI 16384
 SF 300.1300051 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 2.00

F1 - Processing parameters
 SI 512
 MC2 TPI
 SF 300.130000 MHz
 WDW QSINE
 SSB 2
 LB 0.30 Hz

¹³C NMR (APT)



Current Data Parameters
 NAME dksedk
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050218
 Time 16.08
 INSTRUM spect
 PROBHD 5 mm Multinucl
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 147
 DS 4
 SWH 20325.203 Hz
 FIDRES 0.620276 Hz
 AQ 0.8061428 sec
 RG 1824.6
 DW 24.500 usec
 DE 20.00 usec
 TE 303.2 K
 CNST2 145.0000000
 CNST11 1.0000000
 D1 2.0000000 sec
 d20 0.00689655 sec
 DELTA 0.00001426 sec
 MCREST 0.0000000 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 11.20 usec
 P2 22.40 usec
 PL1 0.00 dB
 PL2 0.00 dB
 SFO1 75.4767190 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 -3.00 dB
 PL12 19.40 dB
 SFO2 300.1320000 MHz

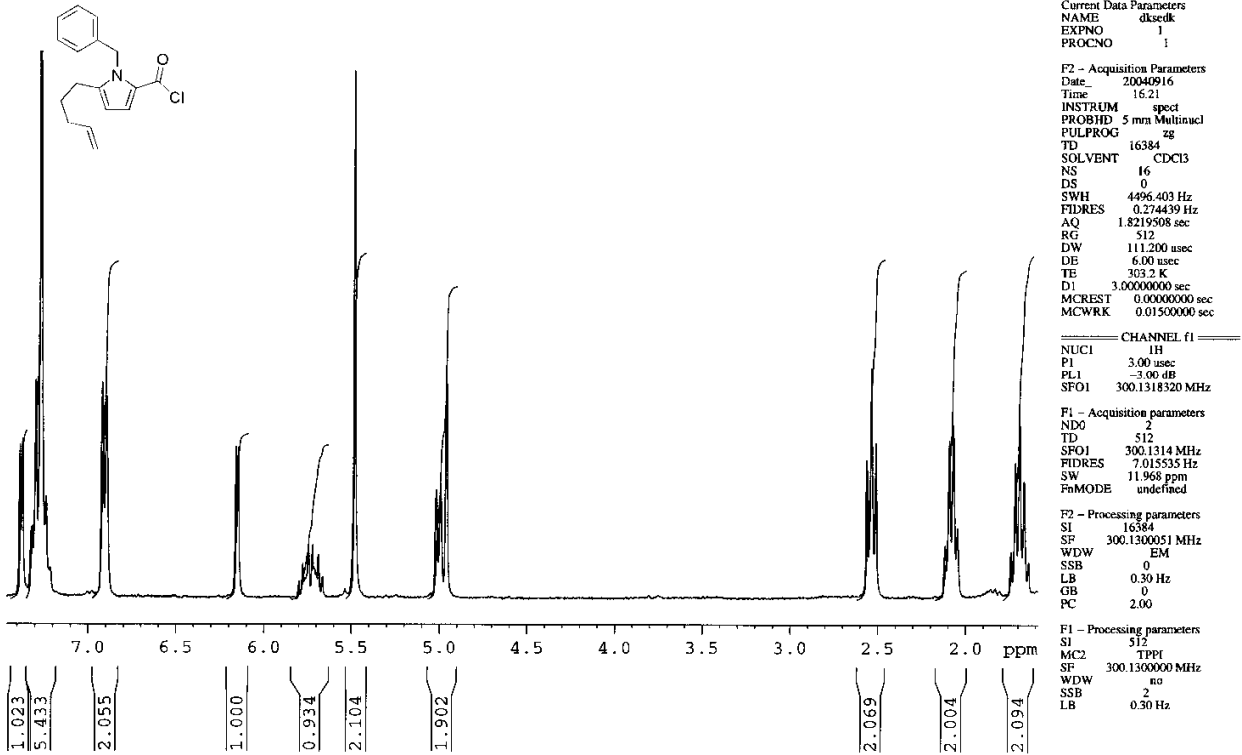
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 TD 512
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 FIDRES 7.01535 Hz
 SW 11.968 ppm
 FaMODE undefined

F2 - Processing parameters
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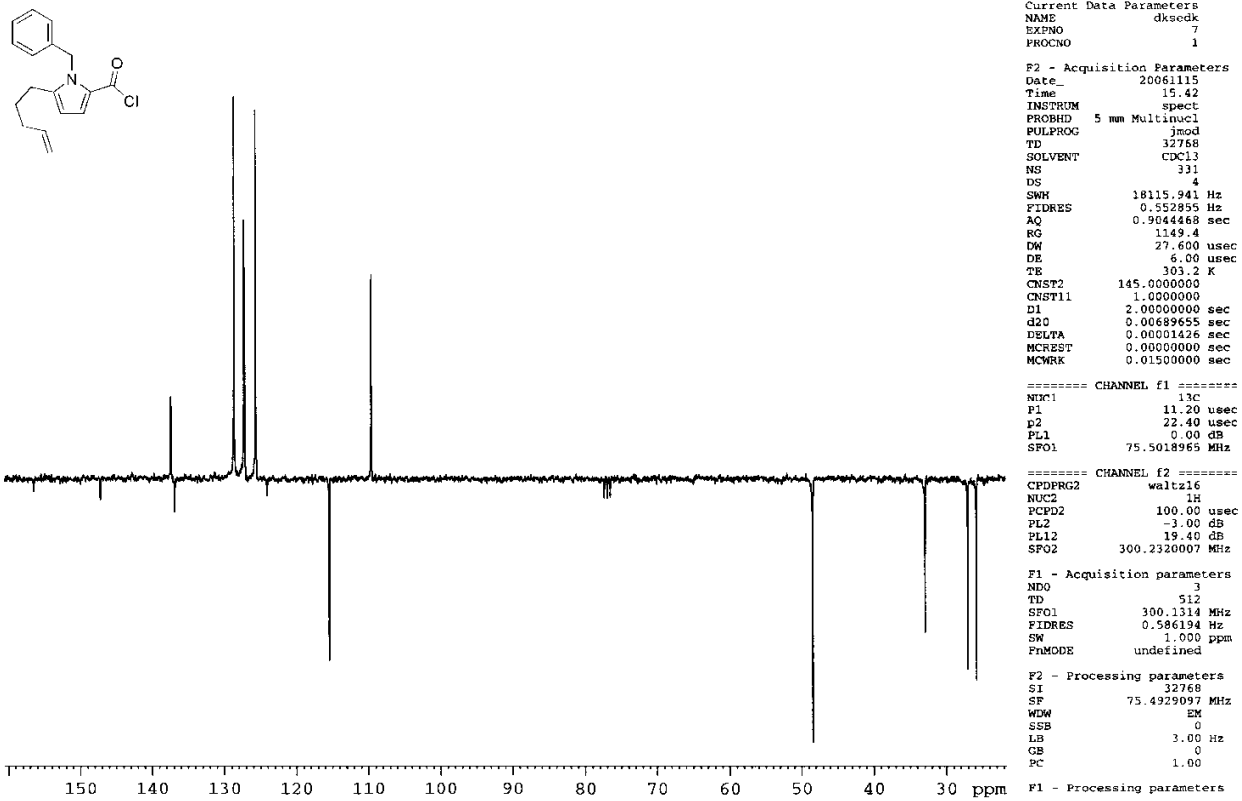
F1 - Processing parameters

1-Benzyl-5-(pent-4-enyl)-1H-pyrrole-2-carbonyl chloride (16)

¹H NMR

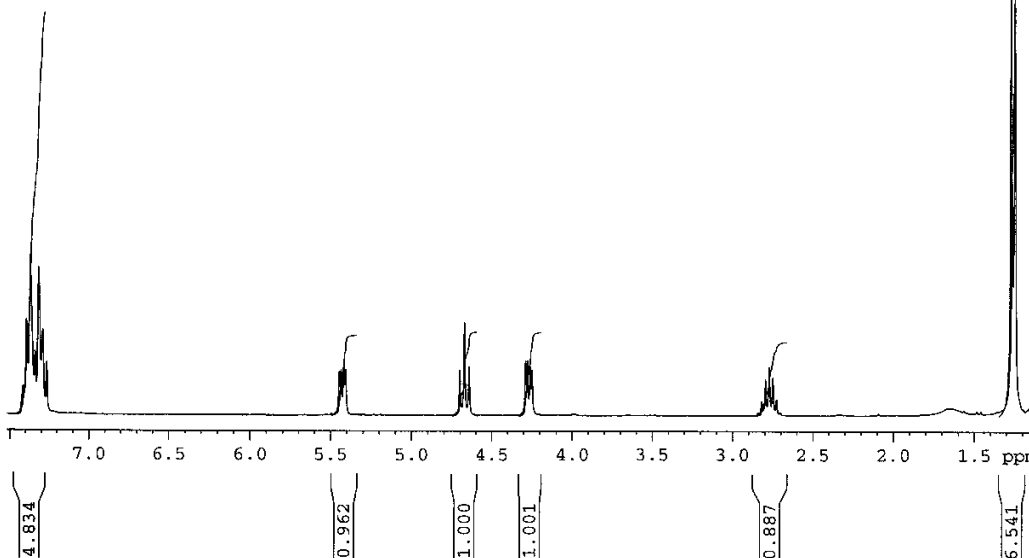
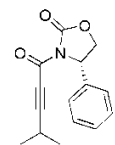


