Total Synthesis of Buergerinin F *via* Effective Construction of the Asymmetric Quaternary Carbons Using Enantioselective Aldol Reaction

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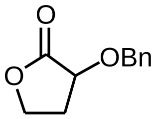
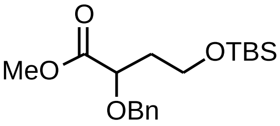
Supporting Information

General Information. All reactions were carried out under argon atmosphere in dried glassware, unless otherwise noted. Dichloromethane was distilled from diphosphorus pentoxide, then calcium hydride, and dried over MS 4Å, benzene and toluene were distilled from diphosphorus pentoxide, and dried over MS 4 Å, and THF and diethyl ether were distilled from sodium/benzophenone immediately prior to use. All reagents were purchased from Tokyo Kasei Kogyo Co., Ltd., Kanto Chemical Co., Inc. or Aldrich Chemical Co., Inc., and used without further purification unless otherwise noted.

Column chromatography was performed on Silica gel 60 (Merck) or Wakogel B5F. Thin layer chromatography was performed on Wakogel B5F. ^{1}H and ^{13}C NMR spectra were recorded with tetramethylsilane (TMS), chloroform (in chloroform-*d*) or benzene (in benzene-d_6) as internal standard.



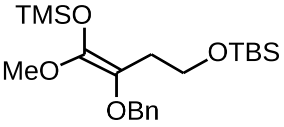
3-Benzyloxy-3,4,5-trihydrofuran-2-one: To a suspension of sodium hydride (60%, 4.70 g, 118 mmol) in THF (60 mL) at 0 °C was added a solution of 3-hydroxy-3,4,5-trihydrofuran-2-one (10.0 g, 98.0 mmol) in THF (40 mL). After the reaction mixture had been stirred for 15 min at rt, benzyl bromide (15.2 mL, 128 mmol) and DMF (10 mL) was added at 0 °C. The reaction mixture was stirred for 46 h at room temperature and then saturated aqueous ammonium chloride was added. The mixture was extracted with diethyl ether, and the organic layer was washed with water and brine, dried over sodium sulfate. After filtration of the mixture and evaporation of the solvent, the crude product was purified by column chromatography (AcOEt / hexane = 1 / 10) to afford 3-benzyloxy-3,4,5-trihydrofuran-2-one (15.6 g, 83%) as a pale yellow oil.



7

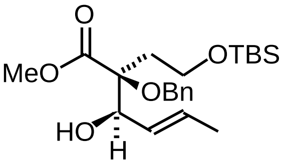
Methyl 2-benzyloxy-4-(*t*-butyldimethylsiloxy)butanoate (7): To a solution of 3-benzyloxy-3,4,5-trihydrofuran-2-one (3.00 g, 15.6 mmol) in methanol (23 mL) at room temperature was added sodium methoxide in methanol (1.50 M, 22.0 mL, 33.0 mmol). The reaction mixture was stirred for 20 min at room temperature and then solvent was removed under the reduced pressure. The reaction mixture was neutralized with 1 M hydrogen chloride in diethyl ether at 0 °C. The residue was dissolved with water and the mixture was extracted with cooled diethyl ether, and the organic layer was washed with water and brine, dried over sodium sulfate. After filtration of the mixture and evaporation of the solvent at 0 °C, the crude product was filtered again through a short pad of silica gel 60N (neutral, Kanto Chemical Co., Inc.) with cooled diethyl ether. Concentration of the filtrate by evaporation of the solvent at 0 °C afforded crude methyl 2-benzyloxy-4-hydroxybutanoate as a colorless oil. Above prepared methyl 2-benzyloxy-4-hydroxybutanoate was instantly used in the following reaction without further purification.

To a solution of *t*-butylchlorodimethylsilane (3.75 g, 25.0 mmol) and imidazole (3.40 g, 49.9 mmol) in DMF (20 mL) at 0 °C was added the above prepared methyl 2-benzyloxy-4-hydroxybutanoate in DMF (11.2 mL). After the reaction mixture had been stirred for 10 min at 0 °C, it was allowed to warm to room temperature. The reaction mixture was stirred for 2 h at room temperature and then phosphate buffer (pH = 7) was added at 0 °C. The mixture was extracted with diethyl ether, and the organic layer was washed with water and brine, dried over sodium sulfate. After filtration of the mixture and evaporation of the solvent, the crude product was purified by column chromatography (AcOEt / hexane = 1 / 20) to afford ester **7** (4.20 g, 80%) as a colorless oil.



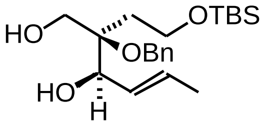
6

(*E*)-2-Benzyloxy-4-(*t*-butyldimethylsiloxy)-1-methoxy-1-(trimethylsiloxy)butene (6): To a solution of diisopropylamine (1.31 g, 12.9 mmol) in THF (8.3 mL) at 0 °C was added *n*-butyllithium in hexane (1.66 M, 7.46 mL, 12.4 mmol). After the reaction mixture had been stirred for 10 min at 0 °C, a solution of ester **7** (4.00 g, 11.8 mmol) in THF (4 mL) was added at -78 °C. The reaction mixture was stirred for 30 min at -78 °C and then a solution of chlorotrimethylsilane (1.67 g, 15.4 mmol) in THF (2 mL) was added. After the reaction mixture had been stirred for 10 min at -78 °C, it was warmed to room temperature. The reaction mixture was stirred for 30 min at room temperature, and then it was concentrated by evaporation of the solvent. Petroleum ether was added to the residue, and the suspension was filtered through a short pad of Celite under argon atmosphere. The filtrate was concentrated by evaporation of the solvent to afford ketene silyl acetal **6** (*E*/*Z* = 92/8, 4.71 g, 97%) as a pale yellow oil. Above prepared ketene silyl acetal **6** was used in the following reaction without further purification.



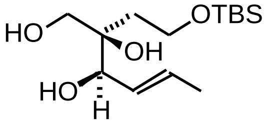
5

Methyl (2*S*,3*R*,4*E*)-2-benzyloxy-2-(2-[*t*-butyldimethylsiloxy]ethyl)-3-hydroxyhex-4-enoate (5): To tin(II) trifluoromethanesulfonate (3.55 g, 8.52 mmol) at room temperature were successively added a solution of (*S*)-1-methyl-2-(1-naphthylaminomethyl)pyrrolidine (2.32 g, 9.65 mmol) in propionitrile (20 mL) and a solution of dibutyltin diacetate (3.19 g, 9.09 mmol) in propionitrile (20 mL). After the reaction mixture had been stirred for 5 min at room temperature, a solution of ketene silyl acetal **6** (3.50 g, 8.52 mmol) in propionitrile (10 mL) and a solution of crotonaldehyde (398 mg, 5.68 mmol) in propionitrile (10 mL) were added at -78 °C. The reaction mixture was stirred for 41 h at -78 °C and then saturated aqueous sodium hydrogencarbonate was added. The mixture was filtered through a short pad of Celite, and the filtrate was extracted with diethyl ether. The organic layer was washed with water and brine, dried over sodium sulfate. After filtration of the mixture and evaporation of the solvent, the crude product was purified by column chromatography (AcOEt / hexane = 1 / 10) to afford aldol **5** (1.72 g, 74%, 94% ee): $[\alpha]_D^{25}$ = +25.1° (c 0.913, benzene); HPLC (CHIRALCEL OD, *i*-PrOH / hexane = 1 / 50, flow rate = 0.7 mL / min): t_R = 11.5 min (3.2%), t_R = 14.2 min (96.8%).



12

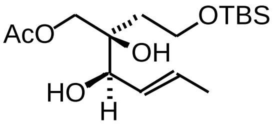
(2*R*,3*R*,4*E*)-2-Benzyloxy-2-(2-[*t*-butyldimethylsiloxy]ethyl)hex-4-ene-1,3-diol (12): To a solution of aldol **5** (4.47 g, 10.9 mmol) in toluene (186 mL) at -45 °C was added Red-Al® in toluene (65%, 32.8 mL, 109 mmol). The reaction mixture was stirred for 20 min at -45 °C and 1 h at 0 °C and then methanol was added. The mixture was allowed to warm to room temperature and then saturated aqueous potassium sodium tartrate was added. The mixture was extracted with ethyl acetate, and the organic layer was washed with water and brine, dried over sodium sulfate. After filtration of the mixture and evaporation of the solvent, the crude product was purified by column chromatography (AcOEt / hexane = 1 / 5) to afford diol **12** (4.12 g, 99%) as a pale yellow oil: $[\alpha]_D^{24}$ = +32.8° (c 1.10, benzene).



13

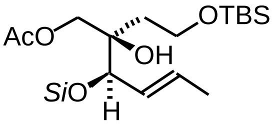
(2*R*,3*R*,4*E*)-2-(2-[*t*-Butyldimethylsiloxy]ethyl)hex-4-ene-1,2,3-triol (13): To a solution of 4,4'-di-*t*-butylbiphenyl (DBB) (26.6 g, 100 mmol) in THF (200 mL) at 0 °C was added lithium (694 mg, 100 mmol). The reaction mixture was stirred for 9 h at room temperature. Thus prepared a solution of lithium di-*t*-butylbiphenylide (LDBB) in THF (0.50 M) was instantly used in the following reaction.

To a solution of diol **12** (2.22 g, 5.84 mmol) in THF (30 mL) at -78 °C was added LDBB in THF (0.50 M, 164 mL, 81.8 mmol). The reaction mixture was stirred for 9 h at -78 °C and then saturated aqueous ammonium chloride was added. The mixture was extracted with ethyl acetate, and the organic layer was washed with water and brine, dried over sodium sulfate. After filtration of the mixture and evaporation of the solvent, the crude product was purified by column chromatography (AcOEt / hexane = 1 / 2) to afford triol **12** (1.69 g, quant.) as a colorless oil: $[\alpha]_D^{23}$ = +21.6° (c 0.70, benzene).



14

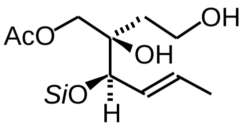
(2*R*,3*R*,4*E*)-2-(2-[*t*-Butyldimethylsiloxy]ethyl)-2,3-dihydroxyhex-4-enyl acetate (14): To a solution of triol **12** (578 mg, 1.99 mmol) in dichloromethane (34 mL) at 0 °C were added a solution of triethylamine (1.00 g, 9.88 mmol) in dichloromethane (3 mL) and a solution of acetic anhydride (305 mL, 2.98 mmol) in dichloromethane (3 mL). The reaction mixture was stirred for 20 h at 0 °C and then saturated aqueous sodium hydrogencarbonate was added. The mixture was extracted with dichloromethane, and organic layer was washed with water and brine, dried over sodium sulfate. After filtration of the mixture and evaporation of the solvent, the crude product was purified by column chromatography (AcOEt / hexane = 1 / 5) to afford acetate **14** (470 mg, 71%) as a colorless oil: $[\alpha]_D^{23}$ = +25.8° (c 1.01, benzene).



Si = $PhMe_2CMe_2Si$

(2*R*,3*R*,4*E*)-2-(2-[*t*-Butyldimethylsiloxy]ethyl)-3-(cumyldimethylsiloxy)-2-hydroxyhex-4-enyl acetate: To a suspension of silver trifluoromethanesulfonate (771 mg, 3.00 mmol) in toluene (4 mL) at 0 °C was added chlorocumyldimethylsilane (638 mg, 3.00 mmol) in toluene (3.5 mL). The reaction mixture was stirred for 1 h at room temperature and then it was allowed to stand. The top clear layer was instantly used as a solution of cumyldimethylsilyl trifluoromethanesulfonate in toluene (0.40 M) for the following reaction without further purification.

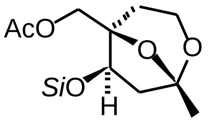
To a solution of acetate **14** (187 mg, 0.562 mmol) in pyridine (5.6 mL) at 0 °C was added a solution of cumyldimethylsilyl trifluoromethanesulfonate in toluene (0.40 M, 2.80 mL, 1.12 mmol). The reaction mixture was stirred for 2 h at 0 °C and then saturated aqueous sodium hydrogencarbonate was added. The mixture was extracted with diethyl ether, and organic layer was washed with saturated aqueous copper(II) sulfate, water and brine, dried over sodium sulfate. After filtration of the mixture and evaporation of the solvent, the crude product was purified by column chromatography (AcOEt / hexane = 1 / 10) to afford (2*R*,3*R*,4*E*)-2-(2-[*t*-butyldimethylsiloxy]ethyl)-3-(cumyldimethylsiloxy)-2-hydroxyhex-4-enyl acetate (265 mg, 93%) as a colorless oil: $[\alpha]_D^{23}$ = +6.3° (c 1.09, benzene).



10

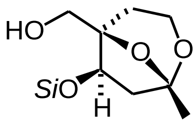
Si = $PhMe_2CMe_2Si$

[2-(1*R*,2*E*)-1-(Cumyldimethylsiloxy)but-2-enyl)](2*R*)-2,4-dihydroxybutyl acetate (10): To a solution of (2*R*,3*R*,4*E*)-2-(2-[*t*-butyldimethylsiloxy]ethyl)-3-(cumyldimethylsiloxy)-2-hydroxyhex-4-enyl acetate (107 mg, 0.211 mmol) in THF (4.2 mL) at -19 °C was added 1 M hydrochloric acid (2.1 mL, 2.1 mmol). The reaction mixture was stirred for 90 min at 0 °C and then saturated aqueous sodium hydrogencarbonate were added. The mixture was extracted with ethyl acetate, and the organic layer was washed with water and brine, dried over sodium sulfate. After filtration of the mixture and evaporation of the solvent, the crude product was purified by thin layer chromatography (AcOEt / hexane = 2 / 3) to afford diol **10** (81.8 mg, 98%) as a colorless oil: $[\alpha]_D^{20}$ = +12.2° (c 2.13, benzene).



Si = $PhMe_2CMe_2Si$

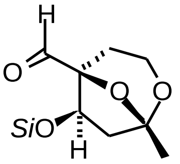
((1*R*,5*S*,7*R*)-7-(Cumyldimethylsiloxy)-5-methyl-4,8-dioxabicyclo[3.2.1]octyl)methyl acetate: To a suspension of palladium(II) chloride (56.0 mg, 0.316 mmol) and copper(I) chloride (85.5 mg, 0.864 mmol) in DME (10 mL) at room temperature under oxygen atmosphere was added a solution of diol **10** (249 mg, 0.631 mmol) in DME (2.6 mL). The reaction mixture was stirred for 7 h at room temperature and then the mixture was diluted with diethyl ether. After filtration of the mixture through a short pad of Florisil® and evaporation of the solvent, the crude product was purified by column chromatography (AcOEt / hexane = 2 / 3) to afford ((1*R*,5*S*,7*R*)-7-(cumyldimethylsiloxy)-5-methyl-4,8-dioxabicyclo[3.2.1]octyl)methyl acetate (218 mg, 88%) as a colorless oil: $[\alpha]_D^{23}$ = –27.4° (c 2.66, benzene).



15

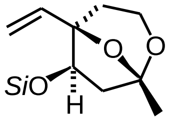
Si = PhMe$_2$CMe$_2$Si

((1*R*,5*S*,7*R*)-7-(Cumyldimethylsiloxy)-5-methyl-4,8-dioxabicyclo[3.2.1]octyl)methanol (15): To a solution of ((1*R*,5*S*,7*R*)-7-(cumyldimethylsiloxy)-5-methyl-4,8-dioxabicyclo[3.2.1]octyl)methyl acetate (218 mg, 0.555 mmol) in methanol (6.3 mL) at 0 °C was added potassium carbonate (95.9 mg, 0.694 mmol). The reaction mixture was stirred for 30 min at room temperature and then water was added at 0 °C. The mixture was extracted with diethyl ether, and the organic layer was washed with water and brine, dried over sodium sulfate. After filtration of the mixture and evaporation of the solvent, the crude product was purified by thin layer chromatography (AcOEt / hexane = 1 / 1) to afford alcohol **15** (194 mg, quant.) as a colorless oil: $[\alpha]_D^{21}$ = –43.4° (c 2.19, benzene).



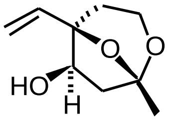
Si = PhMe$_2$CMe$_2$Si

((1*S*,5*S*,7*R*)-7-(Cumyldimethylsiloxy)-5-methyl-4,8-dioxabicyclo[3.2.1]octyl)formaldehyde: To a suspension of MS 4Å (97.7 mg), potassium carbonate (136.6 mg, 0.988 mmol) and NCS (19.6 mg, 0.147 mmol) in dichloromethane (0.3 mL) at 0 °C were added a solution of alcohol **15** (34.4 mg, 98.1 μmol) in dichloromethane (0.8 mL) and a solution of *N*-*t*-butylbenzenesulfenamide (2.5 mg, 13.8 μmol) in dichloromethane (0.4 mL). After the reaction mixture had been stirred for 90 min at room temperature, the mixture was filtered through a short pad of Celite and saturated aqueous ammonium chloride was added to the filtrate. The mixture was extracted with dichloromethane, and the organic layer was washed with saturated aqueous ammonium chloride, water and brine, dried over sodium sulfate. After filtration of the mixture and evaporation of the solvent, the crude product was purified by thin layer chromatography (AcOEt / hexane = 1 / 6) to afford ((1*S*,5*S*,7*R*)-7-(cumyldimethylsiloxy)-5-methyl-4,8-dioxabicyclo[3.2.1]octyl)formaldehyde (34.2 mg, quant.) as a colorless oil: $[\alpha]_D^{21}$ = –96.2° (c 1.45, benzene).



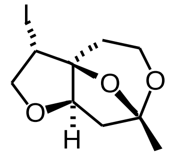
Si = $PhMe_2CMe_2Si$

(1*S*,5*R*,6*R*)-6-(Cumyldimethylsiloxy)-1-methyl-2,8-dioxa-5-vinylbicyclo[3.2.1]octane: To a solution of methyltriphenylphosphonium iodide (880 mg, 2.18 mmol) in THF (2.6 mL) at -78 °C was added KHMDS in toluene (0.50 M, 2.60 mL, 1.30 mmol). After the reaction mixture had been stirred for 30 min at -78 °C, a solution of ((1*S*,5*S*,7*R*)-7-(cumyldimethylsiloxy)-5-methyl-4,8-dioxabicyclo[3.2.1]octyl)formaldehyde (40.0 mg, 0.115 mmol) in THF (1.0 mL) was added. The reaction mixture was stirred for 30 min at room temperature and then the mixture was diluted with hexane. After filtration of the mixture through a short pad of Celite with hexane and evaporation of the solvent, the crude product was purified by thin layer chromatography (AcOEt / hexane = 1 / 4) to afford (1*S*,5*R*,6*R*)-6-(cumyldimethylsiloxy)-1-methyl-2,8-dioxa-5-vinylbicyclo[3.2.1]octane (40.0 mg, quant.) as a colorless oil: $[\alpha]_D^{26}$ = –19.9° (c 1.58, benzene).



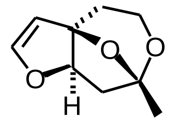
16

(1*S*,5*R*,6*R*)-1-Methyl-2,8-dioxa-5-vinylbicyclo[3.2.1]octan-6-ol (16): To a solution of (1*S*,5*R*,6*R*)-6-(cumyldimethylsiloxy)-1-methyl-2,8-dioxa-5-vinylbicyclo[3.2.1]octane (13.1 mg, 37.8 μmol) in THF (0.5 mL) at 0 °C was added a solution of TBAF in THF (1.0 M, 42.0 μL, 42.0 μmol). The reaction mixture was stirred for 1 h at 0 °C and then phosphate buffer (pH = 7) was added. The mixture was extracted with dichloromethane, and the organic layer was washed with water and brine, dried over sodium sulfate. After filtration of the mixture and evaporation of the solvent, the crude product was purified by thin layer chromatography (AcOEt / hexane = 1 / 1) to afford alcohol **16** (6.5 mg, quant.) as a colorless oil: $[\alpha]_D^{24}$ = –17.5° (c 1.33, benzene).