¹³C NMR (APT)



(S)-3-(4-Methylpent-2-ynoyl)-4-phenyloxazolidin-2-one (20)

¹H NMR



Current Data Parameters
NAME dksedk
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070206
Time 22.42
INSTRUM spect
PROBHD 5 mm Multinacl
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 4496.403 Hz
FIDRES 0.274439 Hz
AQ 1.821958 sec
RG 161.3
DW 111.200 usec
DE 6.00 usec
TE 303.2 K
D1 2.0000000 sec
MCREST 0.0000000 sec
MCWRK 0.0150000 sec

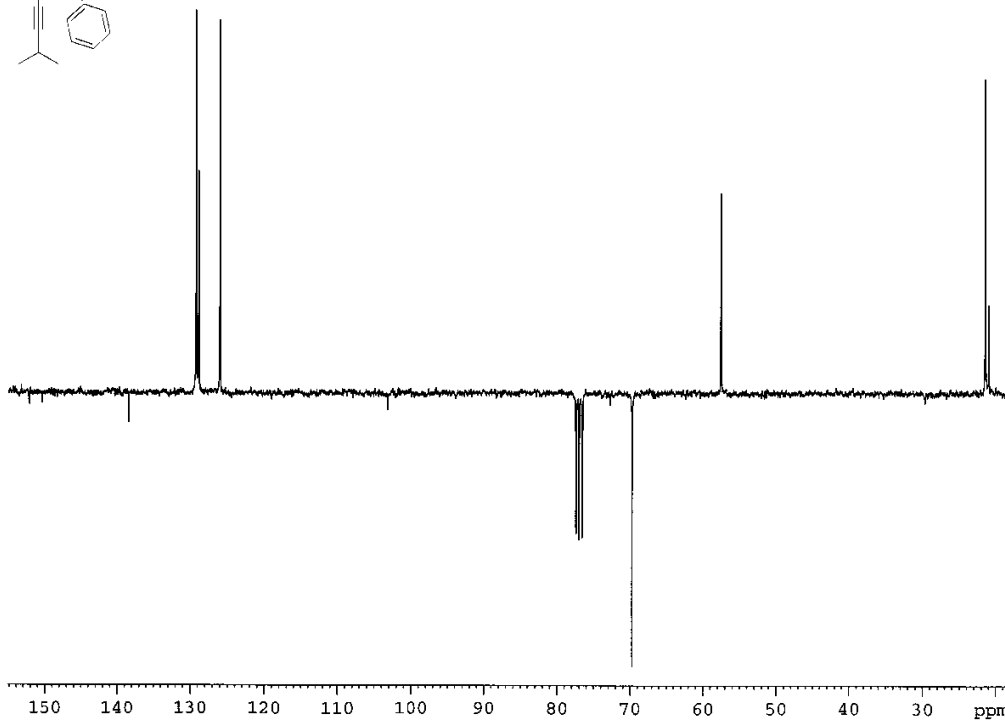
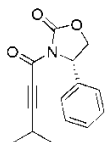
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P1 3.00 usec
PL1 -3.00 dB
SFO1 300.2319515 MHz

F1 - Acquisition parameters
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TD 512
SFO1 300.1314 MHz
FIDRES 0.586194 Hz
SW 1.000 ppm
FhMODE undefined

F2 - Processing parameters
SI 16384
SF 300.2300168 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

F1 - Processing parameters
SI 512
MC2 TPI
SF 300.1300000 MHz
WDW QSINE
SSB 2
LB 0.30 Hz

¹³C NMR (APT)



Current Data Parameters
NAME dksedk
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070206
Time 22.50
INSTRUM spect
PROBHD 5 mm Multinacl
PULPROG jmod
TD 32768
SOLVENT CDCl3
NS 12027
DS 4
SWH 18115.941 Hz
FIDRES 0.552855 Hz
AQ 0.9044468 sec
RG 1149.4
DW 27.600 usec
DE 6.00 usec
TE 303.2 K
CNST2 145.0000000
CNST1 1.0000000
D1 2.0000000 sec
d20 0.0068955 sec
DELTA 0.00001426 sec
MCREST 0.0000000 sec
MCWRK 0.0150000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 11.20 usec
P2 22.40 usec
PL1 0.00 dB
PL2 -3.00 dB
PL3 19.40 dB
SFO1 75.5018965 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -3.00 dB
PL3 19.40 dB
SFO2 300.2320007 MHz

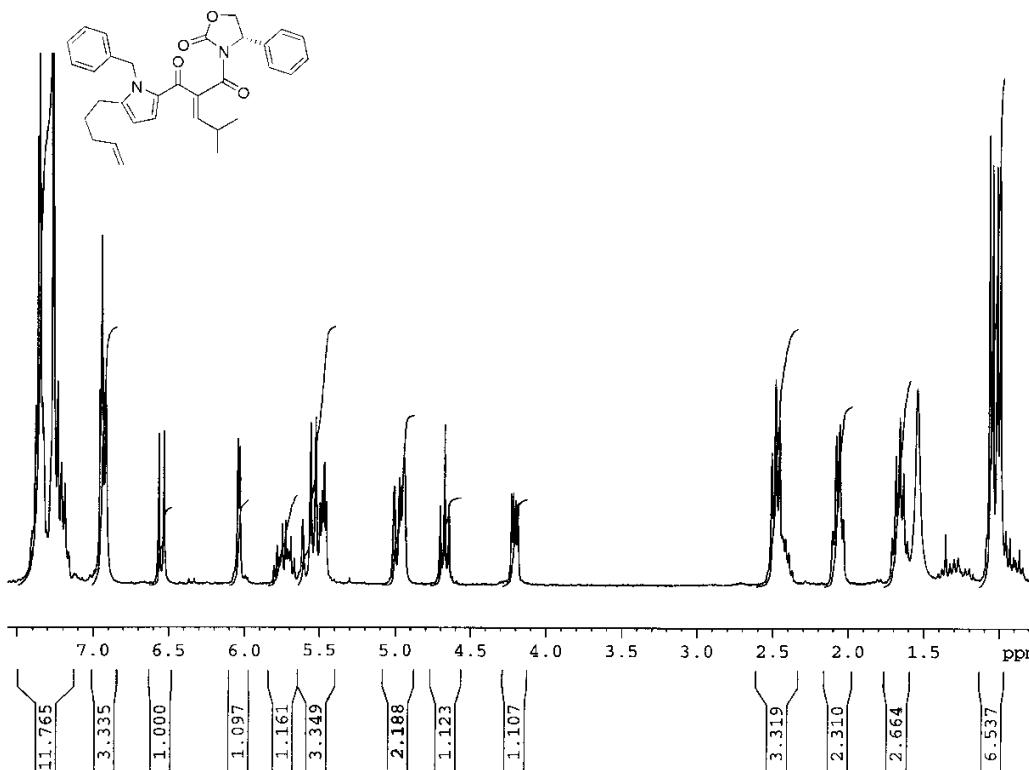
F1 - Acquisition parameters
NDO 3
TD 512
SFO1 300.1314 MHz
FIDRES 0.586194 Hz
SW 1.000 ppm
FhMODE undefined

F2 - Processing parameters
SI 32768
SF 75.4928940 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00

F1 - Processing parameters

(S,Z)-1-(1-Benzyl-5-(pent-4-enyl)-1H-pyrrol-2-yl)-2-(2-methylpropylidene)-3-(2-oxo-4-phenyloxazolidin-3-yl)propane-1,3-dione (21)

¹H NMR



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Current Data Parameters
NAME      dkseck
EXPNO     1
PROCNO    1

F2 - Acquisition Parameters
Date_     20040924
Time      11.49
INSTRUM   spect
PROBHD    5 mm Multinucl
PULPROG   zg
TD         16384
SOLVENT   CDCl3
NS         16
DS         0
SWH        4496.403 Hz
FIDRES     0.274439 Hz
AQ         1.821908 sec
RG         322.5
DW         111.200 usec
DE         6.00 usec
TE         303.2 K
D1         3.0000000 sec
MCREST    0.0000000 sec
MCWRK     0.0150000 sec