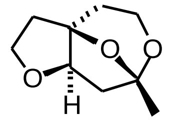


17

(1*S*,5*R*,7*S*)-2-Iodo-7-methyl-4,8,11-trioxatricyclo[5.3.1.0^{1,5}]undecane (17): To a solution of alcohol **16** (3.0 mg, 17.6 μmol) in acetonitrile (0.3 mL) at 0 °C were added iodine (6.7 mg, 26.4 μmol) and sodium hydrogencarbonate (9.0 mg, 0.107 mmol). The reaction mixture was stirred for 1 h at room temperature and then saturated aqueous sodium thiosulfate was added. The mixture was extracted with diethyl ether and the organic layer was washed with water and brine, dried over sodium sulfate. After filtration of the mixture and evaporation of the solvent, the crude product was purified by thin layer chromatography (AcOEt / hexane = 1 / 1) to afford iodide **17** (4.2 mg, 80%) as a colorless oil: $[\alpha]_D^{23}$ = –131.4° (c 1.09, benzene).



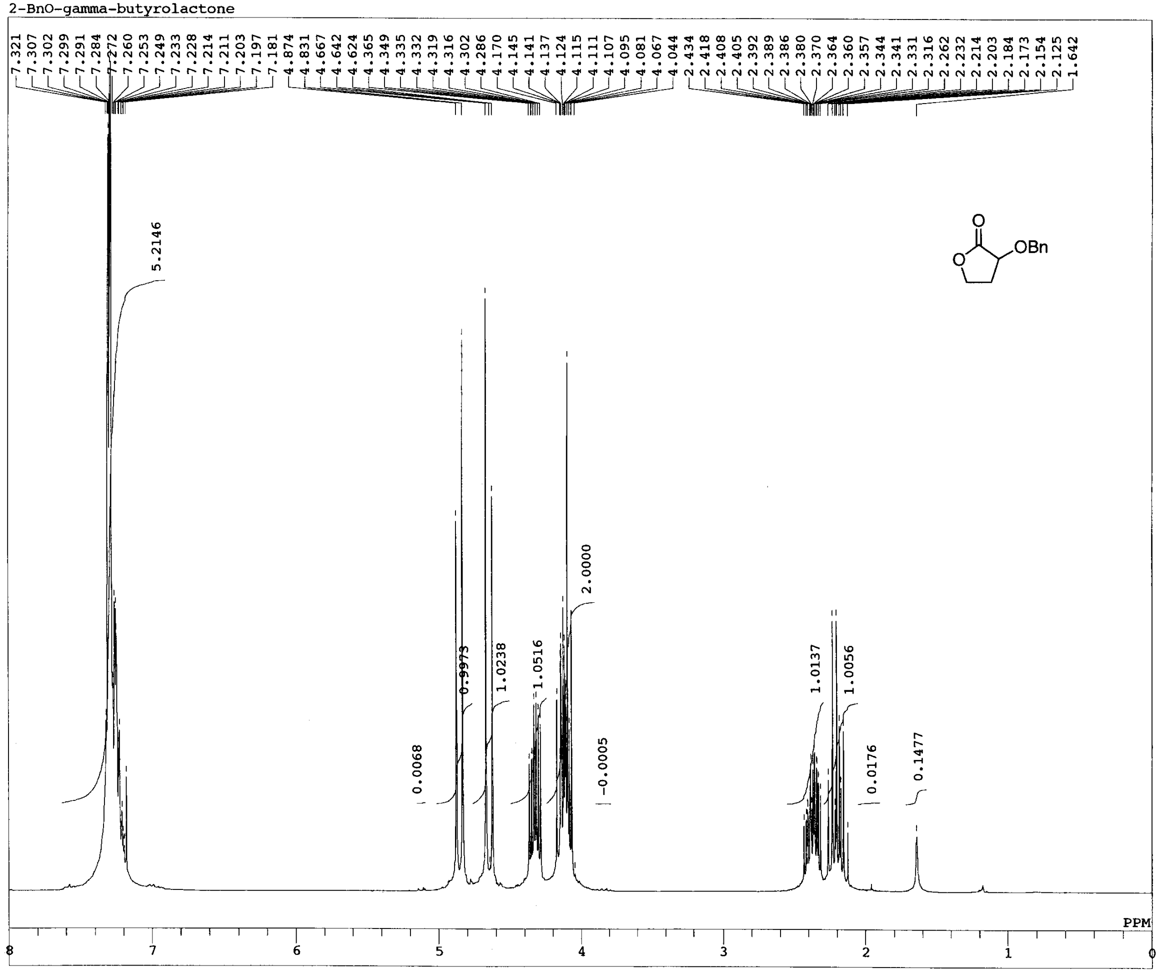
(1*R*,5*R*,7*S*)-7-Methyl-4,8,11-trioxatricyclo[5.3.1.0^{1,5}]undec-2-ene: To a solution of iodide **17** (3.1 mg, 10.5 μmol) in DMSO (1 mL) at room temperature was added potassium *t*-butoxide (12.0 mg, 0.107 mmol). The reaction mixture was stirred for 5 min at room temperature and then saturated aqueous ammonium chloride was added. The mixture was extracted with diethyl ether, and the organic layer was washed with water and brine, and dried over sodium sulfate. After filtration of the mixture and evaporation of the solvent, the crude product was purified by thin layer chromatography (AcOEt / hexane = 1 / 1) to afford (1*R*,5*R*,7*S*)-7-methyl-4,8,11-trioxatricyclo[5.3.1.0^{1,5}]undec-2-ene (1.8 mg, quant.) as a colorless oil: $[\alpha]_D^{23}$ = –44.4° (c 0.44, benzene).



(+)-**1**

(+)-Buergerinin F (1): To a solution of (1*R*,5*R*,7*S*)-7-methyl-4,8,11-trioxatricyclo[5.3.1.0^{1,5}]undec-2-ene (1.8 mg, 10.7 μmol) in ethyl acetate (1 mL) at room temperature was added 10% palladium on activated carbon (10.4 mg). The reaction mixture was stirred for 30 min at room temperature under hydrogen atmosphere. After filtration of the mixture and evaporation of the solvent at 0 °C, the crude product was purified by thin layer chromatography (AcOEt / hexane = 1 / 1) to afford buergerinin F (**1**) (1.8 mg, quant.) as a colorless oil: $[\alpha]_D^{23}$ = +38.1° (c 0.41, $CHCl_3$).

2-BnO-gamma-butyrolactone



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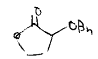
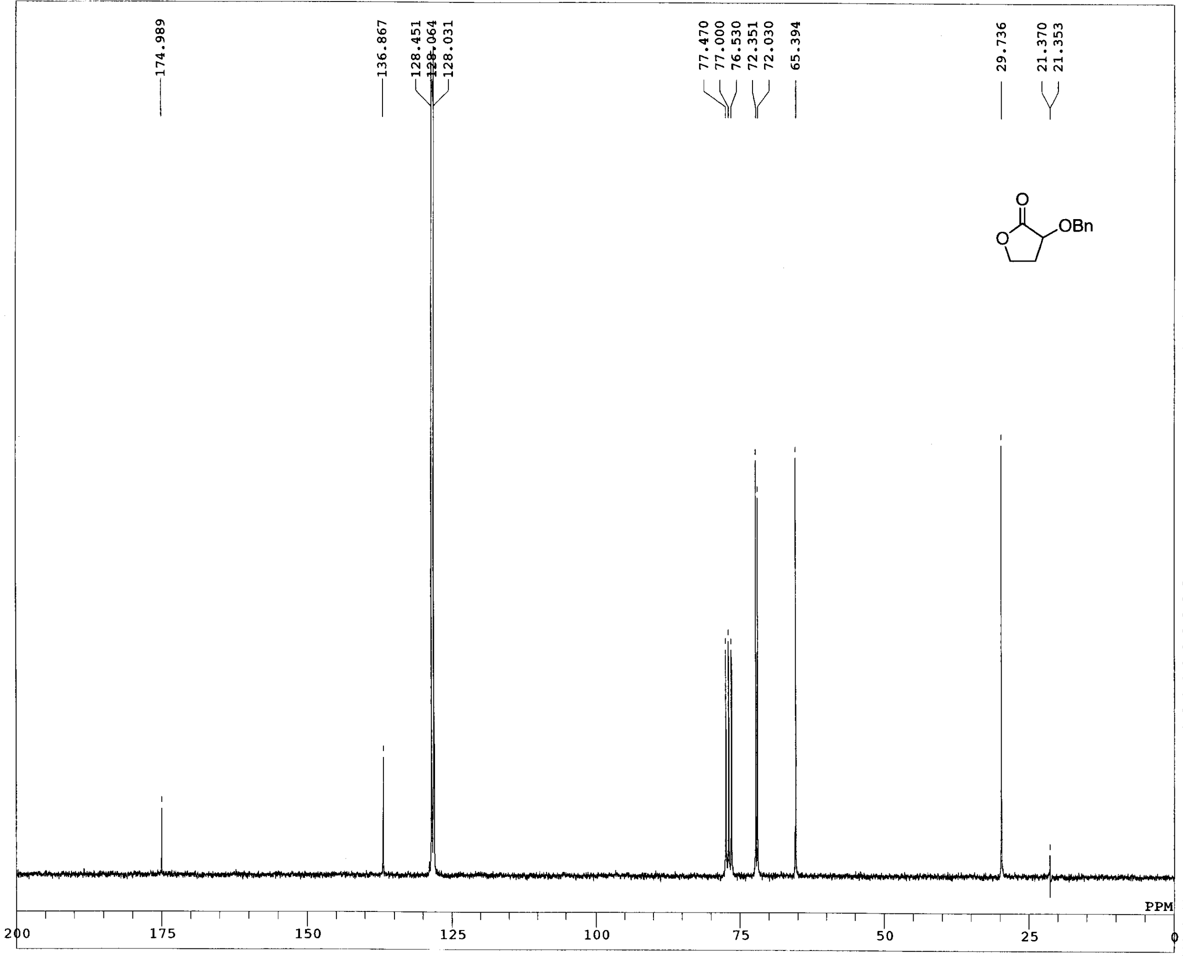
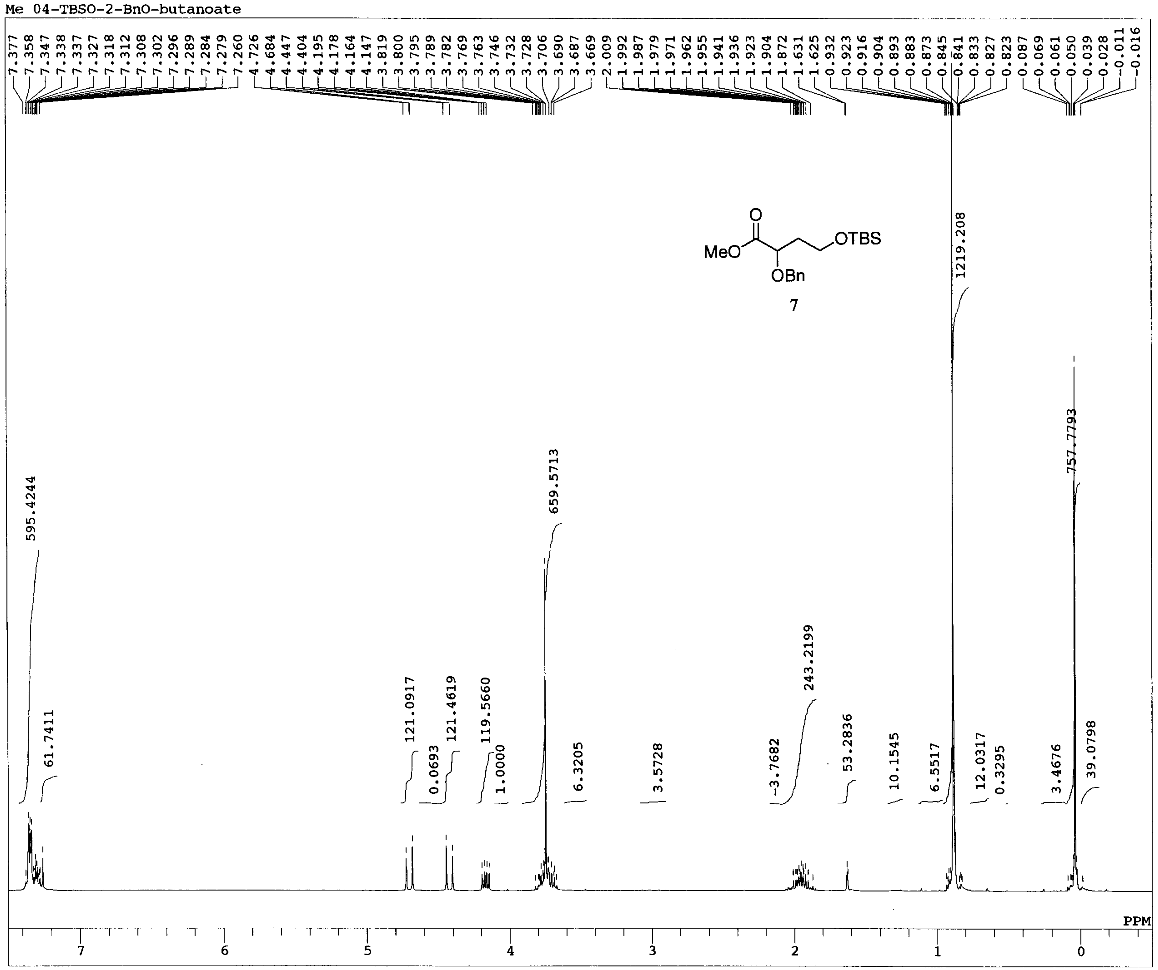
PI2         1.000 ms
PI3          1.00 ms
PI4          1.00 ms
PI5          1.00 ms
LOOP1           1
LOOP2           1
LOOP3           1
LOOP4           1
LOOP5           1
PS1         100.0 us
```



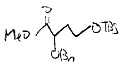
2-BnO-gamma-butyrolactone



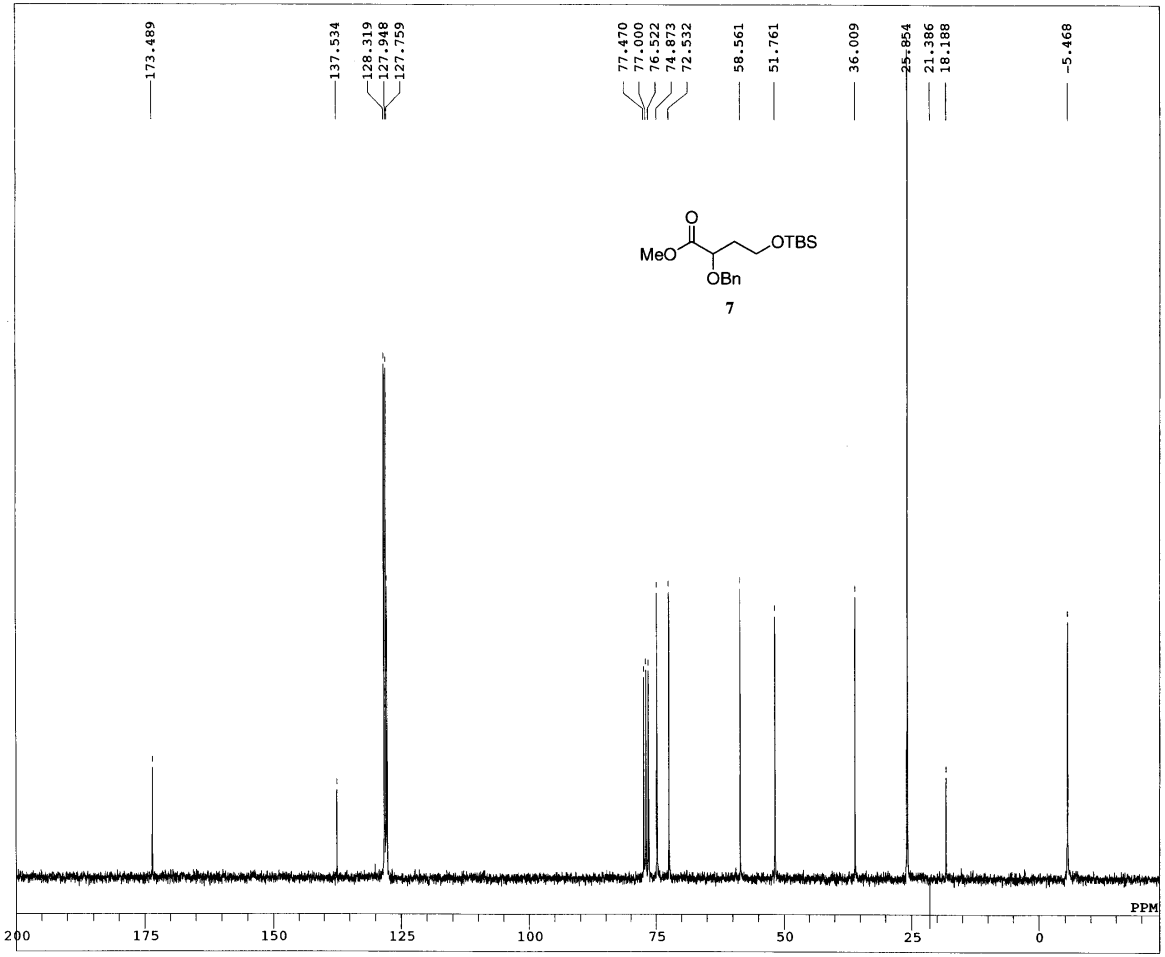
```
SPEED          12 Hz
CSPED          13 Hz
TEMP         80.0 c
CTEMP        21.9 c
TCOMP        1.00
SNO             1
HELEV       88.40
N2LEV      100.00
DATIM  Sat Aug 31 18:36:11 2002
PROBE  TH5
IRCODE        146
PRCODE       1412
DFILE  C:\WINNMR95\TEMPDATA\AUTO2BCM_E1_F
MENUF  BCM
SHMFL  TH5
COMNT  2-BnO-gamma-butyrolactone
SHMCM  th5/2%age/CDCl3/40mm/17JAN02
SLVNT  CDCL3
OBNUC  13C
OBFRQ       67.80 MHz
OBSET      135.00 KHz
OBFIN      5200.0 Hz
OBST2        0.00 KHz
OBFN2         0.0 Hz
OBPHS         0.0 deg
OBATN         511
IRNUC  1H
IRFRQ      270.05 MHz
IRSET      112.00 KHz
IRFIN      5800.0 Hz
IRST2       50.00 KHz
IRFN2      3700.0 Hz
IRPHS         0.0 deg
IRATN         511
IRRPW          40 us
IRRNS           0
TRNUC  1H
TRFRQ      270.05 MHz
TRSET      112.00 KHz
TRFIN      5800.0 Hz
TRST2        0.00 KHz
TRFN2         0.0 Hz
TRPHS         0.0 deg
TRATN         511
TRRPW          70 us
TRRNS           0
PW1           3.7 us
PW2          10.0 us
PW3          10.0 us
PW4           1.0 us
PW5           1.0 us
PI1         1.000 ms
PI2         1.000 ms
PI3          1.00 ms
PI4          1.00 ms
PI5          1.00 ms
LOOP1           1
LOOP2           1
LOOP3           1
LOOP4           1
LOOP5           1
PS1         100.0 us
```



```
SPEED           12 Hz
CSPED           14 Hz
TEMP          80.0 c
CTEMP         21.2 c
TCOMP         1.00
SNO              1
HELEV        88.40
N2LEV       100.00
DATIM   Sat Aug 31 19:31:22 2002
PROBE   TH5
IRCODE         146
PRCODE        1412
DFILE   C:\WINNMR95\DATA\Auto1NON_E1_FT.al
MENUF   NON
SHMFL   TH5
COMNT   Me 04-TBSO-2-BnO-butanoate
SHMCM   th5/2%age/CDCl3/40mm/17JAN02
SLVNT   CDCL3
OBNUC   1H
OBFRQ       270.05 MHz
OBSET       112.00 KHz
OBFIN       5800.0 Hz
OBST2         0.00 KHz
OBFN2          0.0 Hz
OBPHS          0.0 deg
OBATN          511
IRNUC   1H
IRFRQ       270.05 MHz
IRSET       112.00 KHz
IRFIN       5800.0 Hz
IRST2        50.00 KHz
IRFN2       3700.0 Hz
IRPHS          0.0 deg
IRATN          511
IRRPW           50 us
IRRNS            0
TRNUC   1H
TRFRQ       270.05 MHz
TRSET       112.00 KHz
TRFIN       5800.0 Hz
TRST2         0.00 KHz
TRFN2          0.0 Hz
TRPHS          0.0 deg
TRATN          511
TRRPW           70 us
TRRNS            0
PW1            5.5 us
PW2           10.0 us
PW3           10.0 us
PW4            1.0 us
PW5            1.0 us
PI1          1.000 ms
PI2          1.000 ms
PI3           1.00 ms
PI4           1.00 ms
PI5           1.00 ms
LOOP1            1
LOOP2            1
LOOP3            1
LOOP4            1
LOOP5            1
PS1          100.0 us
```

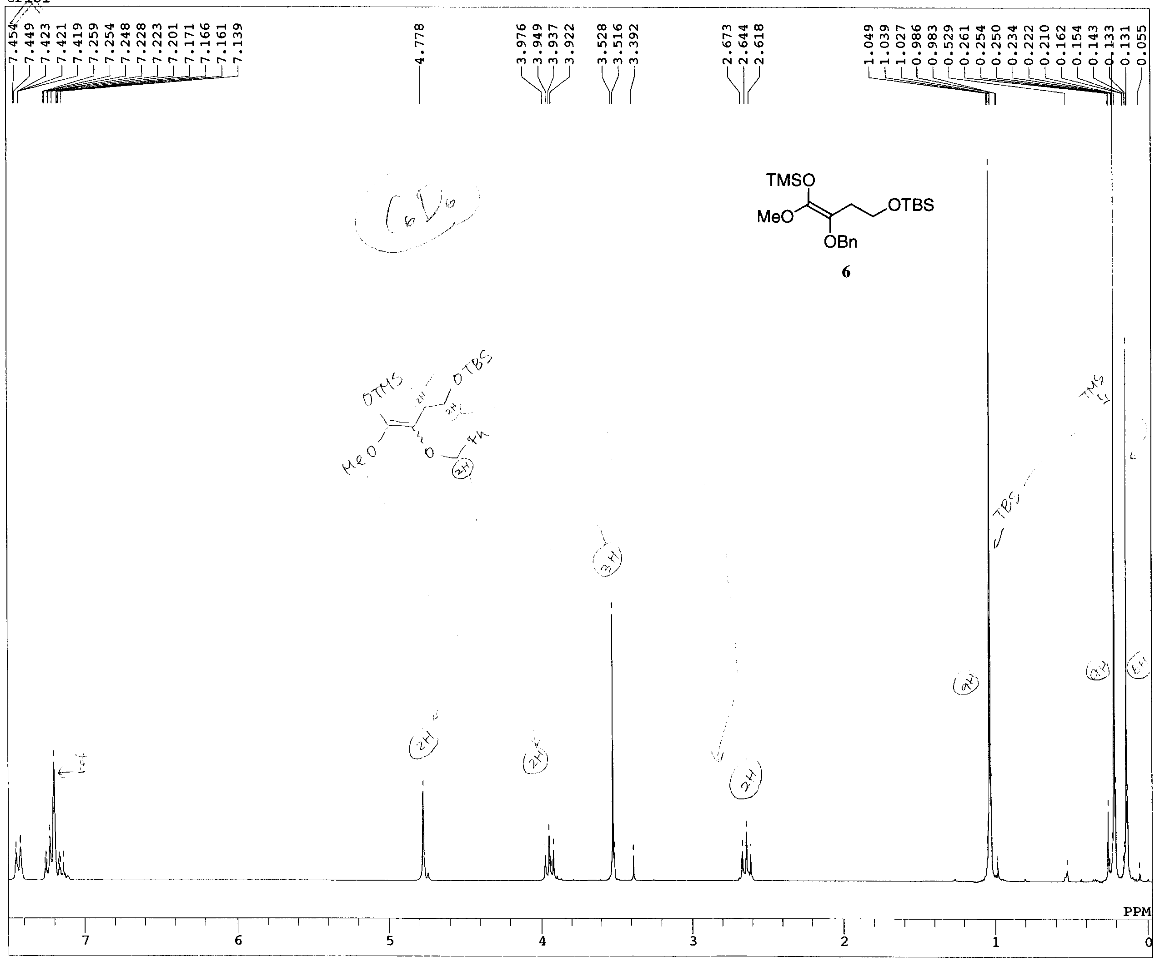


Me 04-TBSO-2-BnO-butanoate



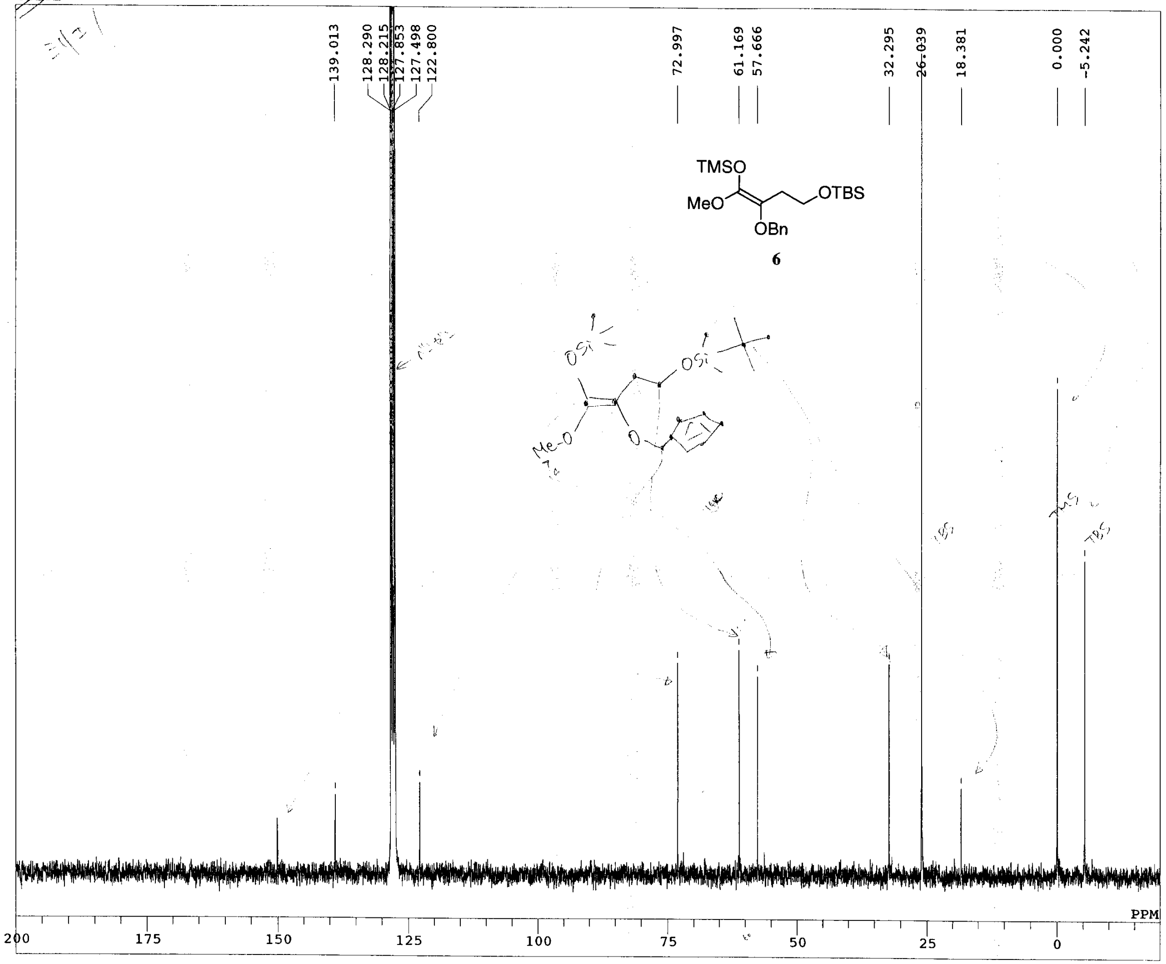
```
SPEED          12 Hz
CSPED          13 Hz
TEMP         80.0 c
CTEMP        21.2 c
TCOMP        1.00
SNO             1
HELEV       88.40
N2LEV      100.00
DATIM  Sat Aug 31 19:13:57 2002
PROBE  TH5
IRCODE        146
PRCODE       1412
DFILE  C:\WINNMR95\TEMPDATA\AUTO2BCM_E1_F
MENUF  BCM
SHMFL  TH5
COMNT  Me 04-TBSO-2-BnO-butanoate
SHMCM  th5/2%age/CDCl3/40mm/17JAN02
SLVNT  CDCL3
OBNUC  13C
OBFRQ       67.80 MHz
OBSET      135.00 KHz
OBFIN      5200.0 Hz
OBST2        0.00 KHz
OBFN2         0.0 Hz
OBPHS         0.0 deg
OBATN         511
IRNUC  1H
IRFRQ      270.05 MHz
IRSET      112.00 KHz
IRFIN      5800.0 Hz
IRST2       50.00 KHz
IRFN2      3700.0 Hz
IRPHS         0.0 deg
IRATN         511
IRRPW          40 us
IRRNS           0
TRNUC  1H
TRFRQ      270.05 MHz
TRSET      112.00 KHz
TRFIN      5800.0 Hz
TRST2        0.00 KHz
TRFN2         0.0 Hz
TRPHS         0.0 deg
TRATN         511
TRRPW          70 us
TRRNS           0
PW1           3.7 us
PW2          10.0 us
PW3          10.0 us
PW4           1.0 us
PW5           1.0 us
PI1         1.000 ms
PI2         1.000 ms
PI3          1.00 ms
PI4          1.00 ms
PI5          1.00 ms
LOOP1           1
LOOP2           1
LOOP3           1
LOOP4           1
LOOP5           1
PS1         100.0 us
```

```
C:\WINNMR98\DATA\Auto1NON_E1_FT.als
triol
```



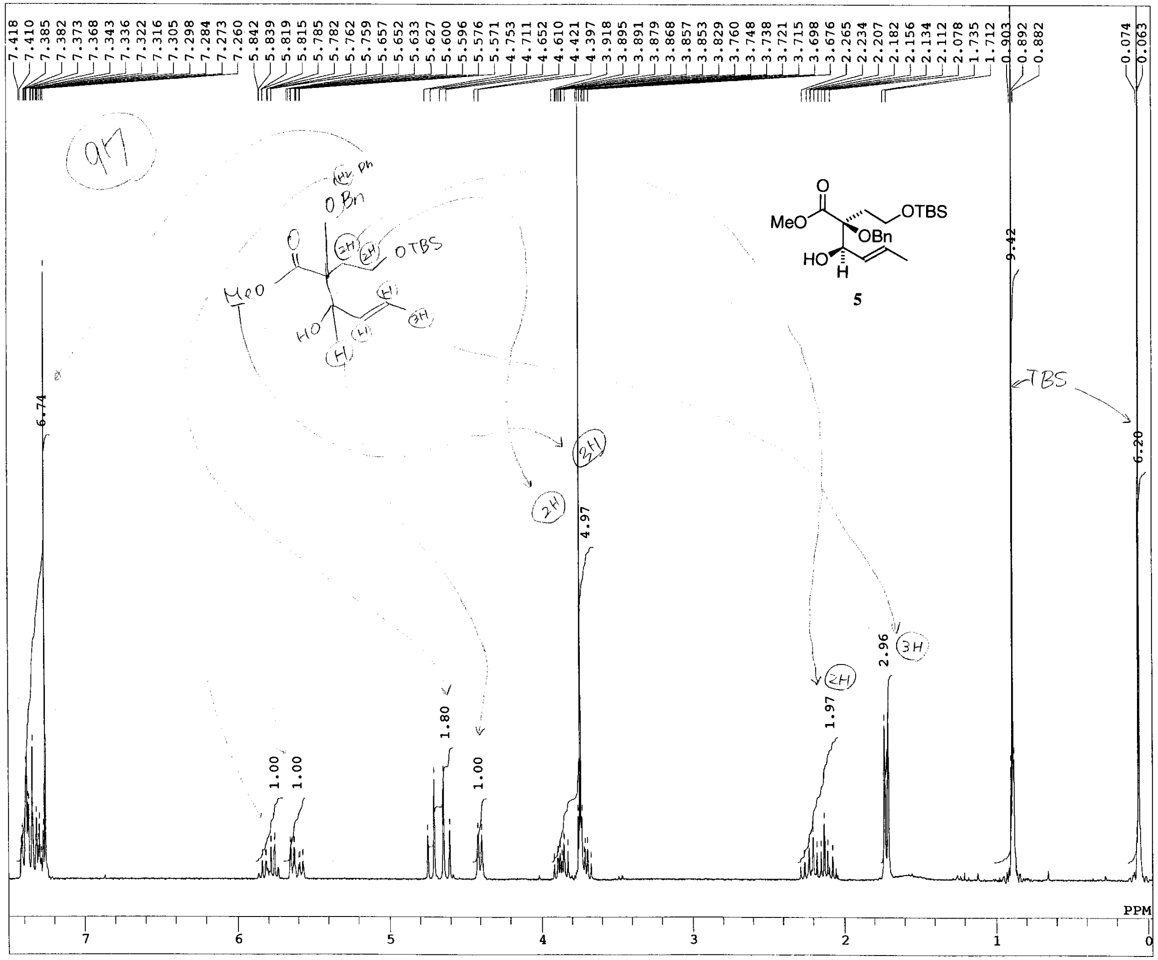
```
SPEED           12 Hz
CSPED           10 Hz
TEMP          20.0 c
CTEMP         22.4 c
TCOMP         1.00
SNO              1
HELEV        30.00
N2LEV       100.00
DATIM  Sun May 18 16:00:55 2003
PROBE  TH5
IRCODE         146
PRCODE        1412
DFILE  C:\WINNMR98\DATA\Auto1NON_E1_FT.al
MENUF  NON
SHMFL  TH5
COMNT  triol
SHMCM  th5/2%age/CDCl3/40mm/17JAN02
SLVNT  C6D6
OBNUC  1H
OBFRQ       270.05 MHz
OBSET       112.00 KHz
OBFIN       5800.0 Hz
OBST2         0.00 KHz
OBFN2          0.0 Hz
OBPHS          0.0 deg
OBATN          511
IRNUC  1H
IRFRQ       270.05 MHz
IRSET       112.00 KHz
IRFIN       5800.0 Hz
IRST2        50.00 KHz
IRFN2       3700.0 Hz
IRPHS          0.0 deg
IRATN          511
IRRPW           50 us
IRRNS            0
TRNUC  1H
TRFRQ       270.05 MHz
TRSET       112.00 KHz
TRFIN       5800.0 Hz
TRST2         0.00 KHz
TRFN2          0.0 Hz
TRPHS          0.0 deg
TRATN          511
TRRPW           70 us
TRRNS            0
PW1            5.5 us
PW2           10.0 us
PW3           10.0 us
PW4            1.0 us
PW5            1.0 us
PI1          1.000 ms
PI2          1.000 ms
PI3           1.00 ms
PI4           1.00 ms
PI5           1.00 ms
LOOP1            1
LOOP2            1
LOOP3            1
LOOP4            1
LOOP5            1
PS1          100.0 us
```

C:\WINNMR98\TEMPDATA\Auto2BCM_E1_FT.als
triol



```
SPEED          12 Hz
CSPED          14 Hz
TEMP         20.0 c
CTEMP        22.4 c
TCOMP        1.00
SNO             1
HELEV       30.00
N2LEV      100.00
DATIM  Sun May 18 16:26:19 2003
PROBE  TH5
IRCODE        146
PRCODE       1412
DFILE  C:\WINNMR98\TEMPDATA\Auto2BCM_E1_F
MENUF  BCM
SHMFL  TH5
COMNT  triol
SHMCM  th5/2%age/CDCl3/40mm/17JAN02
SLVNT  C6D6
OBNUC  13C
OBFRQ       67.80 MHz
OBSET      135.00 KHz
OBFIN      5200.0 Hz
OBST2        0.00 KHz
OBFN2         0.0 Hz
OBPHS         0.0 deg
OBATN         511
IRNUC  1H
IRFRQ      270.05 MHz
IRSET      112.00 KHz
IRFIN      5800.0 Hz
IRST2       50.00 KHz
IRFN2      3700.0 Hz
IRPHS         0.0 deg
IRATN         511
IRRPW          40 us
IRRNS           0
TRNUC  1H
TRFRQ      270.05 MHz
TRSET      112.00 KHz
TRFIN      5800.0 Hz
TRST2        0.00 KHz
TRFN2         0.0 Hz
TRPHS         0.0 deg
TRATN         511
TRRPW          70 us
TRRNS           0
PW1           3.7 us
PW2          10.0 us
PW3          10.0 us
PW4           1.0 us
PW5           1.0 us
PI1         1.000 ms
PI2         1.000 ms
PI3          1.00 ms
PI4          1.00 ms
PI5          1.00 ms
LOOP1           1
LOOP2           1
LOOP3           1
LOOP4           1
LOOP5           1
PS1         100.0 us
```

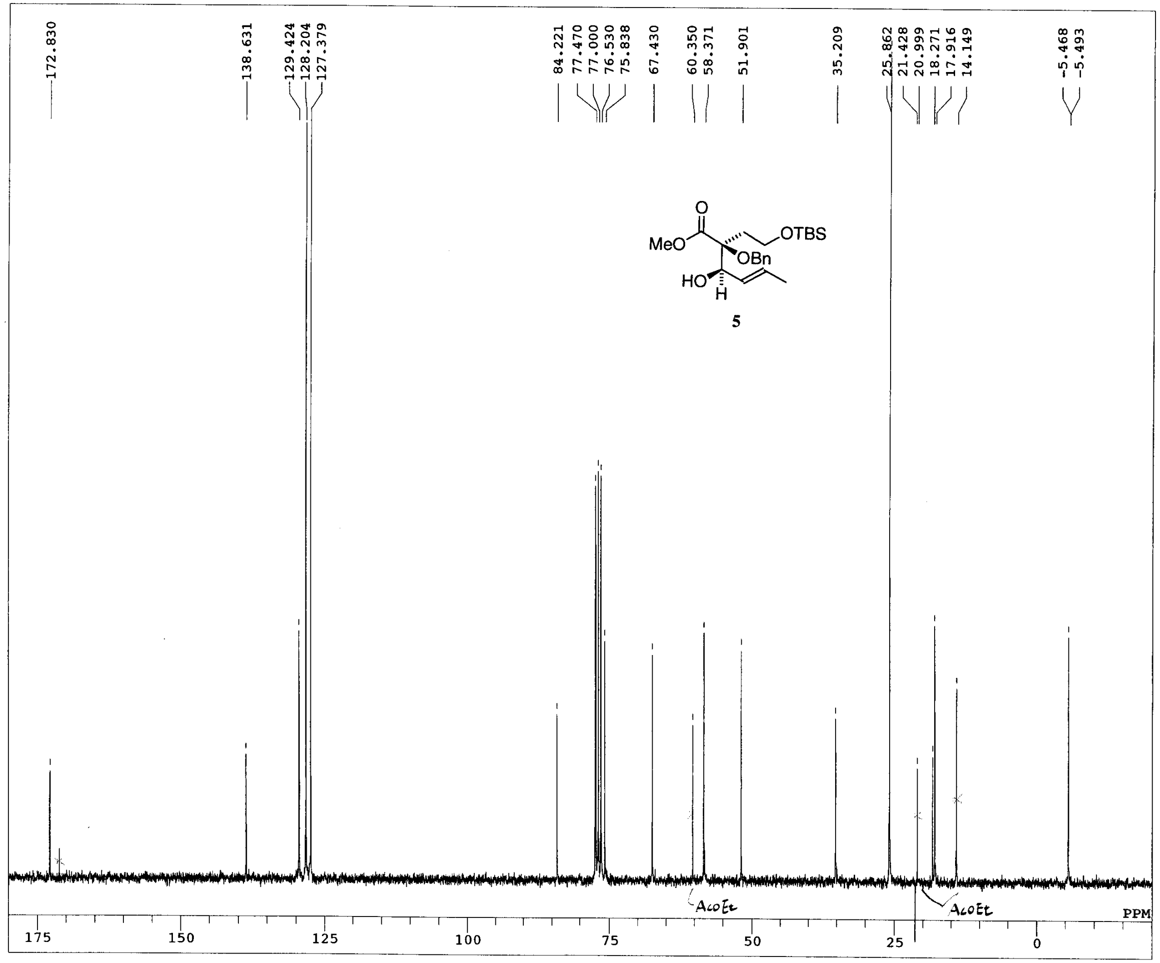
C:\WINNMR98\DATA\Auto1NON_E1_FT.als
chiral 97 down



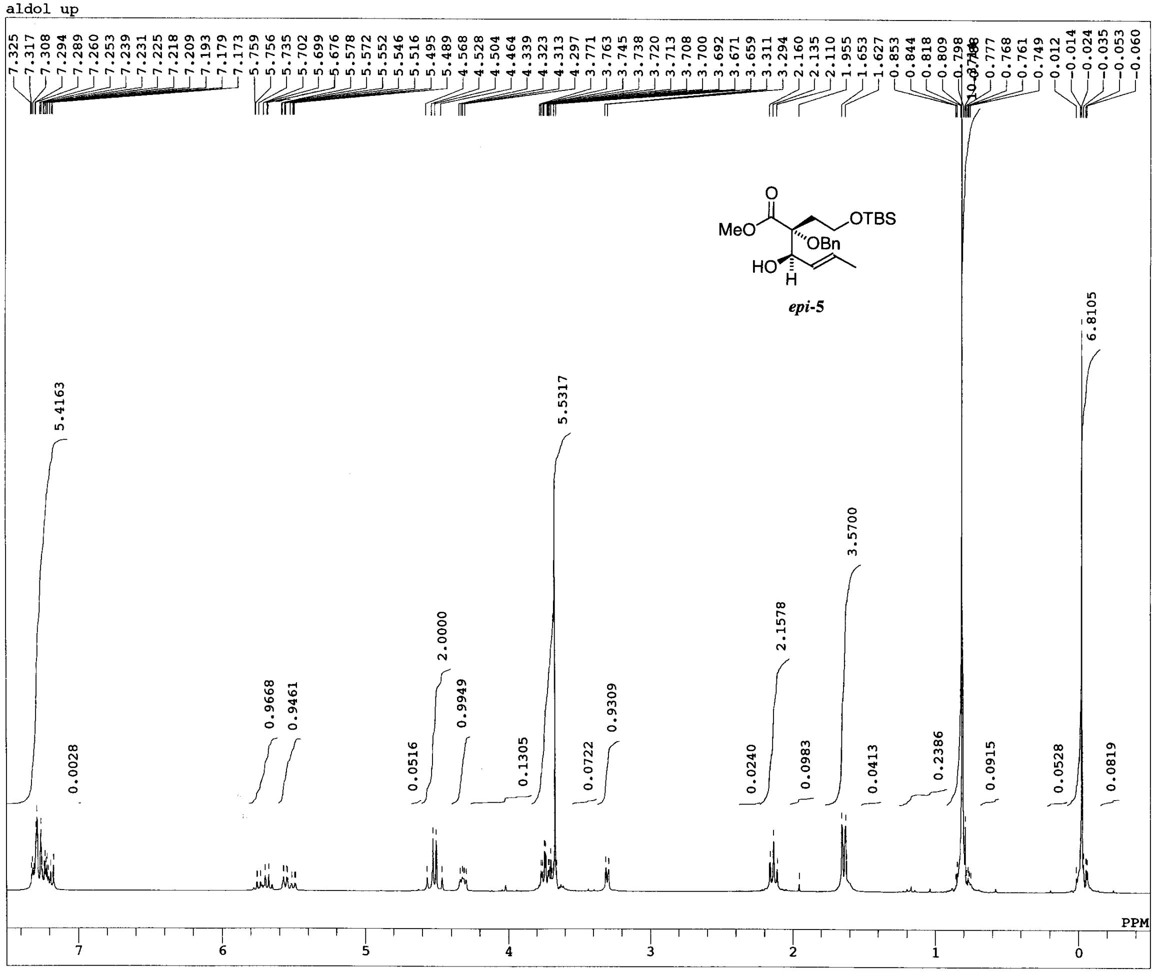
5