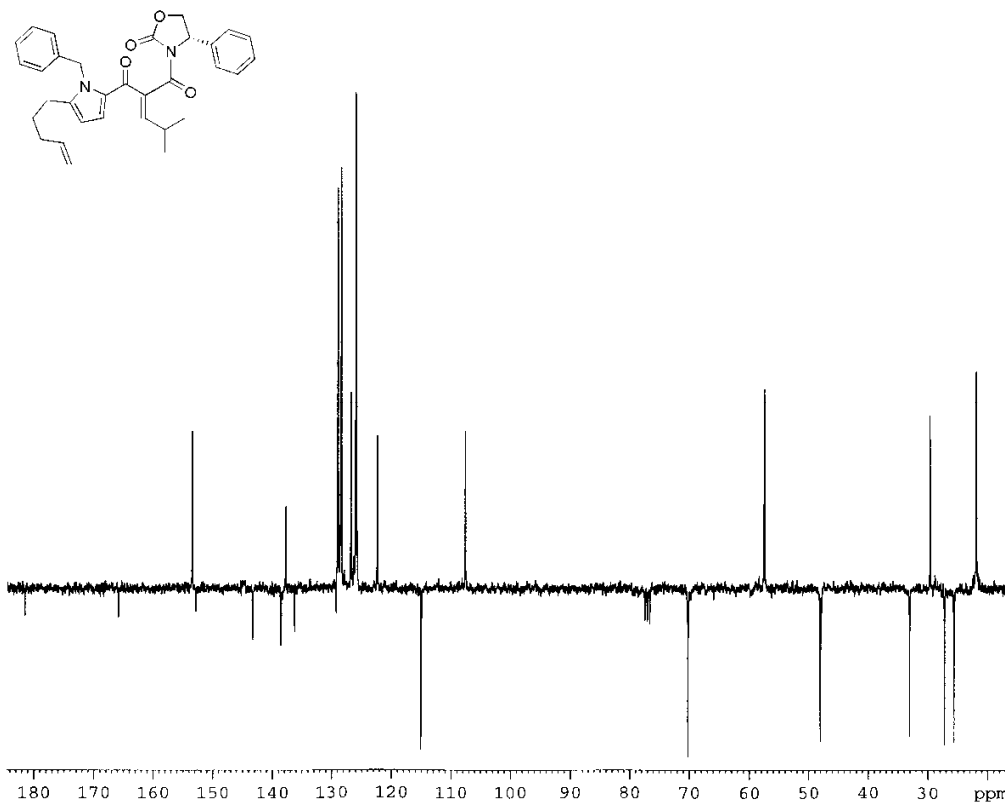
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NUC1      1H
P1         3.00 usec
PL1        -3.00 dB
SFO1      300.1318320 MHz

F1 - Acquisition parameters
ND0        2
TD         512
SFO1      300.1314 MHz
FIDRES     7.01535 Hz
SW         11.98 ppm
FqMODE     undefined

F2 - Processing parameters
SI         16384
SF         300.1300051 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         2.00

F1 - Processing parameters
SI         512
MC2        TPPI
SF         300.1300000 MHz
WDW        no
SSB        2
LB         0.30 Hz
    
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¹³C NMR (APT)



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Current Data Parameters
NAME      dkseck
EXPNO     5
PROCNO    1

F2 - Acquisition Parameters
Date_     20050525
Time      15.40
INSTRUM   spect
PROBHD    5 mm Multinucl
PULPROG   jmod
TD         32768
SOLVENT   CDCl3
NS         247
DS         4
SWH        20325.203 Hz
FIDRES     0.620276 Hz
AQ         0.8061428 sec
RG         1149.4
DW         24.600 usec
DE         20.00 usec
TE         303.2 K
CNST2     145.0000000
CNST11    1.0000000
D1         2.0000000 sec
G20        0.00689655 sec
DELTA      0.00001426 sec
MCREST    0.0000000 sec
MCWRK     0.0150000 sec

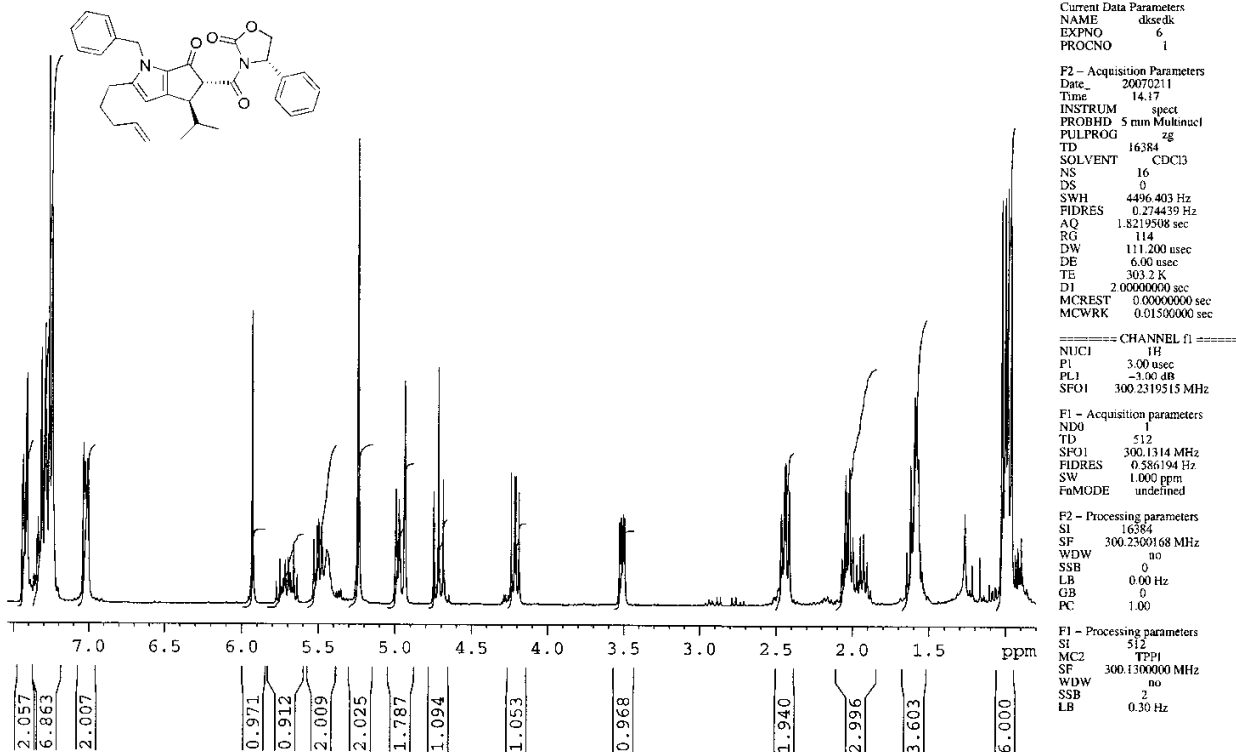
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NUC1      13C
P1         13.00 usec
P2         22.40 usec
PL1         0.00 dB
PL2         0.00 dB
SFO1      75.4767190 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     100.00 usec
PL2        -3.00 dB
PL12      15.40 dB
SFO2      300.1320000 MHz

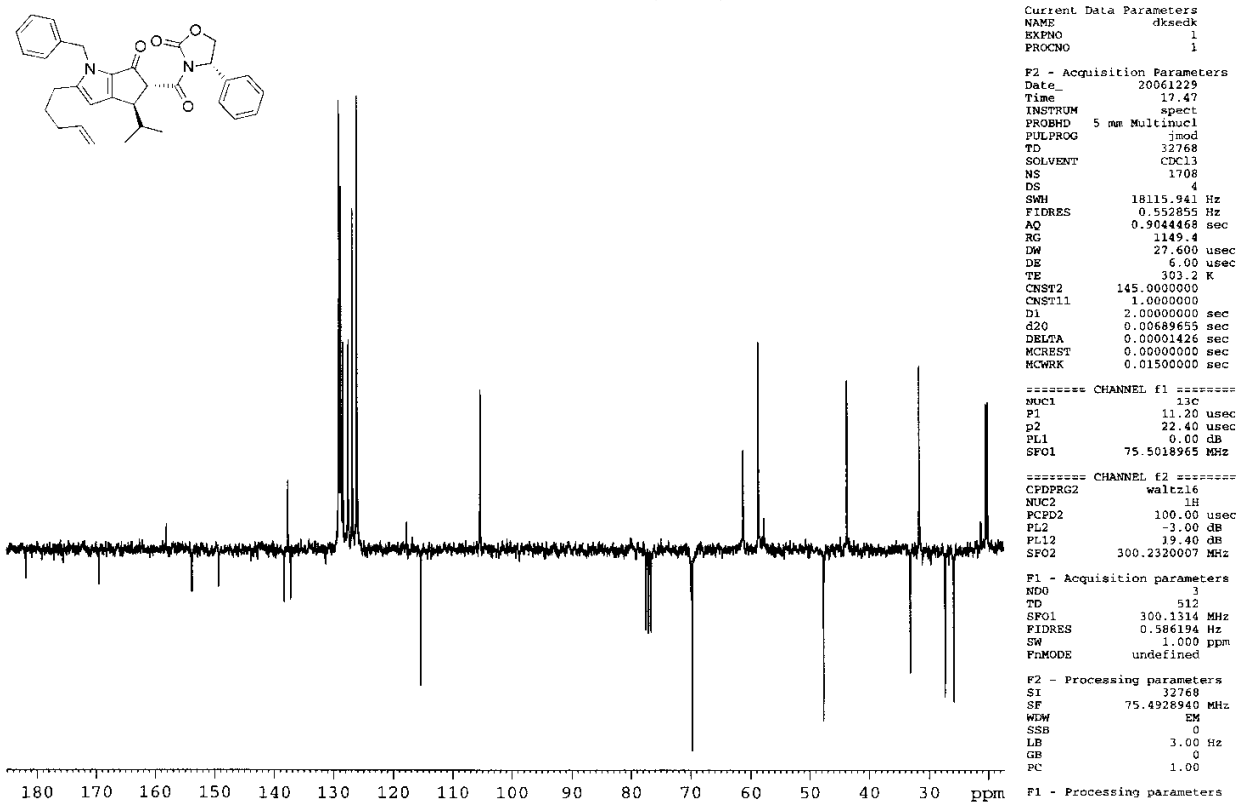
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SF         75.4677556 MHz
WDW        EM
SSB        0
LB         3.00 Hz
GB         0
PC         1.00
    