```
SPEED          12 Hz
CSPED          13 Hz
TEMP         20.0 c
CTEMP        21.7 c
TCOMP        1.00
SNO             1
HELEV       92.00
N2LEV      100.00
DATIM   Tue May 27 17:17:52 2003
PROBE   TH5
IRCODE        146
PRCODE       1412
DFILE   C:\WINNMR98\DATA\Auto1NON_E1_FT.al
MENUF   NON
SHMFL   TH5
COMNT   chiral 97 down
SHMCM   th5/2%age/CDCl3/40mm/17JAN02
SLVNT   CDCL3
OBNUC   1H
OBFRQ      270.05 MHz
OBSET      112.00 KHz
OBFIN      5800.0 Hz
OBST2        0.00 KHz
OBFN2         0.0 Hz
OBPHS         0.0 deg
OBATN         511
IRNUC   1H
IRFRQ      270.05 MHz
IRSET      112.00 KHz
IRFIN      5800.0 Hz
IRST2       50.00 KHz
IRFN2      3700.0 Hz
IRPHS         0.0 deg
IRATN         511
IRRPW          50 us
IRRNS           0
TRNUC   1H
TRFRQ      270.05 MHz
TRSET      112.00 KHz
TRFIN      5800.0 Hz
TRST2        0.00 KHz
TRFN2         0.0 Hz
TRPHS         0.0 deg
TRATN         511
TRRPW          70 us
TRRNS           0
PW1           5.5 us
PW2          10.0 us
PW3          10.0 us
PW4           1.0 us
PW5           1.0 us
PI1         1.000 ms
PI2         1.000 ms
PI3          1.00 ms
PI4          1.00 ms
PI5          1.00 ms
LOOP1           1
LOOP2           1
LOOP3           1
LOOP4           1
LOOP5           1
PS1         100.0 us
```

aldol down



```
SPEED          12 Hz
CSPED          10 Hz
TEMP         80.0 c
CTEMP        21.2 c
TCOMP        1.00
SNO             1
HELEV       88.40
N2LEV      100.00
DATIM   Sat Aug 31 21:30:45 2002
PROBE   TH5
IRCODE        146
PRCODE       1412
DFILE   C:\WINNMR95\DATA\Auto2BCM_E1_FT.al
MENUF   BCM
SHMFL   TH5
COMNT   aldol down
SHMCM   th5/2%age/CDCl3/40mm/17JAN02
SLVNT   CDCL3
OBNUC   13C
OBFRQ       67.80 MHz
OBSET      135.00 KHz
OBFIN      5200.0 Hz
OBST2        0.00 KHz
OBFN2         0.0 Hz
OBPHS         0.0 deg
OBATN         511
IRNUC   1H
IRFRQ      270.05 MHz
IRSET      112.00 KHz
IRFIN      5800.0 Hz
IRST2       50.00 KHz
IRFN2      3700.0 Hz
IRPHS         0.0 deg
IRATN         511
IRRPW          40 us
IRRNS           0
TRNUC   1H
TRFRQ      270.05 MHz
TRSET      112.00 KHz
TRFIN      5800.0 Hz
TRST2        0.00 KHz
TRFN2         0.0 Hz
TRPHS         0.0 deg
TRATN         511
TRRPW          70 us
TRRNS           0
PW1           3.7 us
PW2          10.0 us
PW3          10.0 us
PW4           1.0 us
PW5           1.0 us
PI1         1.000 ms
PI2         1.000 ms
PI3          1.00 ms
PI4          1.00 ms
PI5          1.00 ms
LOOP1           1
LOOP2           1
LOOP3           1
LOOP4           1
LOOP5           1
PS1         100.0 us
```



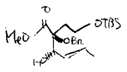
epi-5

```
SPEED           12 Hz
CSPED           10 Hz
TEMP          80.0 c
CTEMP         22.0 c
TCOMP         1.00
SNO              1
HELEV        88.40
N2LEV       100.00
DATIM  Sat Aug 31 20:07:48 2002
PROBE  TH5
IRCODE         146
PRCODE        1412
DFILE  C:\WINNMR95\DATA\Auto1NON_E1_FT.al
MENUF  NON
SHMFL  TH5
COMNT  aldol up
SHMCM  th5/2%age/CDCl3/40mm/17JAN02
SLVNT  CDCL3
OBNUC  1H
OBFRQ       270.05 MHz
OBSET       112.00 KHz
OBFIN       5800.0 Hz
OBST2         0.00 KHz
OBFN2          0.0 Hz
OBPHS          0.0 deg
OBATN          511
IRNUC  1H
IRFRQ       270.05 MHz
IRSET       112.00 KHz
IRFIN       5800.0 Hz
IRST2        50.00 KHz
IRFN2       3700.0 Hz
IRPHS          0.0 deg
IRATN          511
IRRPW           50 us
IRRNS            0
TRNUC  1H
TRFRQ       270.05 MHz
TRSET       112.00 KHz
TRFIN       5800.0 Hz
TRST2         0.00 KHz
TRFN2          0.0 Hz
TRPHS          0.0 deg
TRATN          511
TRRPW           70 us
TRRNS            0
PW1            5.5 us
PW2           10.0 us
PW3           10.0 us
PW4            1.0 us
PW5            1.0 us
PI1          1.000 ms
PI2          1.000 ms
PI3           1.00 ms
PI4           1.00 ms
PI5           1.00 ms
LOOP1            1
LOOP2            1
LOOP3            1
LOOP4            1
LOOP5            1
PS1          100.0 us
```

TG45 up

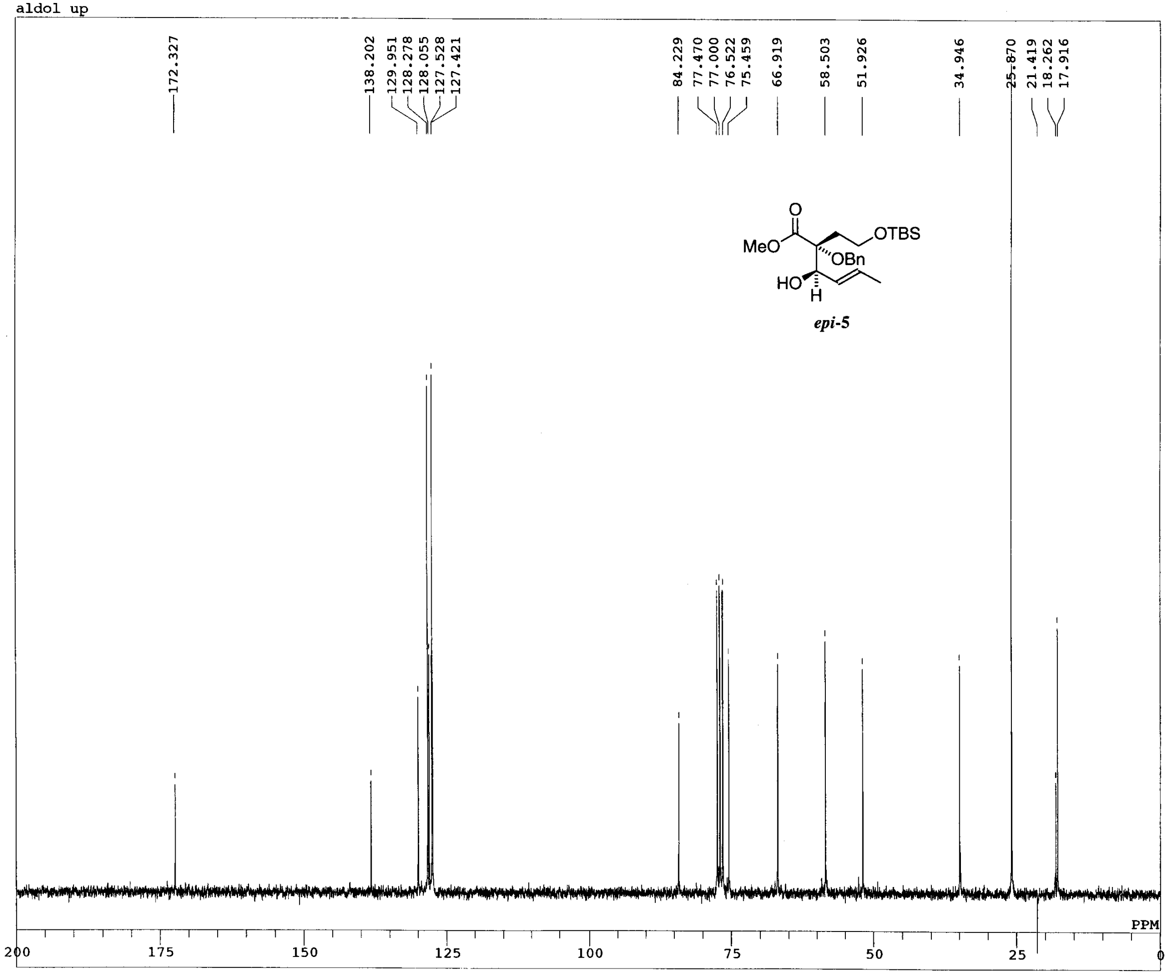
minor

undesired

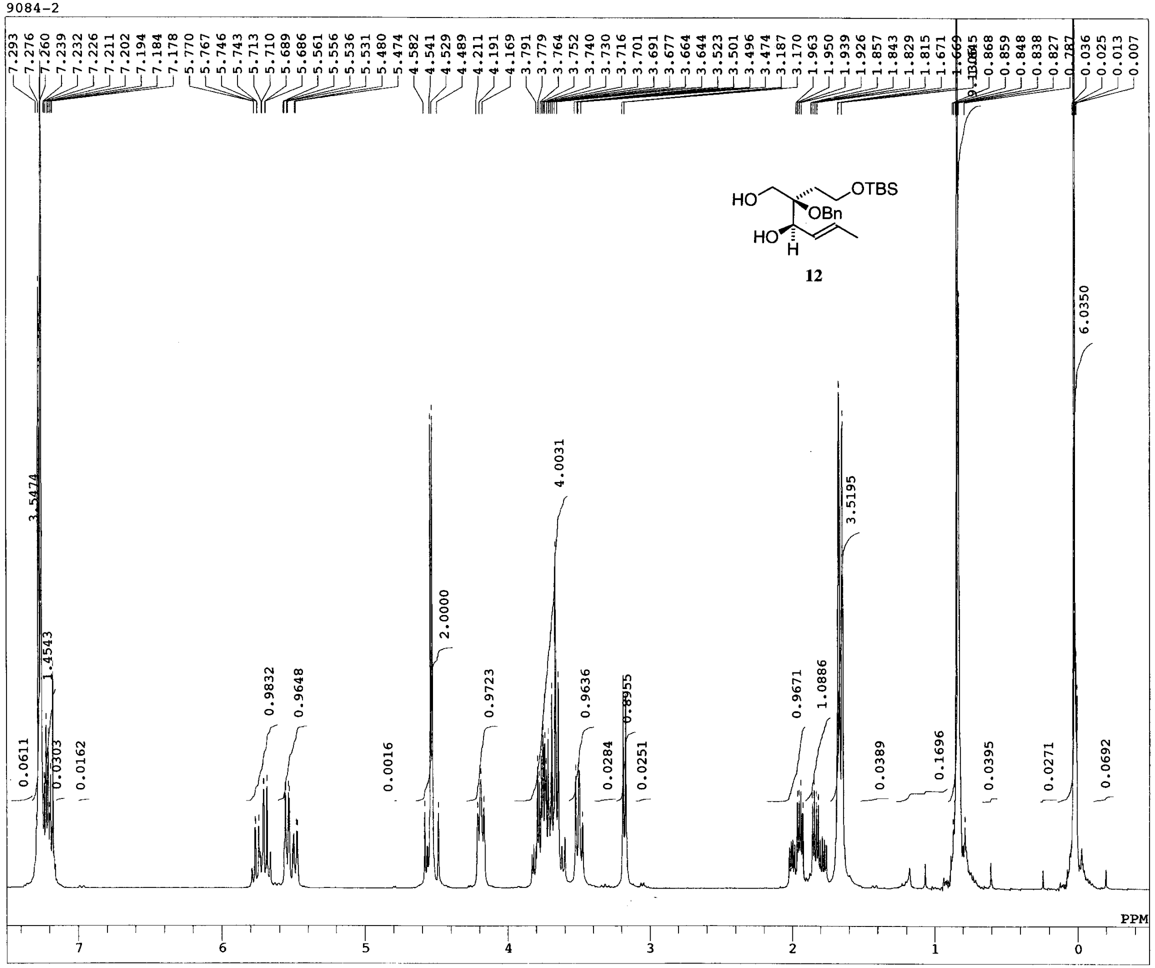


up

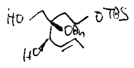
aldol up

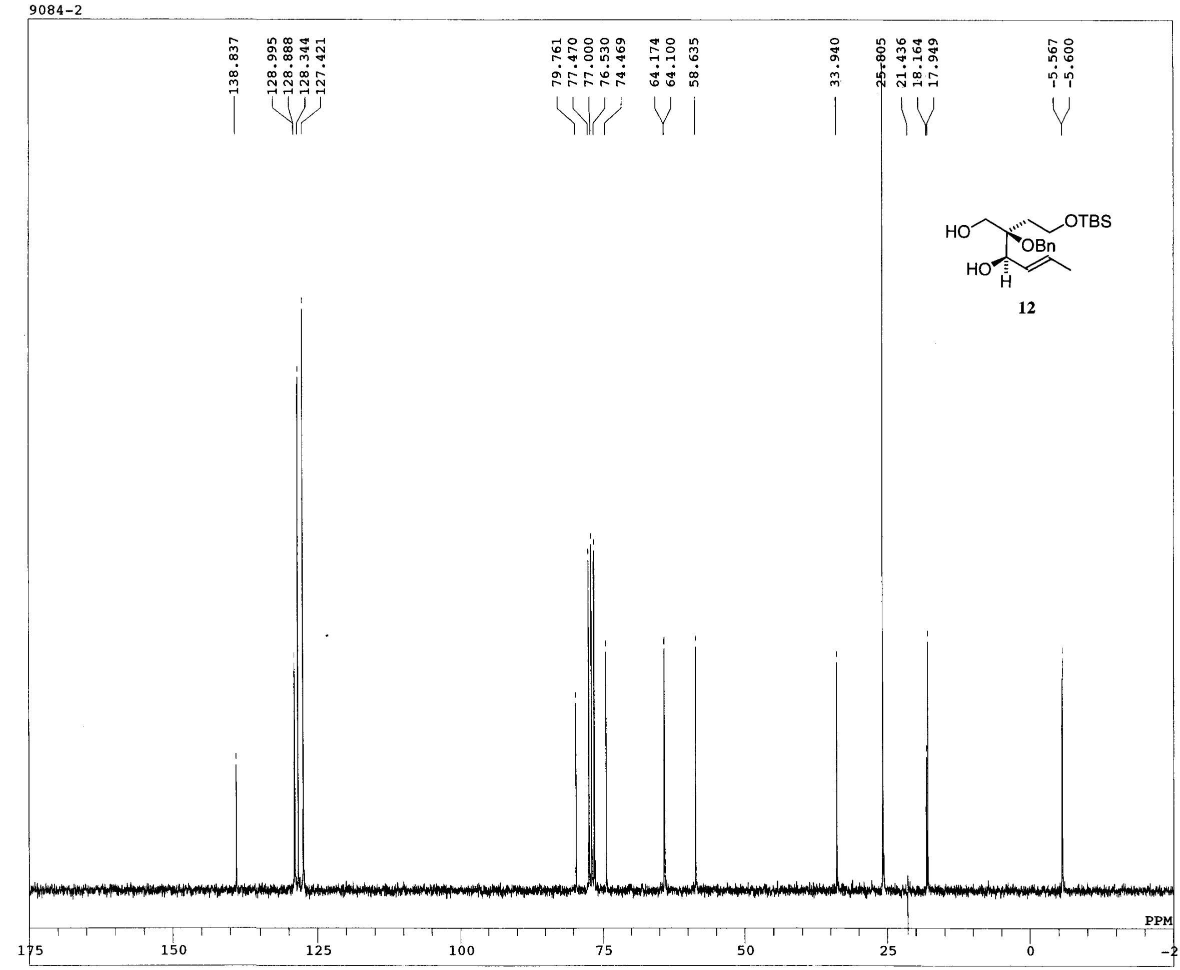


```
SPEED          12 Hz
CSPED          13 Hz
TEMP         80.0 c
CTEMP        22.4 c
TCOMP        1.00
SNO             1
HELEV       88.40
N2LEV      100.00
DATIM  Sat Aug 31 20:33:52 2002
PROBE  TH5
IRCODE        146
PRCODE       1412
DFILE  C:\WINNMR95\DATA\Auto2BCM_E1_FT.al
MENUF  BCM
SHMFL  TH5
COMNT  aldol up
SHMCM  th5/2%age/CDCl3/40mm/17JAN02
SLVNT  CDCL3
OBNUC  13C
OBFRQ       67.80 MHz
OBSET      135.00 KHz
OBFIN      5200.0 Hz
OBST2        0.00 KHz
OBFN2         0.0 Hz
OBPHS         0.0 deg
OBATN         511
IRNUC  1H
IRFRQ      270.05 MHz
IRSET      112.00 KHz
IRFIN      5800.0 Hz
IRST2       50.00 KHz
IRFN2      3700.0 Hz
IRPHS         0.0 deg
IRATN         511
IRRPW          40 us
IRRNS           0
TRNUC  1H
TRFRQ      270.05 MHz
TRSET      112.00 KHz
TRFIN      5800.0 Hz
TRST2        0.00 KHz
TRFN2         0.0 Hz
TRPHS         0.0 deg
TRATN         511
TRRPW          70 us
TRRNS           0
PW1           3.7 us
PW2          10.0 us
PW3          10.0 us
PW4           1.0 us
PW5           1.0 us
PI1         1.000 ms
PI2         1.000 ms
PI3          1.00 ms
PI4          1.00 ms
PI5          1.00 ms
LOOP1           1
LOOP2           1
LOOP3           1
LOOP4           1
LOOP5           1
PS1         100.0 us
```

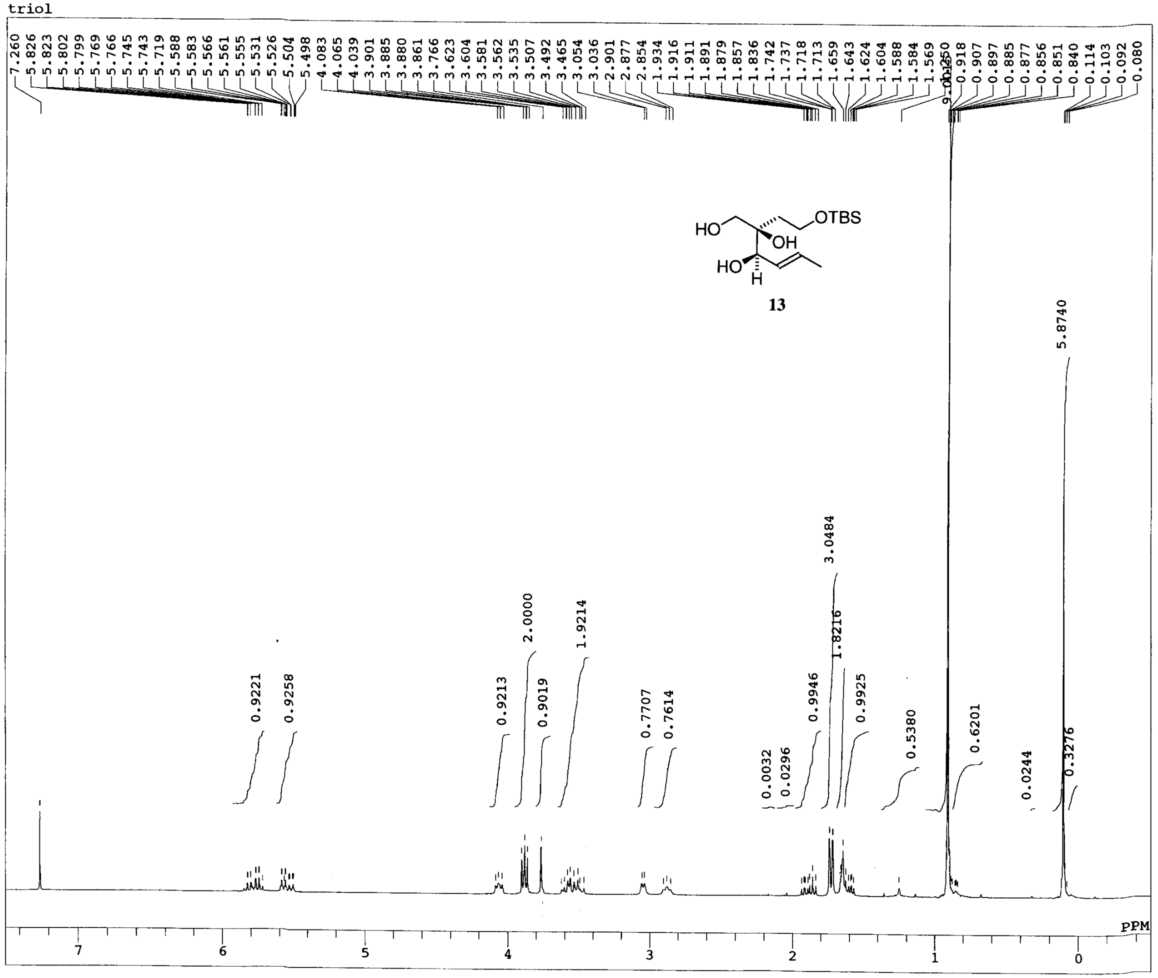


```
SPEED          12 Hz
CSPED          13 Hz
TEMP         80.0 c
CTEMP        21.6 c
TCOMP        1.00
SNO             1
HELEV       85.60
N2LEV      100.00
DATIM  Tue Sep 03 08:40:53 2002
PROBE  TH5
IRCODE        146
PRCODE       1412
DFILE  C:\WINNMR95\DATA\Auto1NON_E1_FT.al
MENUF  NON
SHMFL  TH5
COMNT  9084-2
SHMCM  th5/2%age/CDCl3/40mm/17JAN02
SLVNT  CDCL3
OBNUC  1H
OBFRQ      270.05 MHz
OBSET      112.00 KHz
OBFIN      5800.0 Hz
OBST2        0.00 KHz
OBFN2         0.0 Hz
OBPHS         0.0 deg
OBATN         511
IRNUC  1H
IRFRQ      270.05 MHz
IRSET      112.00 KHz
IRFIN      5800.0 Hz
IRST2       50.00 KHz
IRFN2      3700.0 Hz
IRPHS         0.0 deg
IRATN         511
IRRPW          50 us
IRRNS           0
TRNUC  1H
TRFRQ      270.05 MHz
TRSET      112.00 KHz
TRFIN      5800.0 Hz
TRST2        0.00 KHz
TRFN2         0.0 Hz
TRPHS         0.0 deg
TRATN         511
TRRPW          70 us
TRRNS           0
PW1           5.5 us
PW2          10.0 us
PW3          10.0 us
PW4           1.0 us
PW5           1.0 us
PI1         1.000 ms
PI2         1.000 ms
PI3          1.00 ms
PI4          1.00 ms
PI5          1.00 ms
LOOP1           1
LOOP2           1
LOOP3           1
LOOP4           1
LOOP5           1
PS1         100.0 us
```



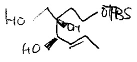


```
SPEED           12 Hz
CSPED           13 Hz
TEMP          80.0 c
CTEMP         21.8 c
TCOMP         1.00
SNO              1
HELEV        85.60
N2LEV       100.00
DATIM  Tue Sep 03 09:06:57 2002
PROBE  TH5
IRCODE         146
PRCODE        1412
DFILE  C:\WINNMR95\DATA\Auto2BCM_E1_FT.al
MENUF  BCM
SHMFL  TH5
COMNT  9084-2
SHMCM  th5/2%age/CDCl3/40mm/17JAN02
SLVNT  CDCL3
OBNUC  13C
OBFRQ        67.80 MHz
OBSET       135.00 KHz
OBFIN       5200.0 Hz
OBST2         0.00 KHz
OBFN2          0.0 Hz
OBPHS          0.0 deg
OBATN          511
IRNUC  1H
IRFRQ       270.05 MHz
IRSET       112.00 KHz
IRFIN       5800.0 Hz
IRST2        50.00 KHz
IRFN2       3700.0 Hz
IRPHS          0.0 deg
IRATN          511
IRRPW           40 us
IRRNS            0
TRNUC  1H
TRFRQ       270.05 MHz
TRSET       112.00 KHz
TRFIN       5800.0 Hz
TRST2         0.00 KHz
TRFN2          0.0 Hz
TRPHS          0.0 deg
TRATN          511
TRRPW           70 us
TRRNS            0
PW1            3.7 us
PW2           10.0 us
PW3           10.0 us
PW4            1.0 us
PW5            1.0 us
PI1          1.000 ms
PI2          1.000 ms
PI3           1.00 ms
PI4           1.00 ms
PI5           1.00 ms
LOOP1            1
LOOP2            1
LOOP3            1
LOOP4            1
LOOP5            1
PS1          100.0 us
```

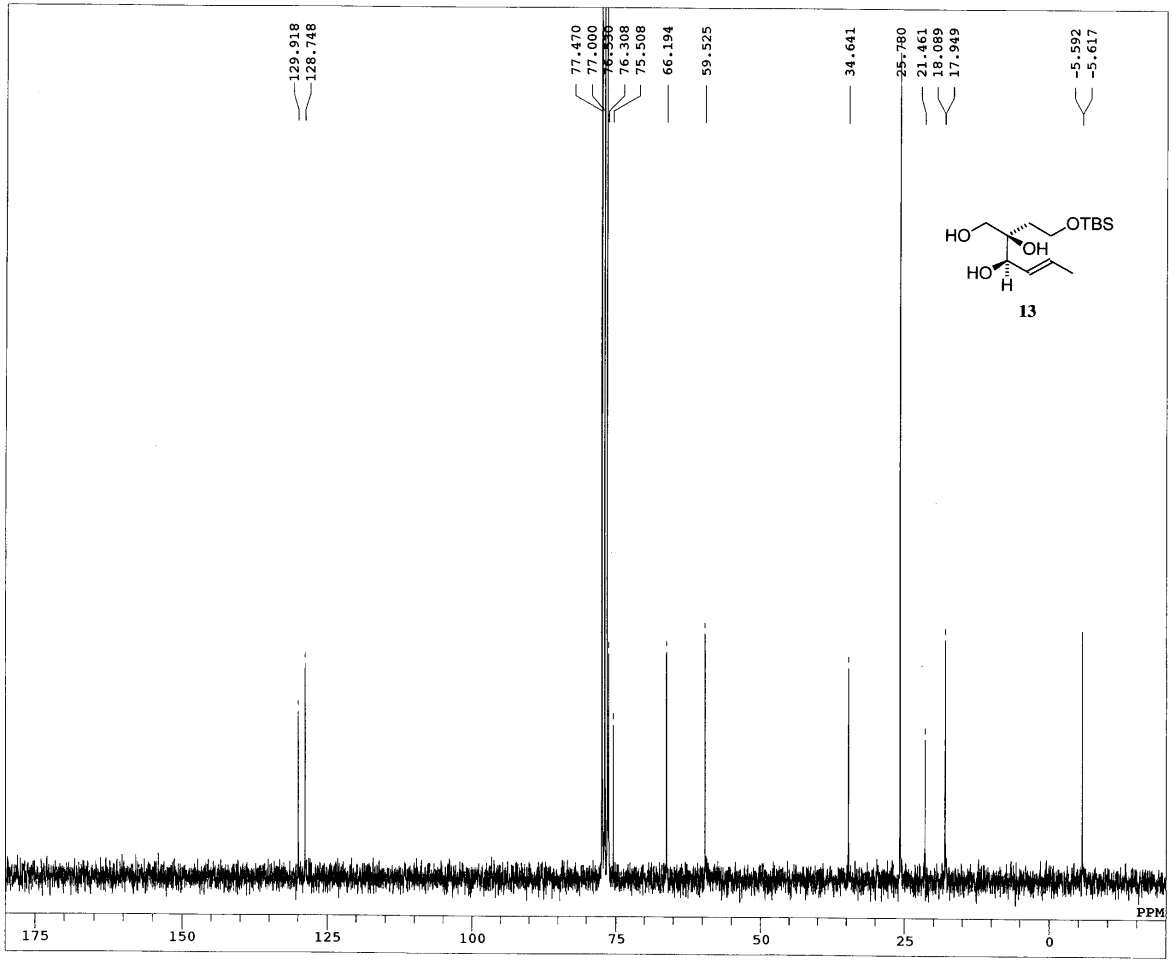


13

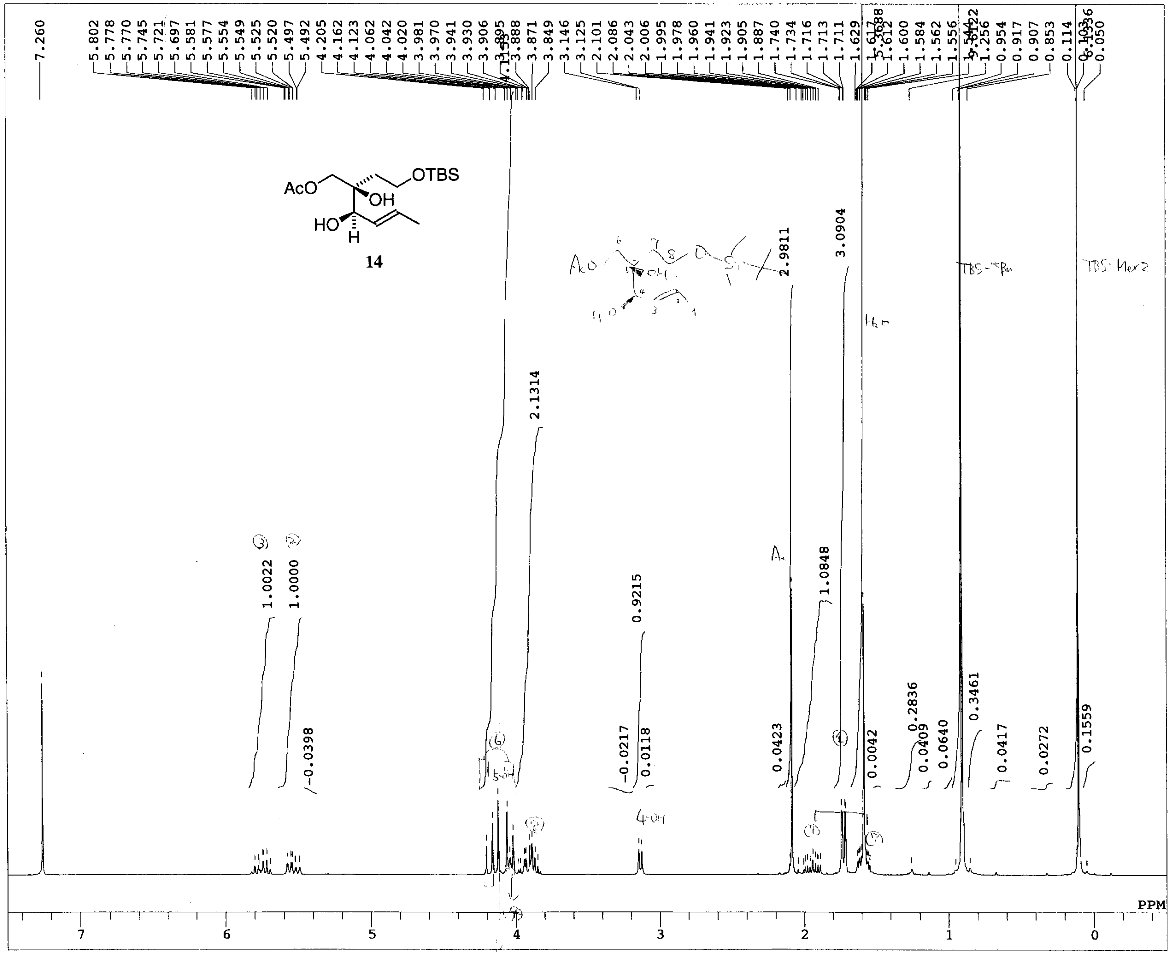
```
SPEED          12 Hz
CSPED          14 Hz
TEMP         80.0 c
CTEMP        21.9 c
TCOMP        1.00
SNO             1
HELEV       74.80
N2LEV      100.00
DATIM  Fri Sep 13 11:03:25 2002
PROBE  TH5
IRCODE        146
PRCODE       1412
DFILE  C:\WINNMR95\DATA\Auto1NON_E1_FT.al
MENUF  NON
SHMFL  TH5
COMNT  triol
SHMCM  th5/2%age/CDCl3/40mm/17JAN02
SLVNT  CDCL3
OBNUC  1H
OBFRQ      270.05 MHz
OBSET      112.00 KHz
OBFIN      5800.0 Hz
OBST2        0.00 KHz
OBFN2         0.0 Hz
OBPHS         0.0 deg
OBATN         511
IRNUC  1H
IRFRQ      270.05 MHz
IRSET      112.00 KHz
IRFIN      5800.0 Hz
IRST2       50.00 KHz
IRFN2      3700.0 Hz
IRPHS         0.0 deg
IRATN         511
IRRPW          50 us
IRRNS           0
TRNUC  1H
TRFRQ      270.05 MHz
TRSET      112.00 KHz
TRFIN      5800.0 Hz
TRST2        0.00 KHz
TRFN2         0.0 Hz
TRPHS         0.0 deg
TRATN         511
TRRPW          70 us
TRRNS           0
PW1           5.5 us
PW2          10.0 us
PW3          10.0 us
PW4           1.0 us
PW5           1.0 us
PI1         1.000 ms
PI2         1.000 ms
PI3          1.00 ms
PI4          1.00 ms
PI5          1.00 ms
LOOP1           1
LOOP2           1
LOOP3           1
LOOP4           1
LOOP5           1
PS1         100.0 us
```



triol

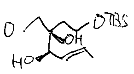