```

(S)-3-((4R,5R)-1-Benzyl-4-isopropyl-6-oxo-2-(pent-4-enyl)-1,4,5,6-tetrahydrocyclopenta[b]pyrrole-5-carbonyl)-4-phenyloxazolidin-2-one (22)

¹H NMR

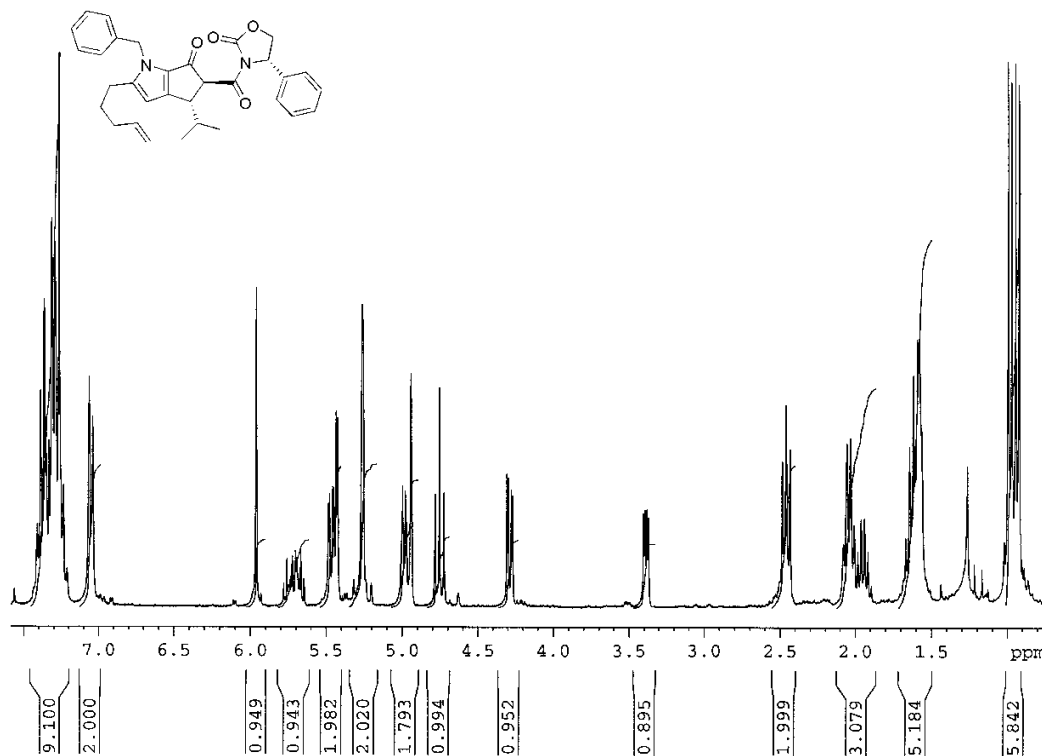


¹³C NMR (APT)



(S)-3-((4S,5S)-1-Benzyl-4-isopropyl-6-oxo-2-(pent-4-enyl)-1,4,5,6-tetrahydrocyclopenta[b]pyrrole-5-carbonyl)-4-phenyloxazolidin-2-one (24)

¹H NMR



Current Data Parameters
 NAME dxsedk
 EXPNO 7
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20070211
 Time 14.32
 INSTRUM spect
 PROBHD 5 mm Multinucl
 PULPROG zg
 TD 16384
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 4496.403 Hz
 FIDRES 0.274439 Hz
 AQ 1.8219508 sec
 RG 203.2
 DW 111.200 usec
 DE 6.00 usec
 TE 303.2 K
 D1 2.0000000 sec
 MCREST 0.0000000 sec
 MCWRK 0.0150000 sec

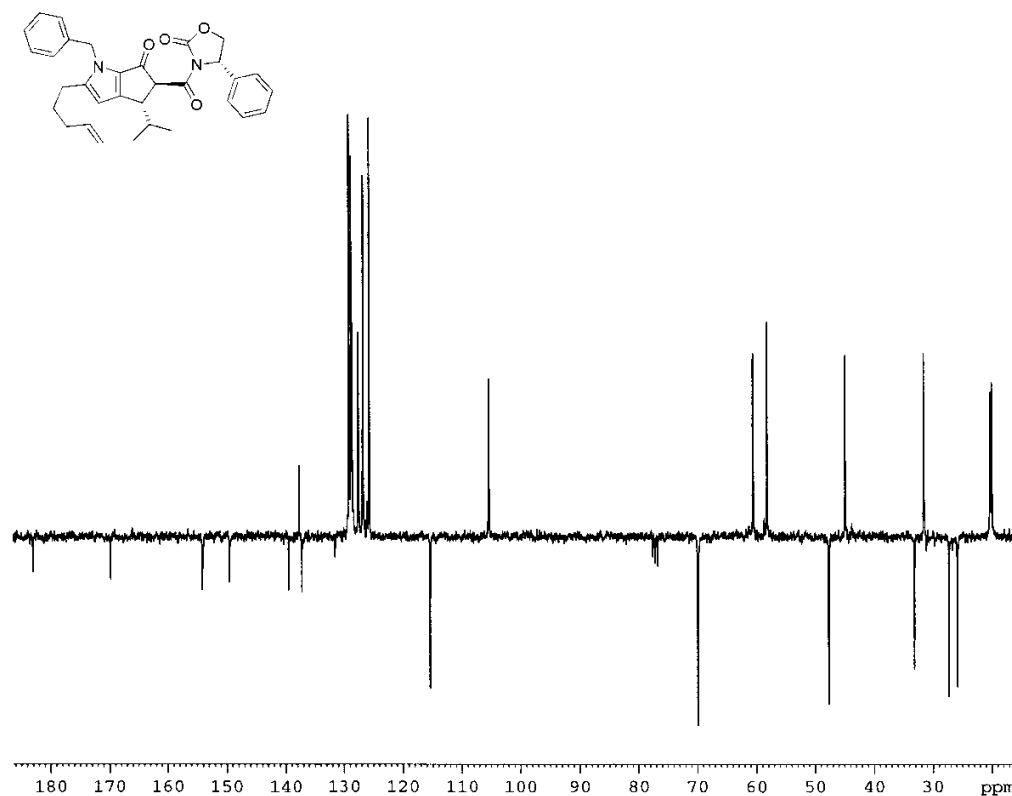
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 NUC1 1H
 P1 3.00 usec
 PL1 -3.00 dB
 SFO1 300.2319515 MHz

F1 - Acquisition parameters
 ND0 1
 TD 512
 SFO1 300.1314 MHz
 FIDRES 0.586194 Hz
 SW 1.000 ppm
 FMODE undefined

F2 - Processing parameters
 SI 16384
 SF 300.2300168 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

F1 - Processing parameters
 SI 512
 MC2 TPPI
 SF 300.1300000 MHz
 WDW no
 SSB 0
 LB 0.30 Hz

¹³C NMR (APT)



Current Data Parameters
 NAME dxsedk
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20061229
 Time 16.50