```
SPEED          12 Hz
CSPED          12 Hz
TEMP         80.0 c
CTEMP        21.9 c
TCOMP        1.00
SNO             1
HELEV       74.80
N2LEV      100.00
DATIM  Fri Sep 13 11:34:24 2002
PROBE  TH5
IRCODE        146
PRCODE       1412
DFILE  C:\WINNMR95\DATA\Auto1BCM_E1_PT.al
MENUF  BCM
SHMFL  TH5
COMNT  triol
SHMCM  th5/2%age/CDCl3/40mm/17JAN02
SLVNT  CDCL3
OBNUC  13C
OBFRQ       67.80 MHz
OBSET      135.00 KHz
OBFIN      5200.0 Hz
OBST2        0.00 KHz
OBFN2         0.0 Hz
OBPHS         0.0 deg
OBATN         511
IRNUC  1H
IRFRQ      270.05 MHz
IRSET      112.00 KHz
IRFIN      5800.0 Hz
IRST2       50.00 KHz
IRFN2      3700.0 Hz
IRPHS         0.0 deg
IRATN         511
IRRPW          40 us
IRRNS           0
TRNUC  1H
TRFRQ      270.05 MHz
TRSET      112.00 KHz
TRFIN      5800.0 Hz
TRST2        0.00 KHz
TRFN2         0.0 Hz
TRPHS         0.0 deg
TRATN         511
TRRPW          70 us
TRRNS           0
PW1           3.7 us
PW2          10.0 us
PW3          10.0 us
PW4           1.0 us
PW5           1.0 us
PI1         1.000 ms
PI2         1.000 ms
PI3          1.00 ms
PI4          1.00 ms
PI5          1.00 ms
LOOP1           1
LOOP2           1
LOOP3           1
LOOP4           1
LOOP5           1
PS1         100.0 us
```

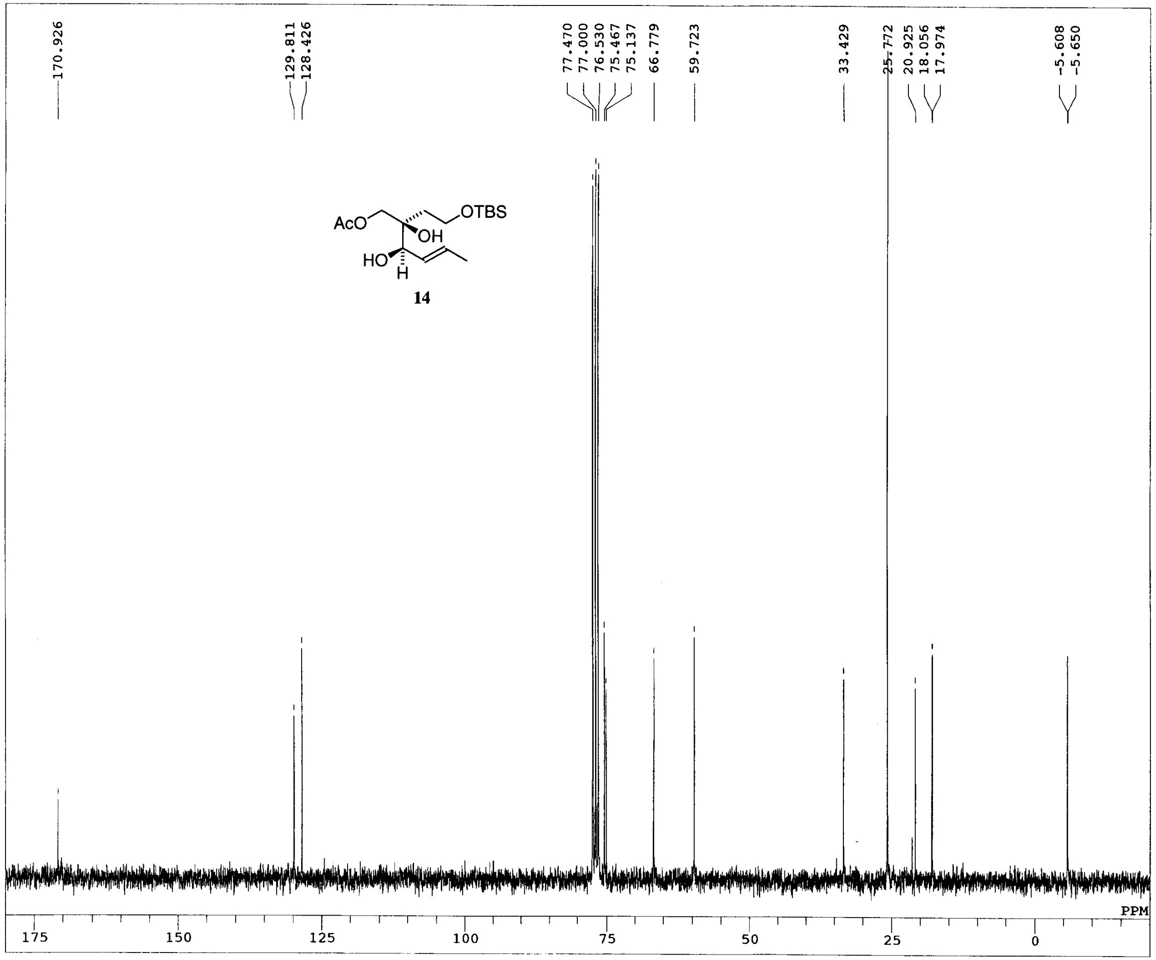


14

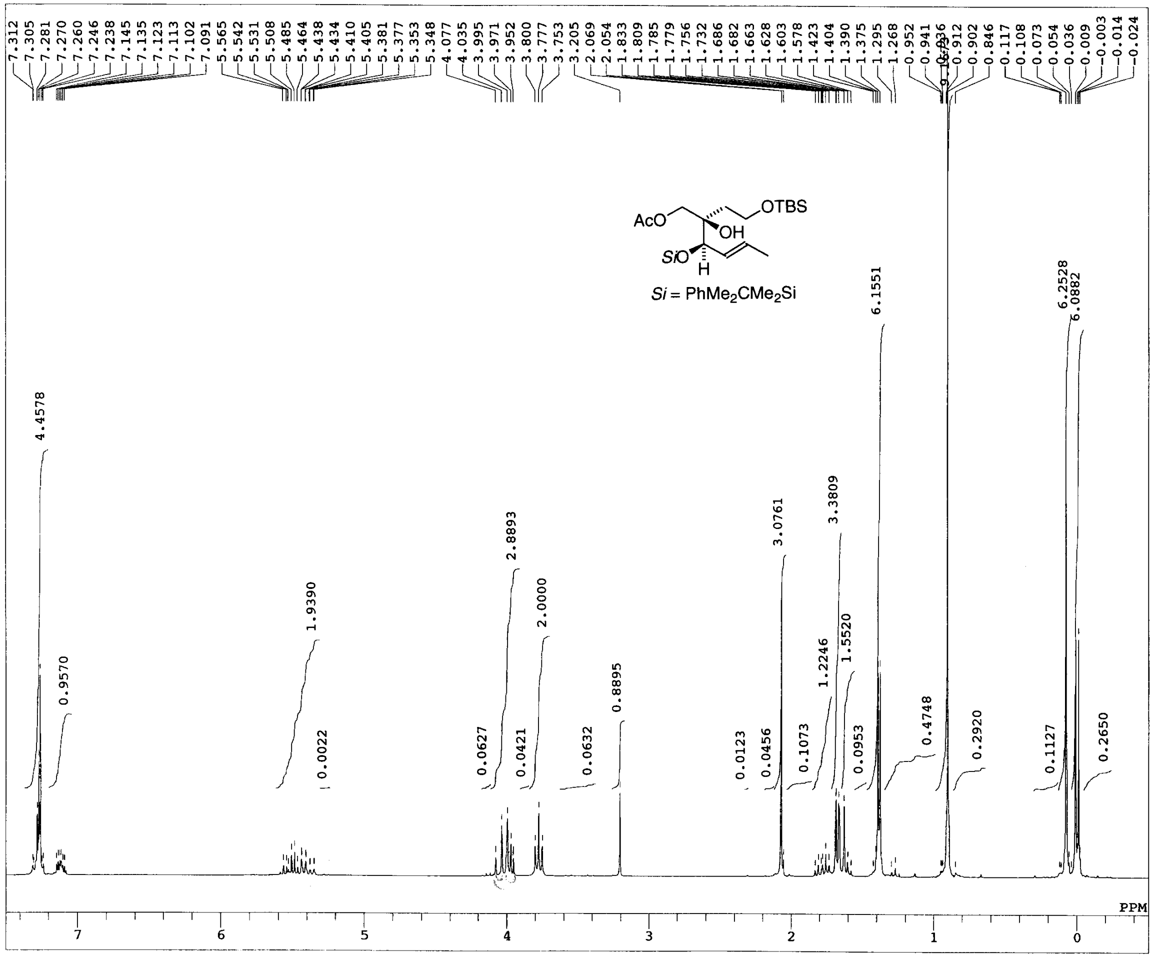
```
SPEED          12 Hz
CSPED          14 Hz
TEMP         80.0 c
CTEMP        22.1 c
TCOMP        1.00
SNO             1
HELEV       54.00
N2LEV       99.60
DATIM  Sun Sep 29 10:10:01 2002
PROBE  TH5
IRCODE        146
PRCODE       1412
DFILE  C:\WINNMR95\DATA\Auto1NON_E1_FT.al
MENUF  NON
SHMFL  TH5
COMNT
SHMCM  th5/2%age/CDC13/40mm/17JAN02
SLVNT  CDCL3
OBNUC  1H
OBFRQ      270.05 MHz
OBSET      112.00 KHz
OBFIN      5800.0 Hz
OBST2        0.00 KHz
OBFN2         0.0 Hz
OBPHS         0.0 deg
OBATN         511
IRNUC  1H
IRFRQ      270.05 MHz
IRSET      112.00 KHz
IRFIN      5800.0 Hz
IRST2       50.00 KHz
IRFN2      3700.0 Hz
IRPHS         0.0 deg
IRATN         511
IRRPW          50 us
IRRNS           0
TRNUC  1H
TRFRQ      270.05 MHz
TRSET      112.00 KHz
TRFIN      5800.0 Hz
TRST2        0.00 KHz
TRFN2         0.0 Hz
TRPHS         0.0 deg
TRATN         511
TRRPW          70 us
TRRNS           0
PW1           5.5 us
PW2          10.0 us
PW3          10.0 us
PW4           1.0 us
PW5           1.0 us
PI1         1.000 ms
PI2         1.000 ms
PI3          1.00 ms
PI4          1.00 ms
PI5          1.00 ms
LOOP1           1
LOOP2           1
LOOP3           1
LOOP4           1
LOOP5           1
PS1         100.0 us
```



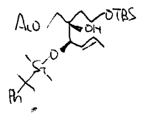
1-OAc

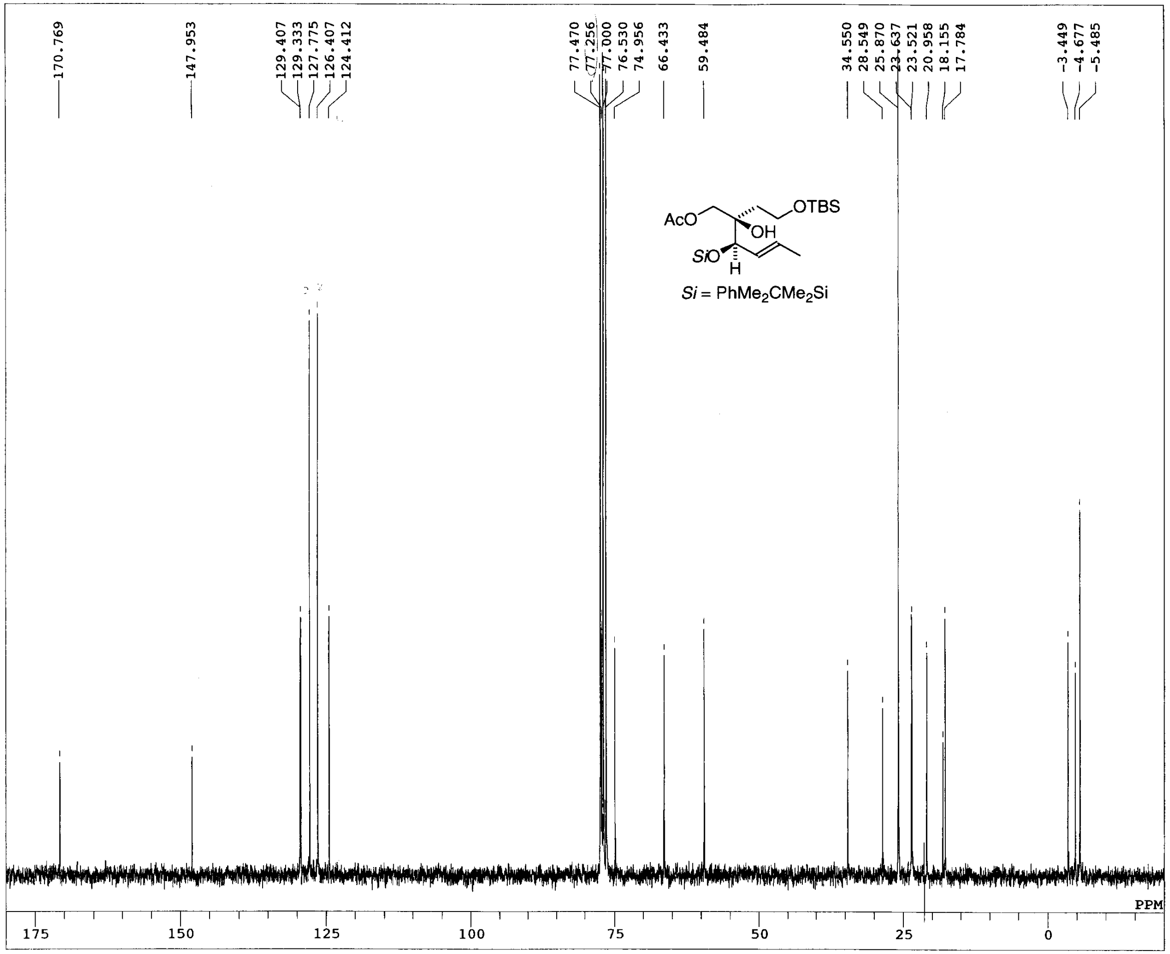


```
SPEED          12 Hz
CSPED          13 Hz
TEMP         80.0 c
CTEMP        22.6 c
TCOMP        1.00
SNO             1
HELEV       51.60
N2LEV       99.60
DATIM  Mon Sep 30 17:15:06 2002
PROBE  TH5
IRCODE        146
PRCODE       1412
DFILE  C:\WINNMR95\DATA\Auto1BCM_E1_FT.al
MENUF  BCM
SHMFL  TH5
COMNT  1-OAc
SHMCM  th5/2%age/CDCl3/40mm/17JAN02
SLVNT  CDCL3
OBNUC  13C
OBFRQ       67.80 MHz
OBSET      135.00 KHz
OBFIN      5200.0 Hz
OBST2        0.00 KHz
OBFN2         0.0 Hz
OBPHS         0.0 deg
OBATN         511
IRNUC  1H
IRFRQ      270.05 MHz
IRSET      112.00 KHz
IRFIN      5800.0 Hz
IRST2       50.00 KHz
IRFN2      3700.0 Hz
IRPHS         0.0 deg
IRATN         511
IRRPW          40 us
IRRNS           0
TRNUC  1H
TRFRQ      270.05 MHz
TRSET      112.00 KHz
TRFIN      5800.0 Hz
TRST2        0.00 KHz
TRFN2         0.0 Hz
TRPHS         0.0 deg
TRATN         511
TRRPW          70 us
TRRNS           0
PW1           3.7 us
PW2          10.0 us
PW3          10.0 us
PW4           1.0 us
PW5           1.0 us
PI1         1.000 ms
PI2         1.000 ms
PI3          1.00 ms
PI4          1.00 ms
PI5          1.00 ms
LOOP1           1
LOOP2           1
LOOP3           1
LOOP4           1
LOOP5           1
PS1         100.0 us
```

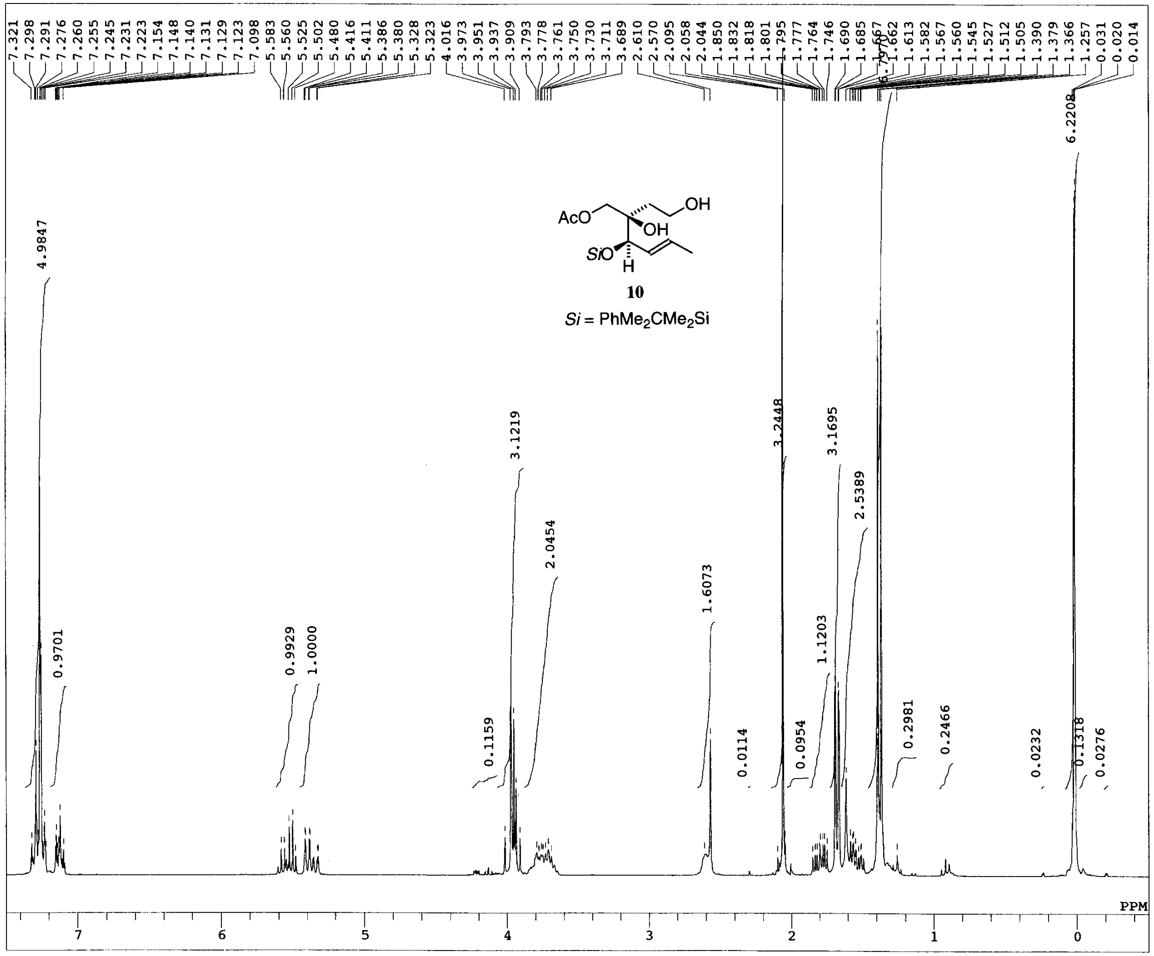


```
SPEED          12 Hz
CSPED          14 Hz
TEMP         80.0 c
CTEMP        22.5 c
TCOMP        1.00
SNO             1
HELEV       38.40
N2LEV       99.60
DATIM  Mon Oct 07 10:40:54 2002
PROBE  TH5
IRCODE        146
PRCODE       1412
DFILE  C:\WINNMR95\DATA\Auto1NON_E1_FT.al
MENUF  NON
SHMFL  TH5
COMNT
SHMCM  th5/2%age/CDCl3/40mm/17JAN02
SLVNT  CDCL3
OBNUC  1H
OBFRQ      270.05 MHz
OBSET      112.00 KHz
OBFIN      5800.0 Hz
OBST2        0.00 KHz
OBFN2         0.0 Hz
OBPHS         0.0 deg
OBATN         511
IRNUC  1H
IRFRQ      270.05 MHz
IRSET      112.00 KHz
IRFIN      5800.0 Hz
IRST2       50.00 KHz
IRFN2      3700.0 Hz
IRPHS         0.0 deg
IRATN         511
IRRPW          50 us
IRRNS           0
TRNUC  1H
TRFRQ      270.05 MHz
TRSET      112.00 KHz
TRFIN      5800.0 Hz
TRST2        0.00 KHz
TRFN2         0.0 Hz
TRPHS         0.0 deg
TRATN         511
TRRPW          70 us
TRRNS           0
PW1           5.5 us
PW2          10.0 us
PW3          10.0 us
PW4           1.0 us
PW5           1.0 us
PI1         1.000 ms
PI2         1.000 ms
PI3          1.00 ms
PI4          1.00 ms
PI5          1.00 ms
LOOP1           1
LOOP2           1
LOOP3           1
LOOP4           1
LOOP5           1
PS1         100.0 us
```





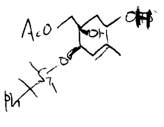
```
SPEED          12 Hz
CSPED          13 Hz
TEMP         80.0 c
CTEMP        21.5 c
TCOMP        1.00
SNO             1
HELEV       38.40
N2LEV       99.60
DATIM  Mon Oct 07 11:32:35 2002
PROBE  TH5
IRCODE        146
PRCODE       1412
DFILE  C:\WINNMR95\DATA\Auto2BCM_E1_FT.al
MENUF  BCM
SHMFL  TH5
COMNT
SHMCM  th5/2%age/CDCl3/40mm/17JAN02
SLVNT  CDCL3
OBNUC  13C
OBFRQ       67.80 MHz
OBSET      135.00 KHz
OBFIN      5200.0 Hz
OBST2        0.00 KHz
OBFN2         0.0 Hz
OBPHS         0.0 deg
OBATN         511
IRNUC  1H
IRFRQ      270.05 MHz
IRSET      112.00 KHz
IRFIN      5800.0 Hz
IRST2       50.00 KHz
IRFN2      3700.0 Hz
IRPHS         0.0 deg
IRATN         511
IRRPW          40 us
IRRNS           0
TRNUC  1H
TRFRQ      270.05 MHz
TRSET      112.00 KHz
TRFIN      5800.0 Hz
TRST2        0.00 KHz
TRFN2         0.0 Hz
TRPHS         0.0 deg
TRATN         511
TRRPW          70 us
TRRNS           0
PW1           3.7 us
PW2          10.0 us
PW3          10.0 us
PW4           1.0 us
PW5           1.0 us
PI1         1.000 ms
PI2         1.000 ms
PI3          1.00 ms
PI4          1.00 ms
PI5          1.00 ms
LOOP1           1
LOOP2           1
LOOP3           1
LOOP4           1
LOOP5           1
PS1         100.0 us
```

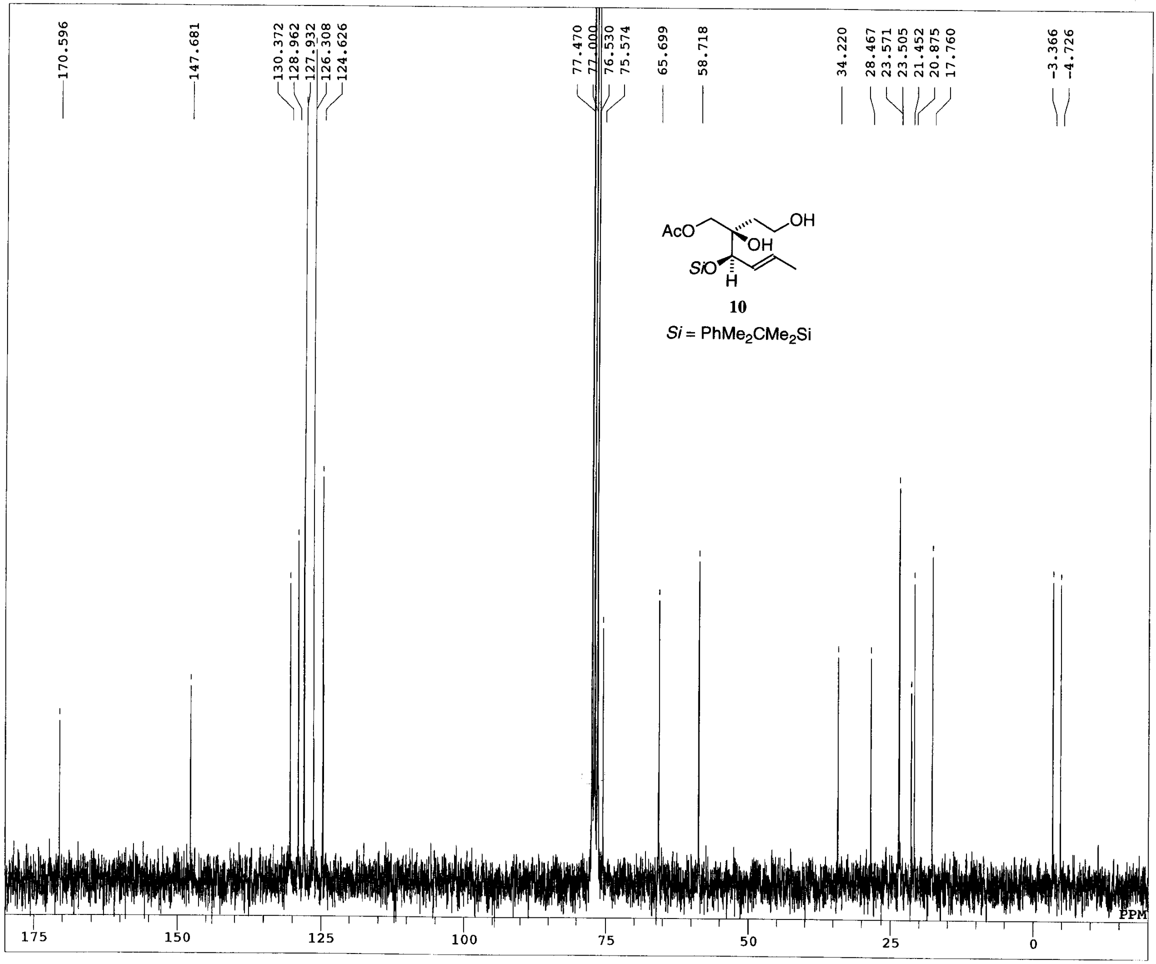


10

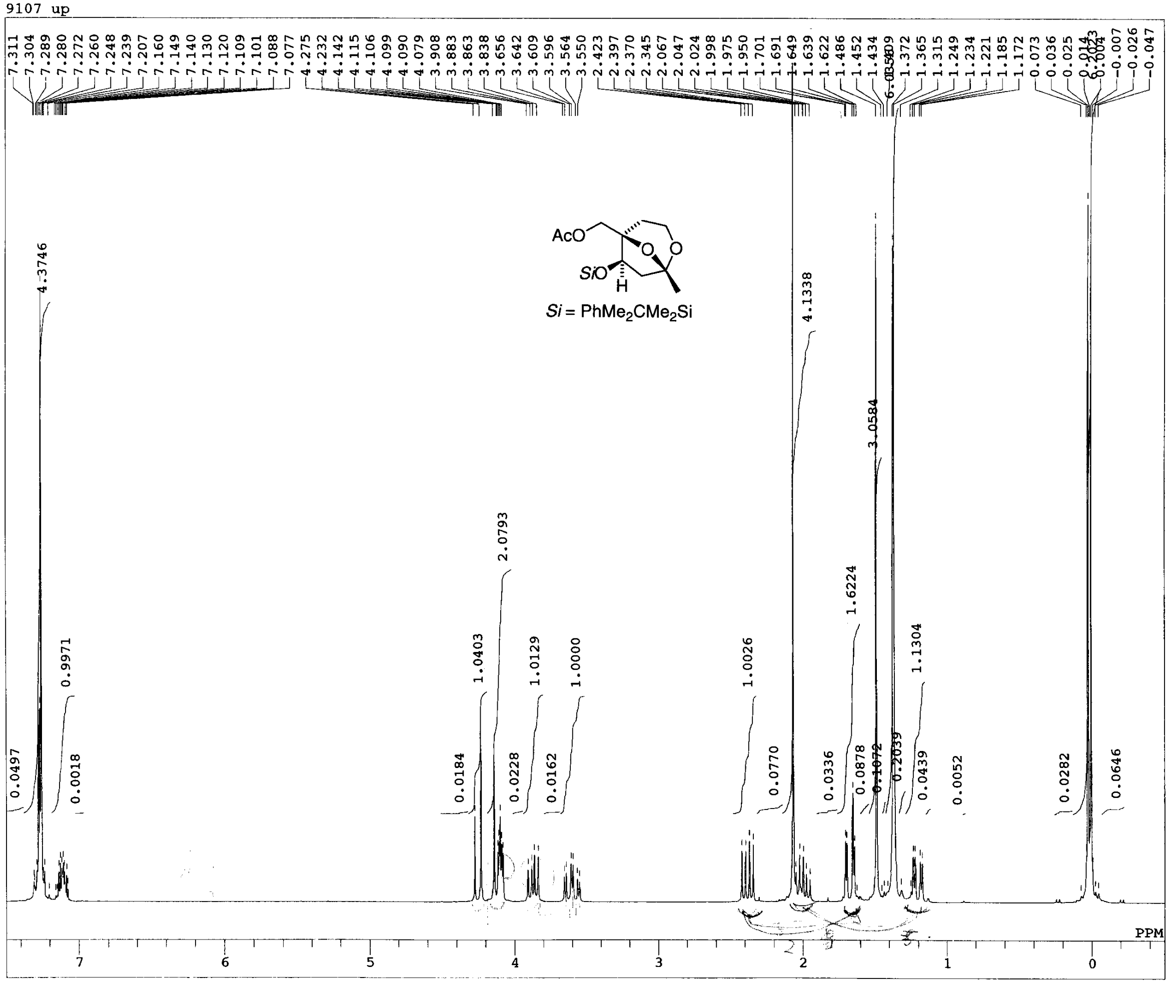
Si = $PhMe_2CMe_2Si$

```
SPEED          12 Hz
CSPED          14 Hz
TEMP         80.0 C
CTEMP        22.5 C
TCOMP        1.00
SNO             1
HELEV       35.60
N2LEV      100.00
DATIM  Wed Oct 09 10:22:32 2002
PROBE  TH5
IRCODE        146
PRCODE       1412
DFILE  C:\WINNMR95\DATA\Auto1NON_E1_FT.al
MENUF  NON
SHMFL  TH5
COMNT
SHMCM  th5/2%age/CDCl3/40mm/17JAN02
SLVNT  CDCL3
OBNUC  1H
OBFRQ      270.05 MHz
OBSET      112.00 KHz
OBFIN      5800.0 Hz
OBST2        0.00 KHz
OBFN2         0.0 Hz
OBPHS         0.0 deg
OBATN         511
IRNUC  1H
IRFRQ      270.05 MHz
IRSET      112.00 KHz
IRFIN      5800.0 Hz
IRST2       50.00 KHz
IRFN2      3700.0 Hz
IRPHS         0.0 deg
IRATN         511
IRRPW          50 us
IRRNS           0
TRNUC  1H
TRFRQ      270.05 MHz
TRSET      112.00 KHz
TRFIN      5800.0 Hz
TRST2        0.00 KHz
TRFN2         0.0 Hz
TRPHS         0.0 deg
TRATN         511
TRRPW          70 us
TRRNS           0
PW1           5.5 us
PW2          10.0 us
PW3          10.0 us
PW4           1.0 us
PW5           1.0 us
PI1         1.000 ms
PI2         1.000 ms
PI3          1.00 ms
PI4          1.00 ms
PI5          1.00 ms
LOOP1           1
LOOP2           1
LOOP3           1
LOOP4           1
LOOP5           1
PS1         100.0 us
```

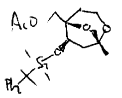


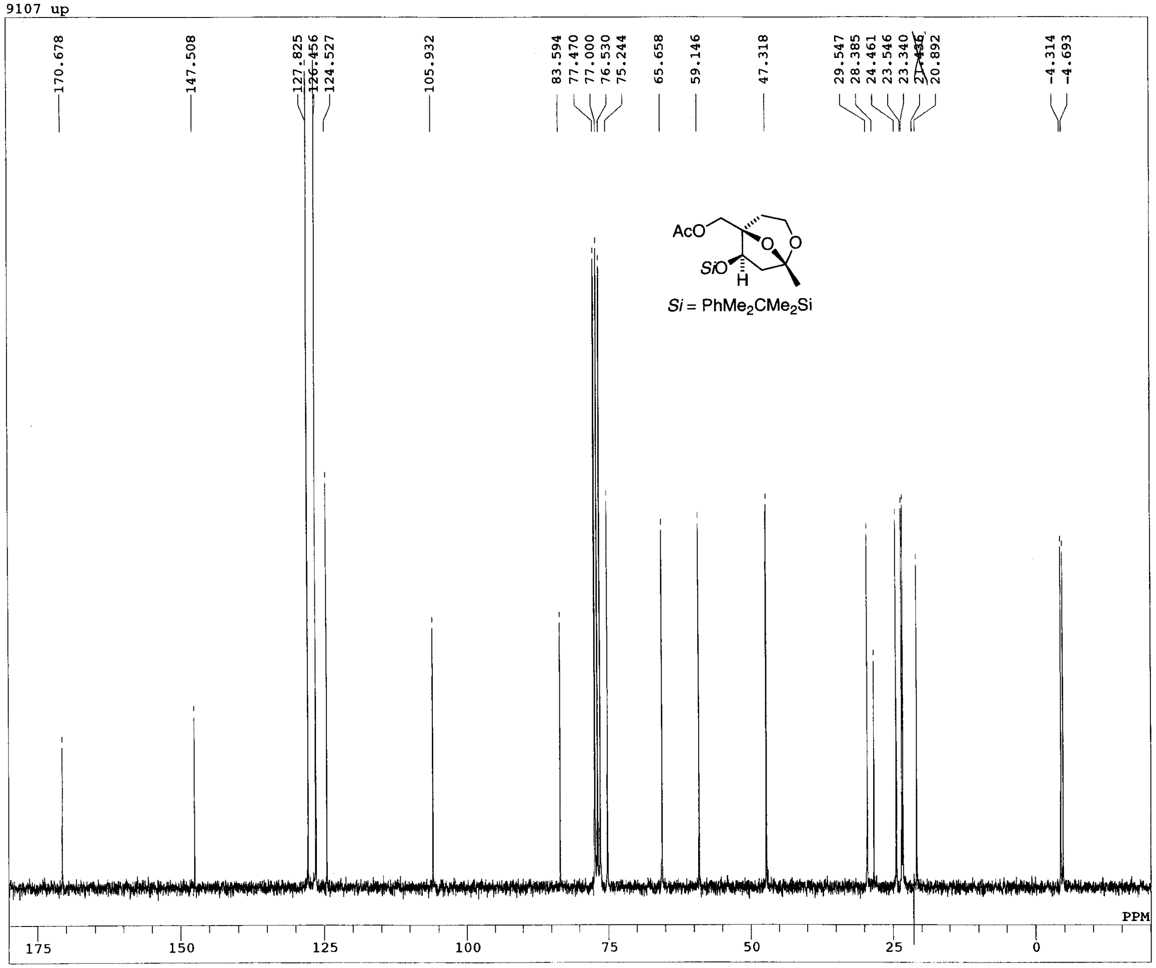


```
SPEED          12 Hz
CSPED          13 Hz
TEMP         80.0 c
CTEMP        22.3 c
TCOMP        1.00
SNO             1
HELEV       35.60
N2LEV      100.00
DATIM  Wed Oct 09 11:14:10 2002
PROBE  TH5
IRCODE        146
PRCODE       1412
DFILE  C:\WINNMR95\DATA\Auto2BCM_E1_FT.al
MENUF  BCM
SHMFL  TH5
COMNT
SHMCM  th5/2%age/CDCl3/40mm/17JAN02
SLVNT  CDCL3
OBNUC  13C
OBFRQ       67.80 MHz
OBSET      135.00 KHz
OBFIN      5200.0 Hz
OBST2        0.00 KHz
OBFN2         0.0 Hz
OBPHS         0.0 deg
OBATN         511
IRNUC  1H
IRFRQ      270.05 MHz
IRSET      112.00 KHz
IRFIN      5800.0 Hz
IRST2       50.00 KHz
IRFN2      3700.0 Hz
IRPHS         0.0 deg
IRATN         511
IRRPW          40 us
IRRNS           0
TRNUC  1H
TRFRQ      270.05 MHz
TRSET      112.00 KHz
TRFIN      5800.0 Hz
TRST2        0.00 KHz
TRFN2         0.0 Hz
TRPHS         0.0 deg
TRATN         511
TRRPW          70 us
TRRNS           0
PW1           3.7 us
PW2          10.0 us
PW3          10.0 us
PW4           1.0 us
PW5           1.0 us
PI1         1.000 ms
PI2         1.000 ms
PI3          1.00 ms
PI4          1.00 ms
PI5          1.00 ms
LOOP1           1
LOOP2           1
LOOP3           1
LOOP4           1
LOOP5           1
PS1         100.0 us
```

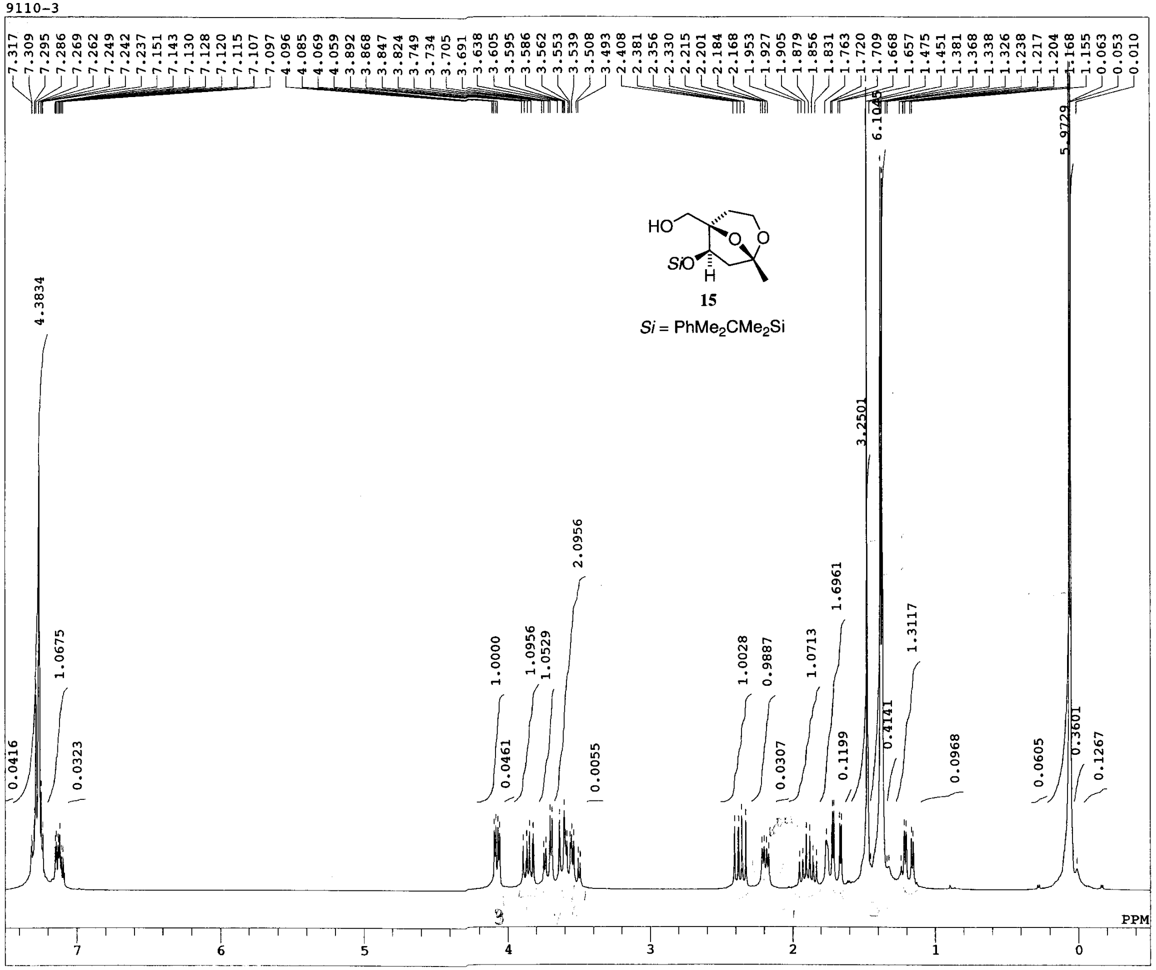


```
SPEED          12 Hz
CSPED          14 Hz
TEMP         80.0 c
CTEMP        22.4 c
TCOMP        1.00
SNO             1
HELEV       38.80
N2LEV       99.60
DATIM  Mon Oct 07 09:15:21 2002
PROBE  TH5
IRCODE        146
PRCODE       1412
DFILE  C:\WINNMR95\DATA\Auto1NON_E1_FT.al
MENUF  NON
SHMFL  TH5
COMNT  9107 up
SHMCM  th5/2%age/CDC13/40mm/17JAN02
SLVNT  CDCL3
OBNUC  1H
OBFRQ      270.05 MHz
OBSET      112.00 KHz
OBFIN      5800.0 Hz
OBST2        0.00 KHz
OBFN2         0.0 Hz
OBPHS         0.0 deg
OBATN         511
IRNUC  1H
IRFRQ      270.05 MHz
IRSET      112.00 KHz
IRFIN      5800.0 Hz
IRST2       50.00 KHz
IRFN2      3700.0 Hz
IRPHS         0.0 deg
IRATN         511
IRRPW          50 us
IRRNS           0
TRNUC  1H
TRFRQ      270.05 MHz
TRSET      112.00 KHz
TRFIN      5800.0 Hz
TRST2        0.00 KHz
TRFN2         0.0 Hz
TRPHS         0.0 deg
TRATN         511
TRRPW          70 us
TRRNS           0
PW1           5.5 us
PW2          10.0 us
PW3          10.0 us
PW4           1.0 us
PW5           1.0 us
PI1         1.000 ms
PI2         1.000 ms
PI3          1.00 ms
PI4          1.00 ms
PI5          1.00 ms
LOOP1           1
LOOP2           1
LOOP3           1
LOOP4           1
LOOP5           1
PS1         100.0 us
```

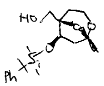


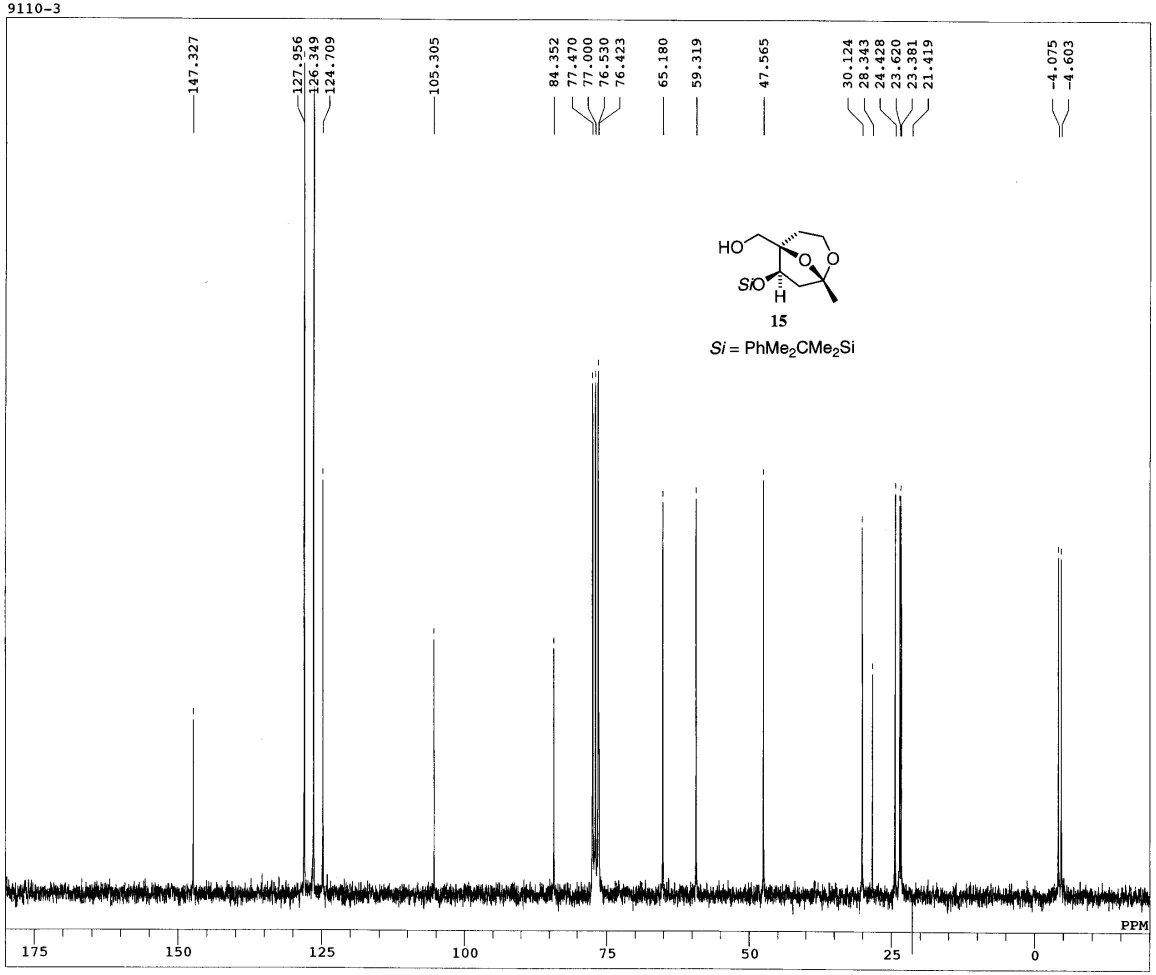