 INSTRUM spect
 PROBHD 5 mm Multinucl
 PULPROG jmod
 TD 32768
 SOLVENT CDCl3
 NS 596
 DS 4
 SWH 18115.941 Hz
 FIDRES 0.552855 Hz
 AQ 0.9044468 sec
 RG 1149.4
 DW 27.600 usec
 DE 6.00 usec
 TE 303.2 K
 CNST2 145.0000000
 CNST11 1.0000000
 D1 2.0000000 sec
 d20 0.00689655 sec
 DELTA 0.00001425 sec
 MCREST 0.0000000 sec
 MCWRK 0.0150000 sec

==== CHANNEL f1 =====
 NUC1
 P1 11.20 usec
 p2 22.40 usec
 PL1 0.00 dB
 SFO1 75.5018965 MHz

==== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 FCPD2 100.00 usec
 PL2 -3.00 dB
 PL12 19.40 dB
 SFO2 300.2320007 MHz

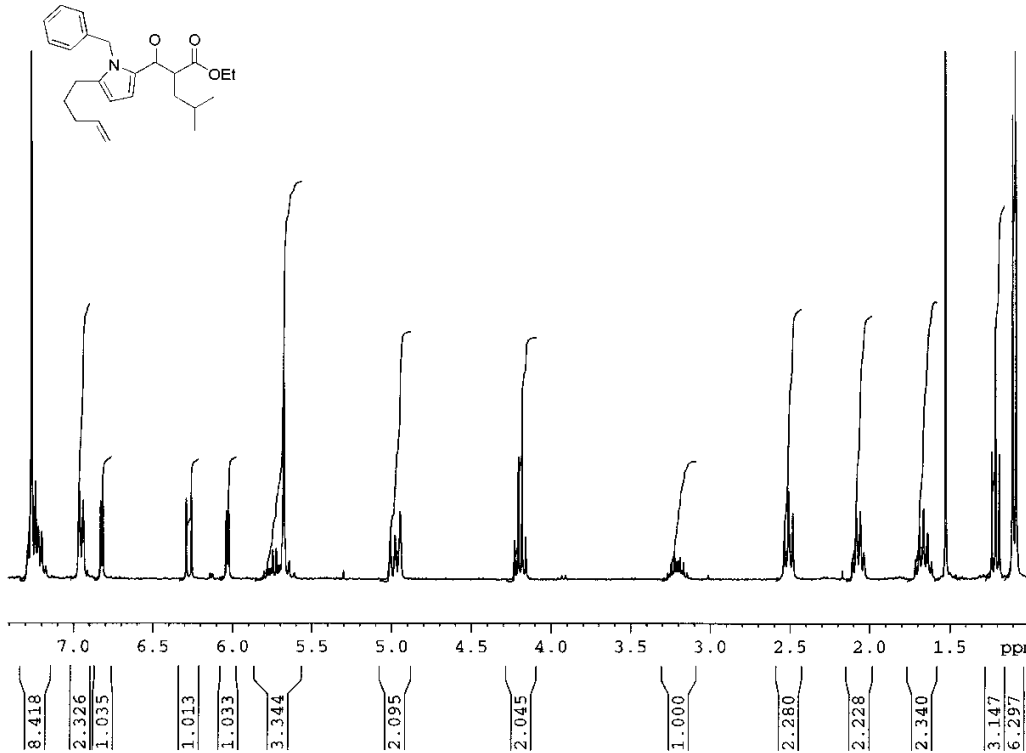
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 TD 512
 SFO1 300.1314 MHz
 FIDRES 0.586194 Hz
 SW 1.000 ppm
 FMODE undefined

F2 - Processing parameters
 SI 32768
 SF 75.4928940 MHz
 WDW EM
 SSB 0
 LB 3.00 Hz
 GB 0
 PC 1.00

F1 - Processing parameters

(Z)-Ethyl 2-(1-benzyl-5-(pent-4-enyl)-1H-pyrrole-2-carbonyl)-4-methylpent-2-enoate (26)

¹H NMR



Current Data Parameters
NAME dxsek
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041118
Time 12.06
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 4496.403 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 812.7
DW 111.200 usec
DE 6.00 usec
TE 303.2 K
D1 3.0000000 sec
MCREST 0.0000000 sec
MCWRK 0.0150000 sec

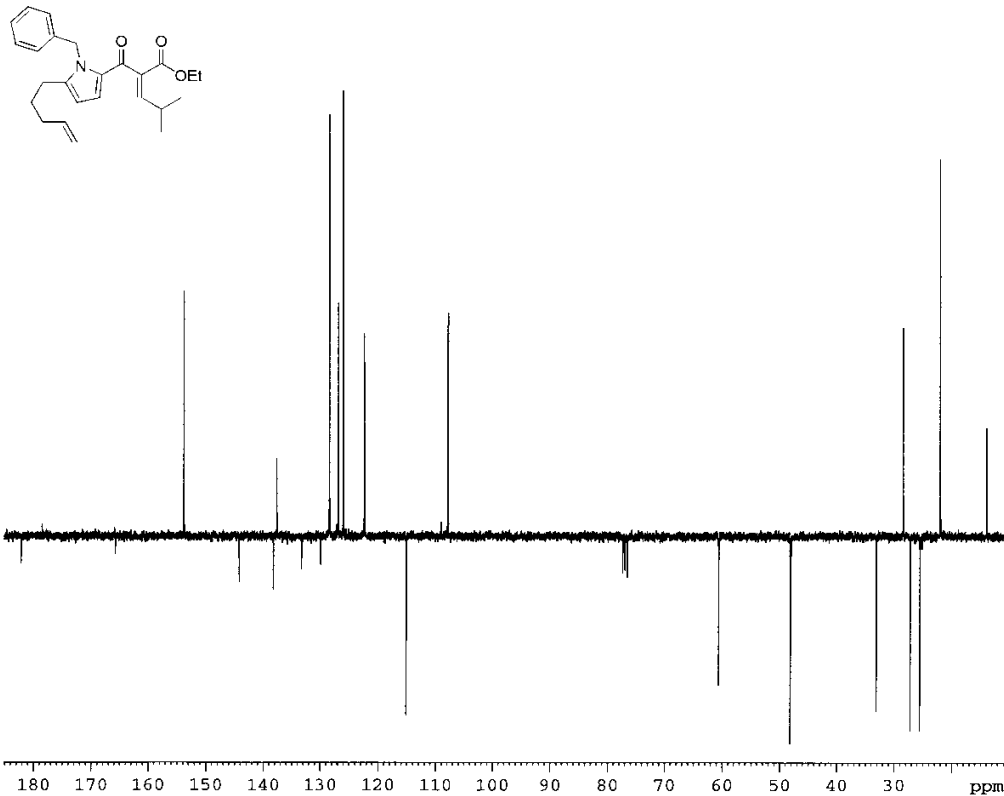
==== CHANNEL f1 =====
NUC1 1H
P1 3.00 usec
PL1 -3.00 dB
SFO1 300.1318320 MHz

F1 - Acquisition parameters
ND0 2
TD 512
SFO1 300.1314 MHz
FIDRES 7.01535 Hz
SW 11.968 ppm
FoMODE undefined

F2 - Processing parameters
SI 16384
SF 300.1300051 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

F1 - Processing parameters
SI 512
MC2 TPPI
SF 300.1300000 MHz
WDW no
SSB 2
LB 0.30 Hz

¹³C NMR (APT)



Current Data Parameters
NAME dxsek
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050118
Time 14.06
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG jmed
TD 32768
SOLVENT CDCl3
NS 469
DS 4
SWH 20325.203 Hz
FIDRES 0.620276 Hz
AQ 0.8061428 sec
RG 1625.5
DW 24.600 usec
DE 20.00 usec
TE 303.2 K
CNST2 145.0000000
CNST11 1.0000000
D1 2.0000000 sec
d20 0.00689655 sec
DELTA 0.00001426 sec
MCREST 0.0000000 sec
MCWRK 0.0150000 sec

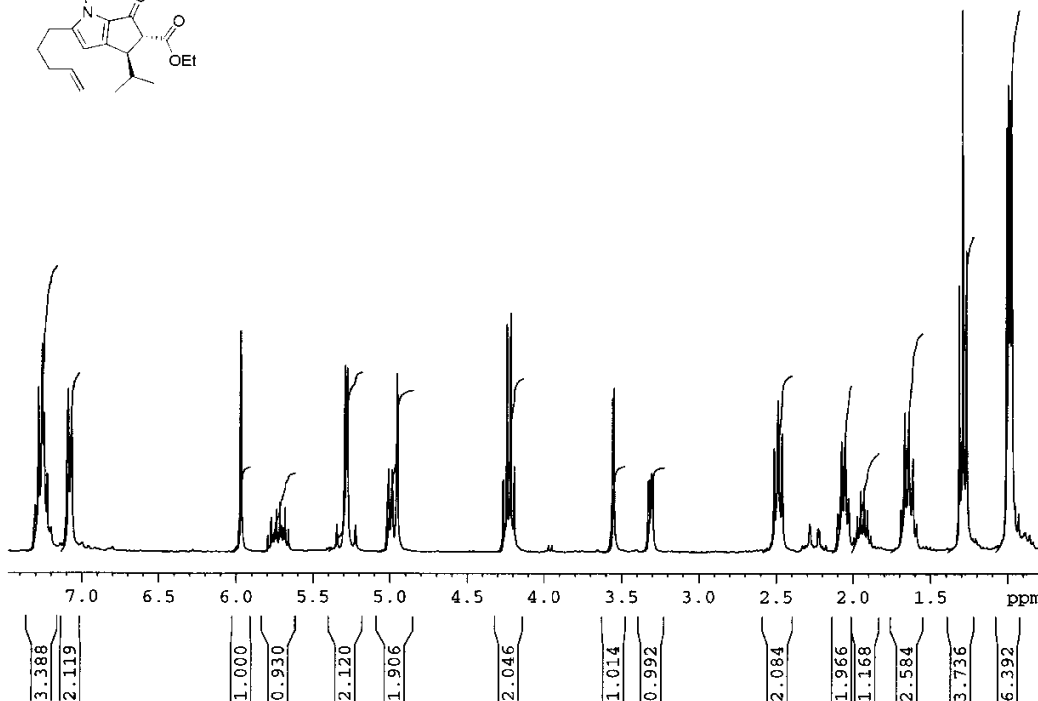
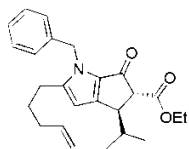
==== CHANNEL f1 =====
NUC1 13C
P1 11.20 usec
p2 22.40 usec
PL1 0.00 dB
SFO1 75.4767190 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -3.00 dB
PL12 19.40 dB
SFO2 300.1320000 MHz

F2 - Processing parameters
SI 32768
SF 75.4677556 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

trans-Ethyl 1-Benzyl-4-isopropyl-6-oxo-2-(pent-4-enyl)-1,4,5,6-tetrahydrocyclopenta[*b*]pyrrole-5-carboxylate (*rac*-27)

¹H NMR



Current Data Parameters
 NAME dksdk
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20041123
 Time 15.10
 INSTRUM spect
 PROBHD 5 mm Multinucl
 PULPROG zg
 TD 16384
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 4495.403 Hz
 FIDRES 0.274439 Hz
 AQ 1.8219508 sec
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 DE 6.00 usec
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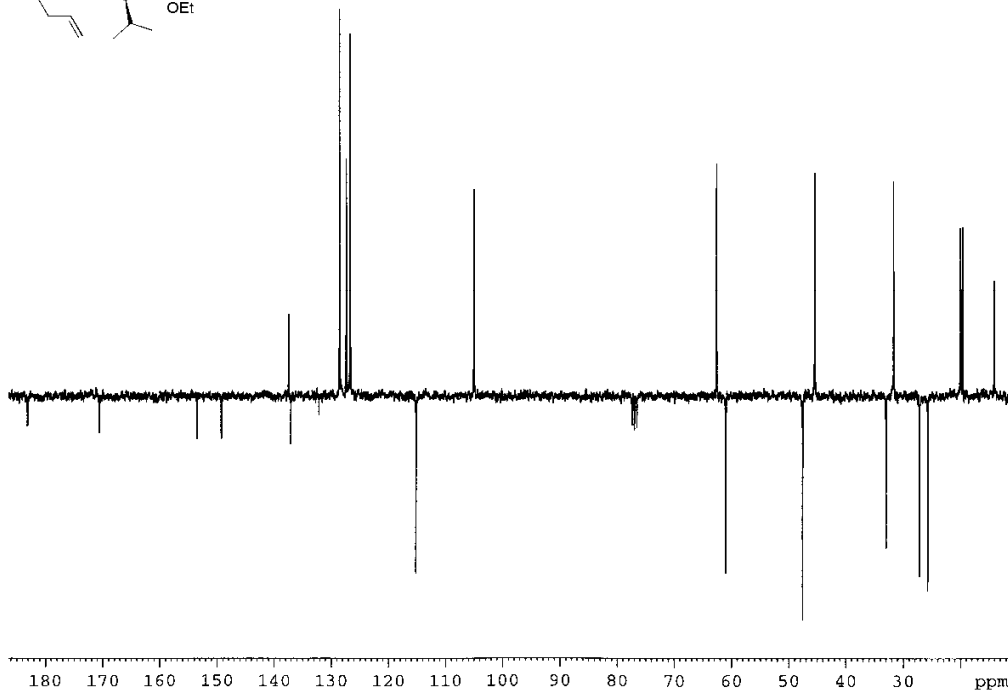
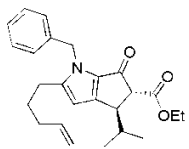
==== CHANNEL f1 =====
 NUC1 1H
 P1 3.00 usec
 PL1 -3.00 dB
 SFO1 300.131820 MHz

F1 - Acquisition parameters
 ND0 2
 TD 512
 SFO1 300.1314 MHz
 FIDRES 7.015535 Hz
 SW 11.968 ppm
 FMODE undefined

F2 - Processing parameters
 SI 16384
 SF 300.1300051 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 2.00

F1 - Processing parameters
 SI 512
 MC2 TPPI
 SF 300.1300000 MHz
 WDW QSINE
 SSB 2
 LB 0.30 Hz

¹³C NMR (APT)



Current Data Parameters
 NAME dksdk
 EXPNO 12
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050719
 Time 12.15
 INSTRUM spect
 PROBHD 5 mm Multinucl
 PULPROG jmod
 TD 32768
 SOLVENT CDCl3
 NS 310
 DS 4
 SWH 20325.203 Hz
 FIDRES 0.620276 Hz
 AQ 0.8061428 sec
 RG 1149.4
 DW 24.600 usec
 DE 20.00 usec
 TE 303.2 K
 CNST2 145.0000000
 CNST11 1.0000000
 D1 2.0000000 sec
 dD0 0.00689655 sec
 DELTA 0.00001428 sec
 MCREST 0.0000000 sec
 MCWRK 0.0150000 sec

==== CHANNEL f1 =====
 NUC1 13C
 P1 11.20 usec
 p2 22.40 usec
 PL1 0.00 dB
 SFO1 75.4757190 MHz

==== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 -3.00 dB
 PL12 19.40 dB
 SFO2 300.1320000 MHz

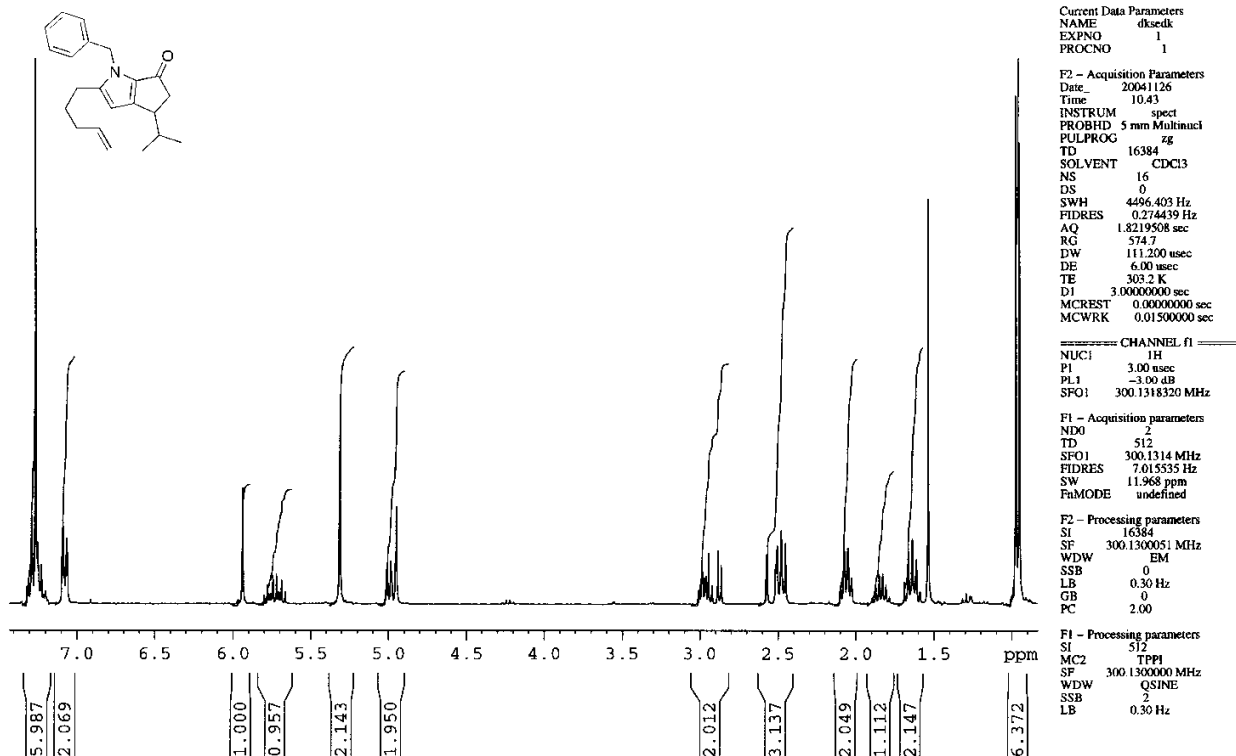
F1 - Acquisition parameters
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 TD 512
 SFO1 300.1314 MHz
 FIDRES 7.015535 Hz
 SW 11.968 ppm
 FMODE undefined

F2 - Processing parameters
 SI 32768
 SF 75.4677556 MHz
 WDW EM
 SSB 0
 LB 3.00 Hz
 GB 0
 PC 1.00

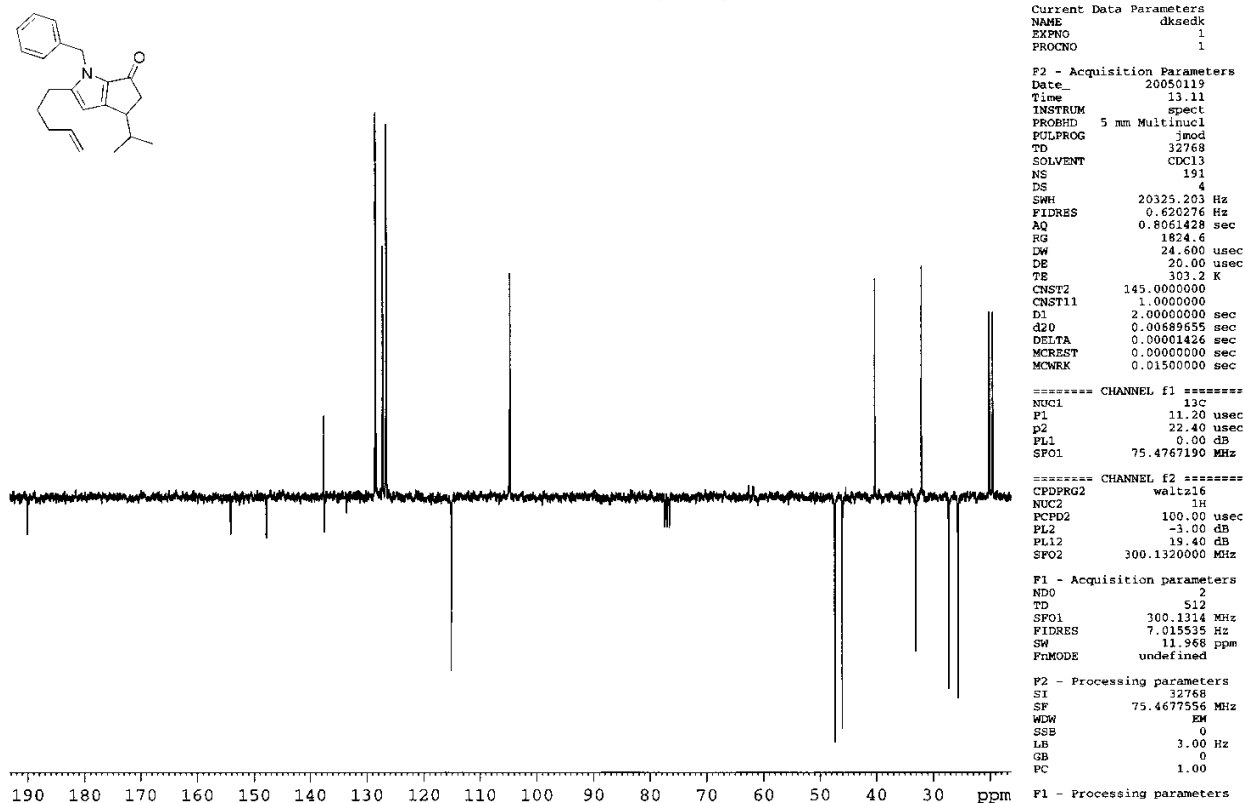
F1 - Processing parameters

1-Benzyl-4-isopropyl-2-(pent-4-enyl)-4,5-dihydrocyclopenta[b]pyrrol-6(1H)-one (rac-28)

¹H NMR

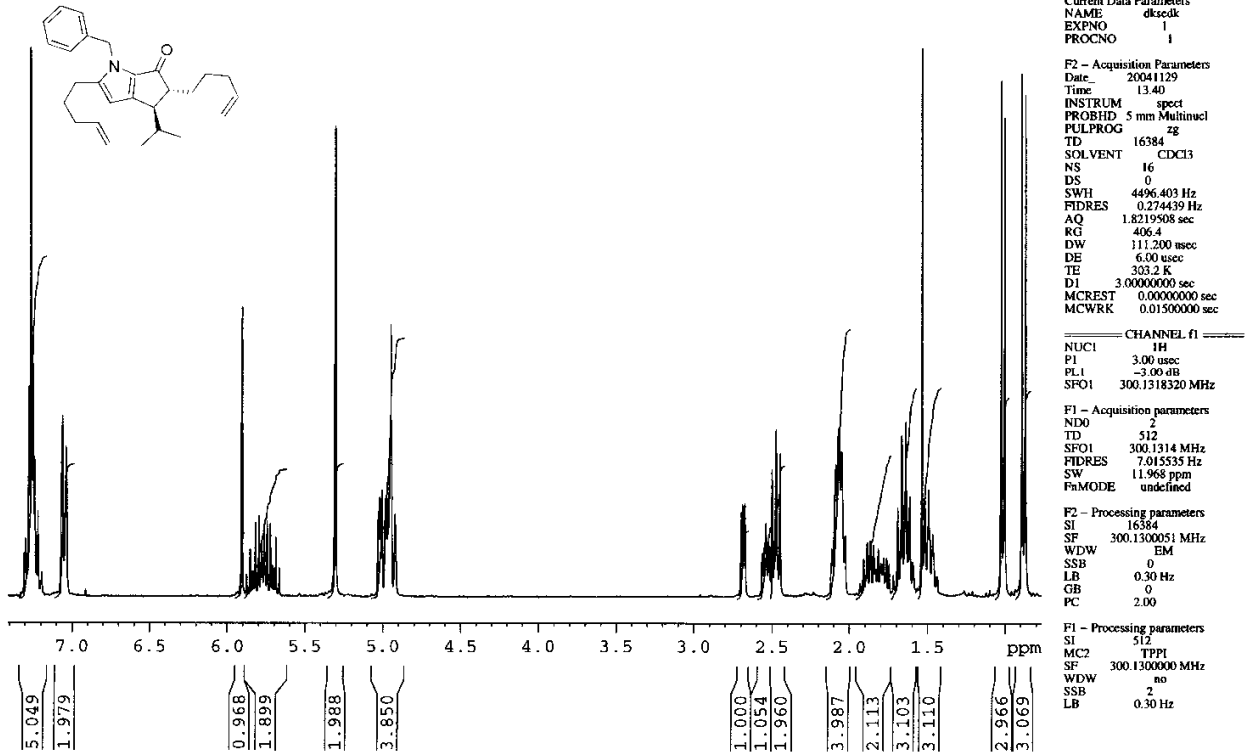


¹³C NMR (APT)

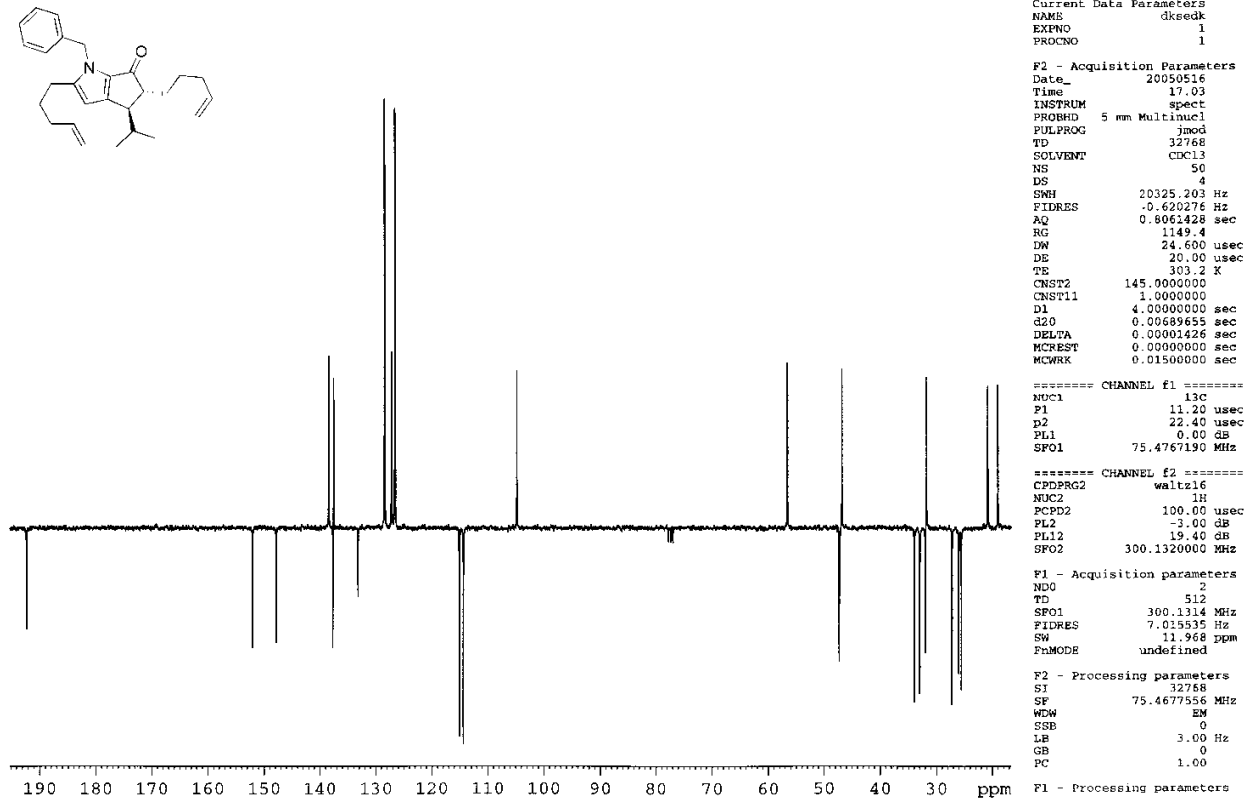


trans-1-Benzyl-4-isopropyl-2,5-di(pent-4-enyl)-4,5-dihydrocyclopenta[*b*]pyrrol-6(1*H*)-one (*rac*-29)

¹H NMR

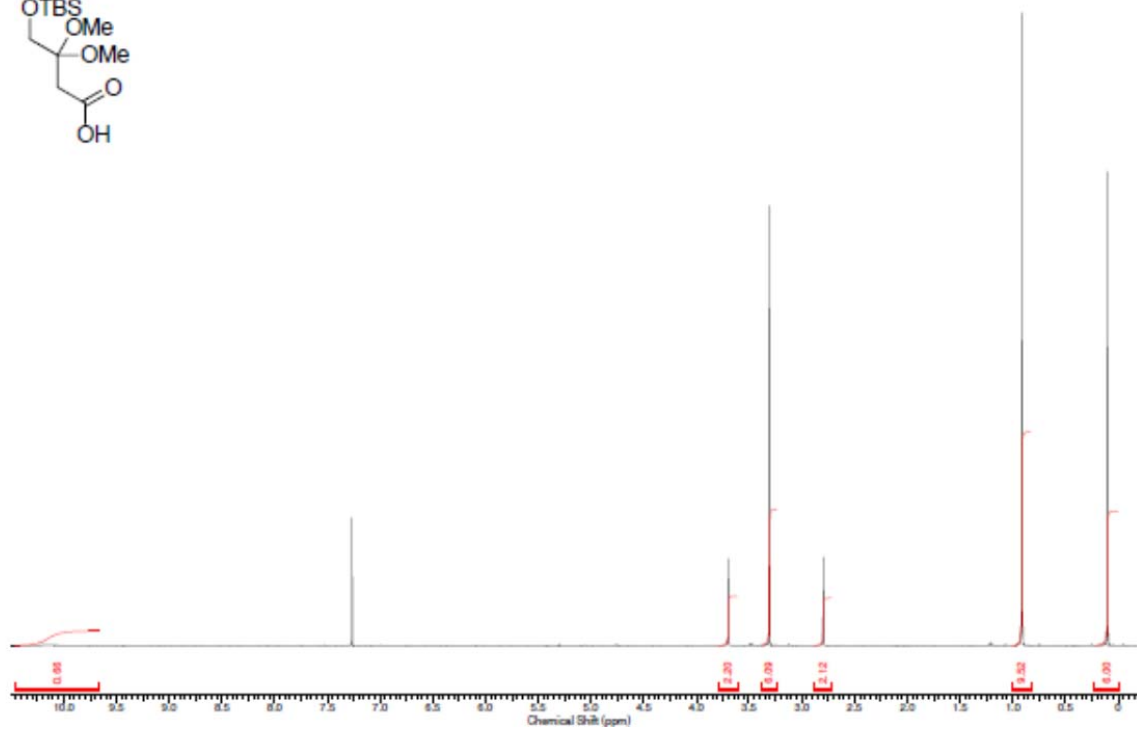
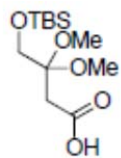


¹³C NMR (APT)



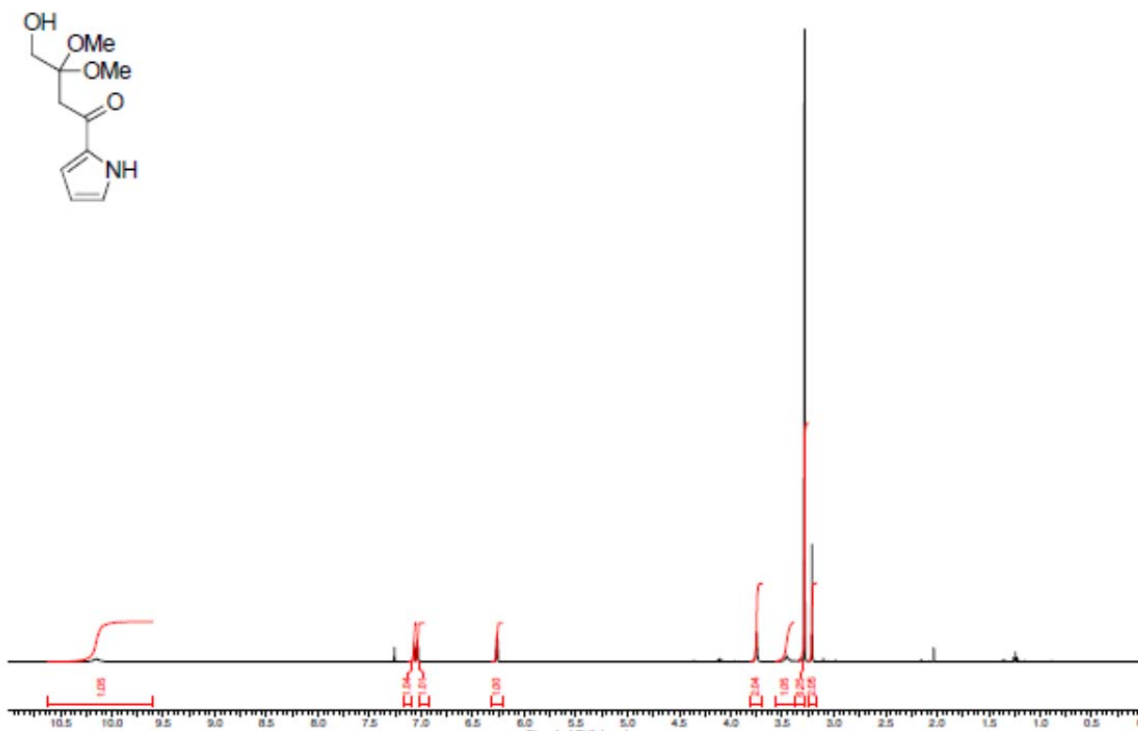
4-[(*tert*-Butyldimethylsilyl)oxy]-3,3-dimethoxy-1-(1*H*-pyrrol-2-yl)butan-1-one (35)

$^1\text{H NMR}$

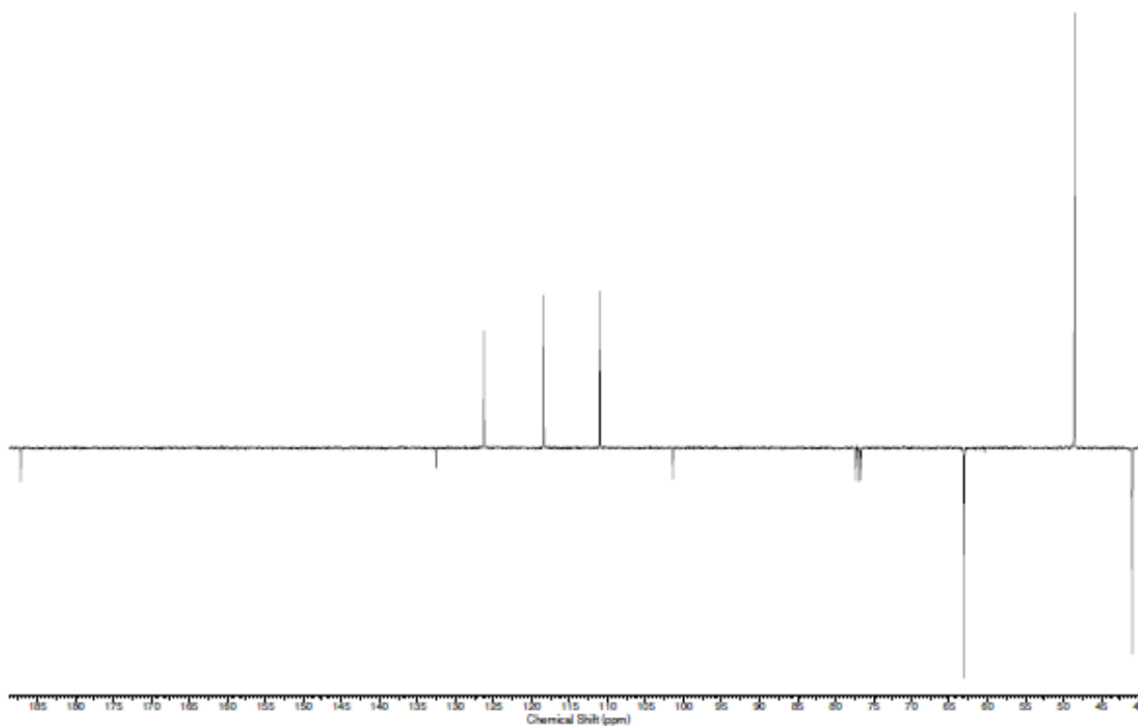


4-Hydroxy-3,3-dimethoxy-1-(1*H*-pyrrol-2-yl)butan-1-one (36)

¹H NMR

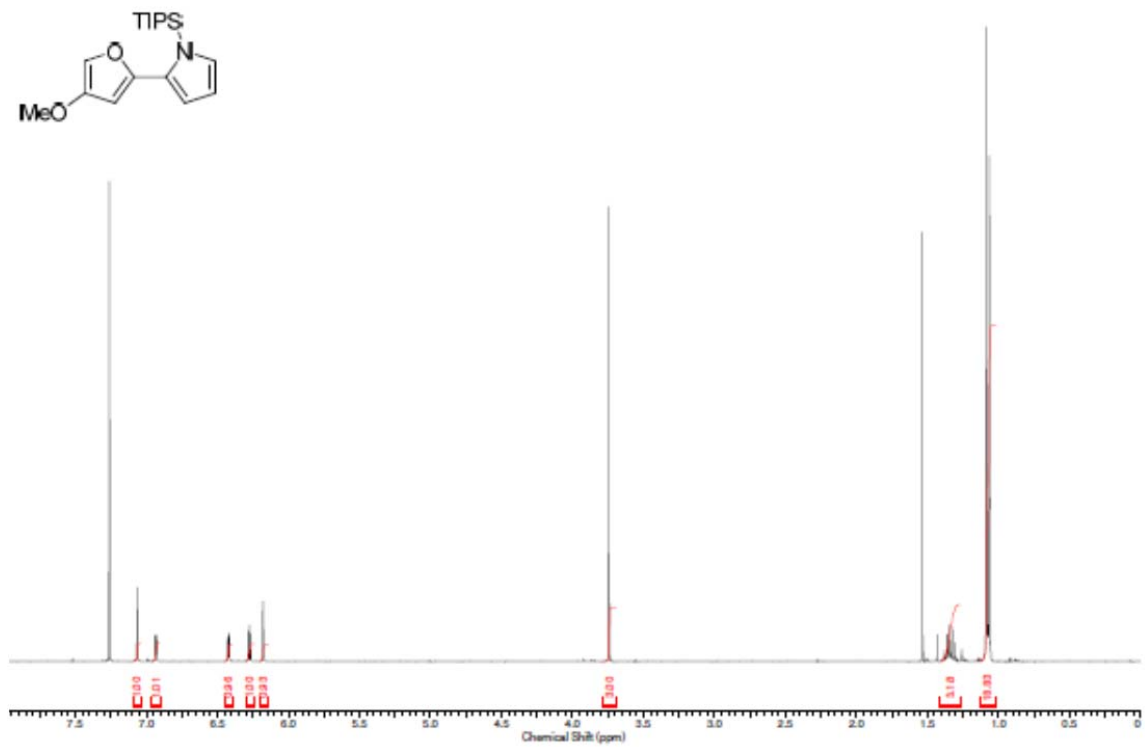


¹³C NMR (APT)



2-(4-Methoxyfuran-2-yl)-1-(triisopropylsilyl)-1H-pyrrole (3b)

^1H NMR



^{13}C NMR (APT)