```
SPEED          12 Hz
CSPED          14 Hz
TEMP         80.0 c
CTEMP        21.5 c
TCOMP        1.00
SNO             1
HELEV       38.40
N2LEV       99.60
DATIM   Mon Oct 07 10:07:02 2002
PROBE   TH5
IRCODE        146
PRCODE       1412
DFILE   C:\WINNMR95\DATA\Auto2BCM_E1_FT.al
MENUF   BCM
SHMFL   TH5
COMNT   9107 up
SHMCM   th5/2%age/CDCl3/40mm/17JAN02
SLVNT   CDCL3
OBNUC   13C
OBFRQ       67.80 MHz
OBSET      135.00 KHz
OBFIN      5200.0 Hz
OBST2        0.00 KHz
OBFN2         0.0 Hz
OBPHS         0.0 deg
OBATN         511
IRNUC   1H
IRFRQ      270.05 MHz
IRSET      112.00 KHz
IRFIN      5800.0 Hz
IRST2       50.00 KHz
IRFN2      3700.0 Hz
IRPHS         0.0 deg
IRATN         511
IRRPW          40 us
IRRNS           0
TRNUC   1H
TRFRQ      270.05 MHz
TRSET      112.00 KHz
TRFIN      5800.0 Hz
TRST2        0.00 KHz
TRFN2         0.0 Hz
TRPHS         0.0 deg
TRATN         511
TRRPW          70 us
TRRNS           0
PW1           3.7 us
PW2          10.0 us
PW3          10.0 us
PW4           1.0 us
PW5           1.0 us
PI1         1.000 ms
PI2         1.000 ms
PI3          1.00 ms
PI4          1.00 ms
PI5          1.00 ms
LOOP1           1
LOOP2           1
LOOP3           1
LOOP4           1
LOOP5           1
PS1         100.0 us
```

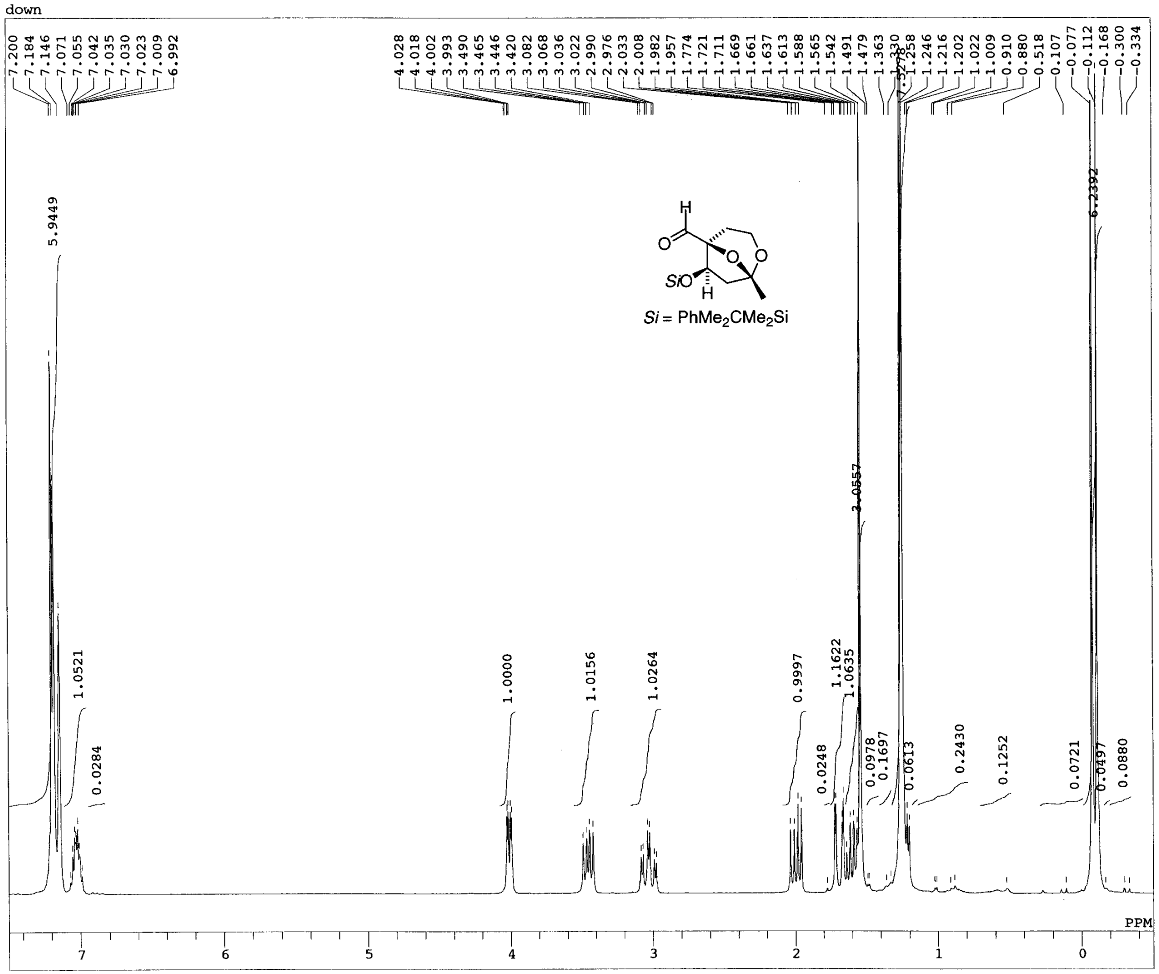


```
SPEED          12 Hz
CSPED          12 Hz
TEMP         80.0 c
CTEMP        21.6 c
TCOMP        1.00
SNO             1
HELEV       37.20
N2LEV      100.00
DATIM  Tue Oct 08 09:33:04 2002
PROBE  TH5
IRCODE        146
PRCODE       1412
DFILE  C:\WINNMR95\DATA\Auto1NON_E1_FT.al
MENUF  NON
SHMFL  TH5
COMNT  9110-3
SHMCM  th5/2%age/CDCl3/40mm/17JAN02
SLVNT  CDCL3
OBNUC  1H
OBFRQ      270.05 MHz
OBSET      112.00 KHz
OBFIN      5800.0 Hz
OBST2        0.00 KHz
OBFN2         0.0 Hz
OBPHS         0.0 deg
OBATN         511
IRNUC  1H
IRFRQ      270.05 MHz
IRSET      112.00 KHz
IRFIN      5800.0 Hz
IRST2       50.00 KHz
IRFN2      3700.0 Hz
IRPHS         0.0 deg
IRATN         511
IRRPW          50 us
IRRNS           0
TRNUC  1H
TRFRQ      270.05 MHz
TRSET      112.00 KHz
TRFIN      5800.0 Hz
TRST2        0.00 KHz
TRFN2         0.0 Hz
TRPHS         0.0 deg
TRATN         511
TRRPW          70 us
TRRNS           0
PW1           5.5 us
PW2          10.0 us
PW3          10.0 us
PW4           1.0 us
PW5           1.0 us
PI1         1.000 ms
PI2         1.000 ms
PI3          1.00 ms
PI4          1.00 ms
PI5          1.00 ms
LOOP1           1
LOOP2           1
LOOP3           1
LOOP4           1
LOOP5           1
PS1         100.0 us
```

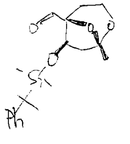


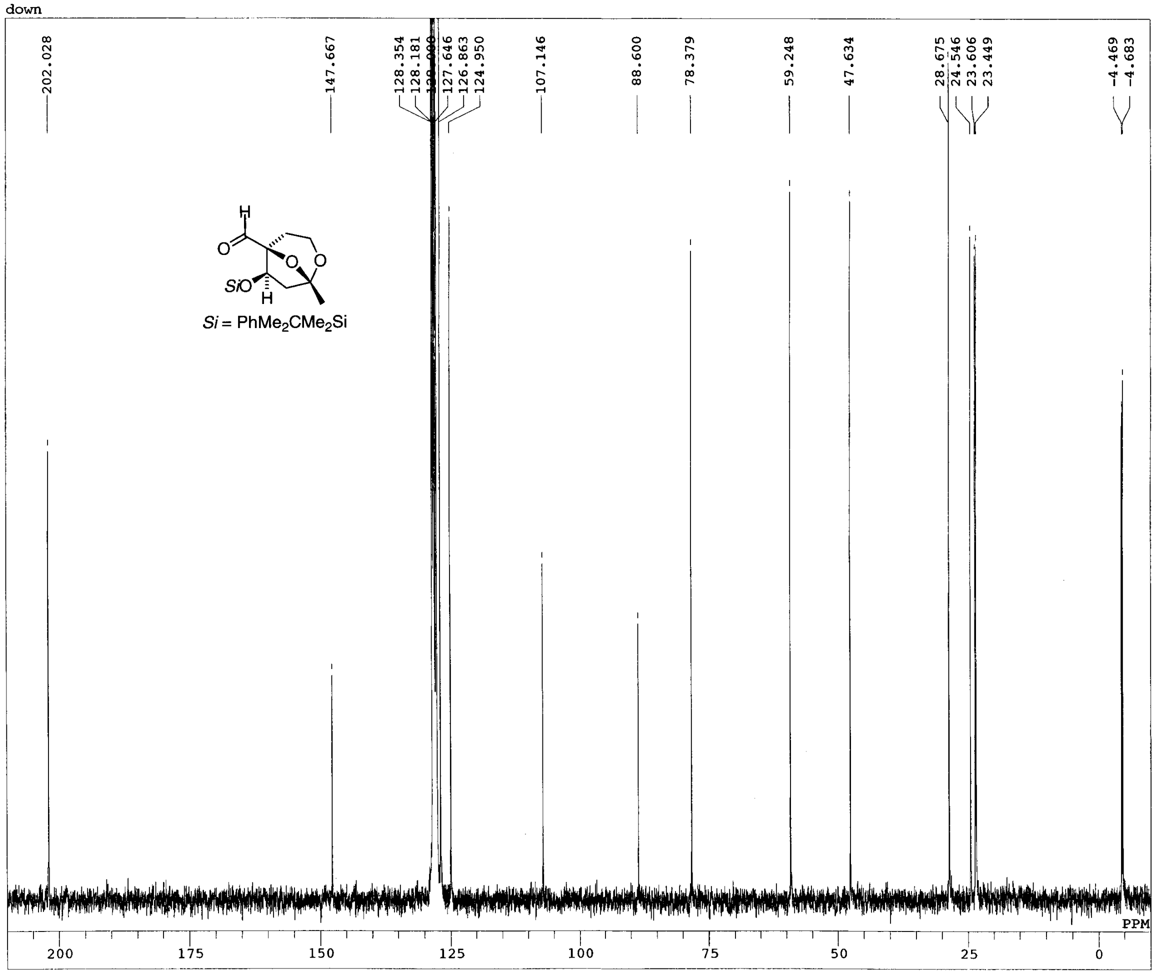


```
SPEED          12 Hz
CSPED          12 Hz
TEMP         80.0 c
CTEMP        22.2 c
TCOMP        1.00
SNO             1
HELEV       37.20
N2LEV      100.00
DATIM  Tue Oct 08 09:59:09 2002
PROBE  TH5
IRCODE        146
PRCODE       1412
DFILE  C:\WINNMR95\DATA\Auto2BCM_E1_FT.al
MENUF  BCM
SHMFL  TH5
COMNT  9110-3
SHMCM  th5/2%age/CDCl3/40mm/17JAN02
SLVNT  CDCL3
OBNUC  13C
OBFRQ       67.80 MHz
OBSET      135.00 KHz
OBFIN      5200.0 Hz
OBST2        0.00 KHz
OBFN2         0.0 Hz
OBPHS         0.0 deg
OBATN         511
IRNUC  1H
IRFRQ      270.05 MHz
IRSET      112.00 KHz
IRFIN      5800.0 Hz
IRST2       50.00 KHz
IRFN2      3700.0 Hz
IRPHS         0.0 deg
IRATN         511
IRRPW          40 us
IRRNS           0
TRNUC  1H
TRFRQ      270.05 MHz
TRSET      112.00 KHz
TRFIN      5800.0 Hz
TRST2        0.00 KHz
TRFN2         0.0 Hz
TRPHS         0.0 deg
TRATN         511
TRRPW          70 us
TRRNS           0
PW1           3.7 us
PW2          10.0 us
PW3          10.0 us
PW4           1.0 us
PW5           1.0 us
PI1         1.000 ms
PI2         1.000 ms
PI3          1.00 ms
PI4          1.00 ms
PI5          1.00 ms
LOOP1           1
LOOP2           1
LOOP3           1
LOOP4           1
LOOP5           1
PS1         100.0 us
```

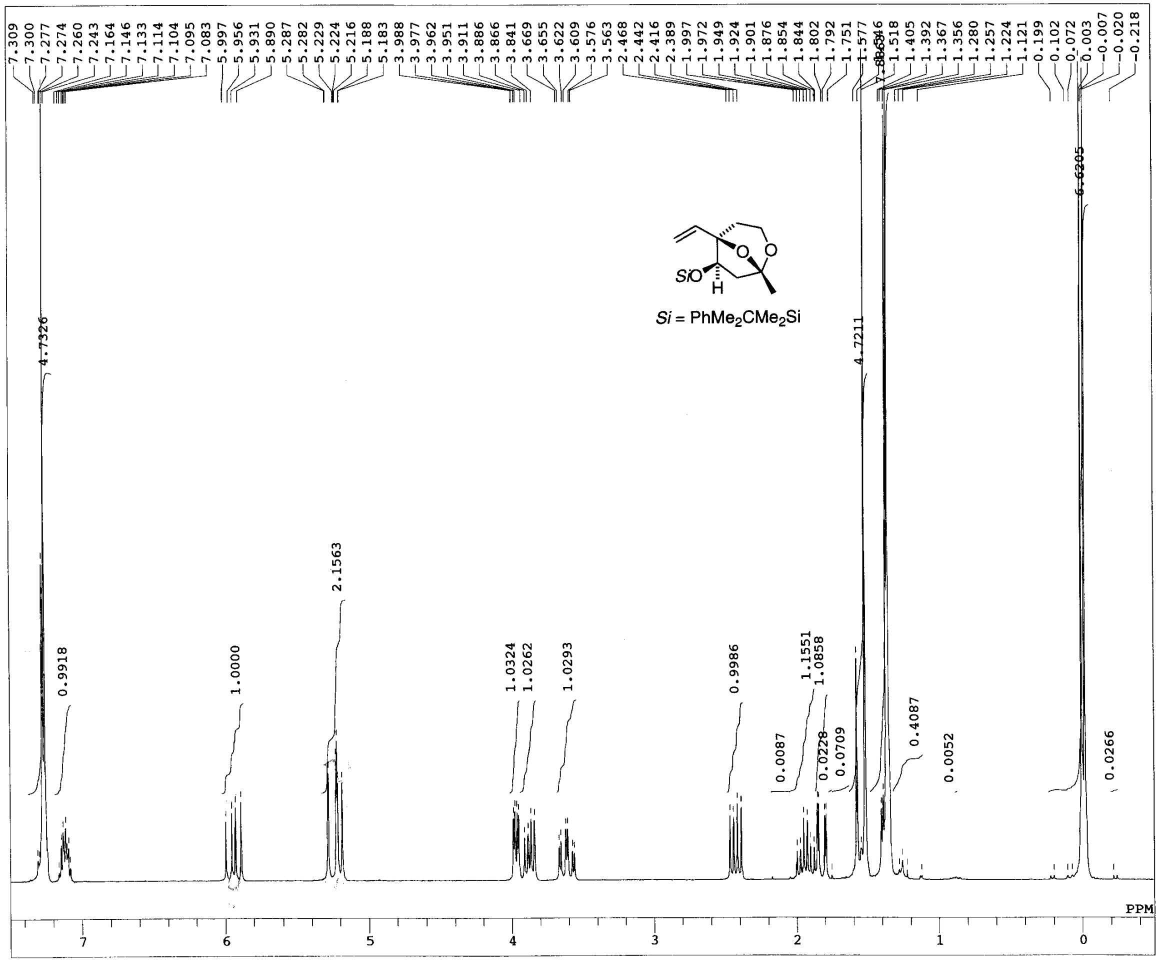


```
SPEED          12 Hz
CSPED          14 Hz
TEMP         80.0 c
CTEMP        23.4 c
TCOMP        1.00
SNO             1
HELEV       85.60
N2LEV       99.60
DATIM  Mon Oct 28 18:55:25 2002
PROBE  TH5
IRCODE        146
PRCODE       1412
DFILE  C:\WINNMR95\DATA\Auto1NON_E1_FT.al
MENUF  NON
SHMFL  TH5
COMNT  down
SHMCM  th5/2%age/CDCl3/40mm/17JAN02
SLVNT  C6D6
OBNUC  1H
OBFRQ      270.05 MHz
OBSET      112.00 KHz
OBFIN      5800.0 Hz
OBST2        0.00 KHz
OBFN2         0.0 Hz
OBPHS         0.0 deg
OBATN         511
IRNUC  1H
IRFRQ      270.05 MHz
IRSET      112.00 KHz
IRFIN      5800.0 Hz
IRST2       50.00 KHz
IRFN2      3700.0 Hz
IRPHS         0.0 deg
IRATN         511
IRRPW          50 us
IRRNS           0
TRNUC  1H
TRFRQ      270.05 MHz
TRSET      112.00 KHz
TRFIN      5800.0 Hz
TRST2        0.00 KHz
TRFN2         0.0 Hz
TRPHS         0.0 deg
TRATN         511
TRRPW          70 us
TRRNS           0
PW1           5.5 us
PW2          10.0 us
PW3          10.0 us
PW4           1.0 us
PW5           1.0 us
PI1         1.000 ms
PI2         1.000 ms
PI3          1.00 ms
PI4          1.00 ms
PI5          1.00 ms
LOOP1           1
LOOP2           1
LOOP3           1
LOOP4           1
LOOP5           1
PS1         100.0 us
```

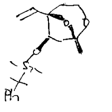


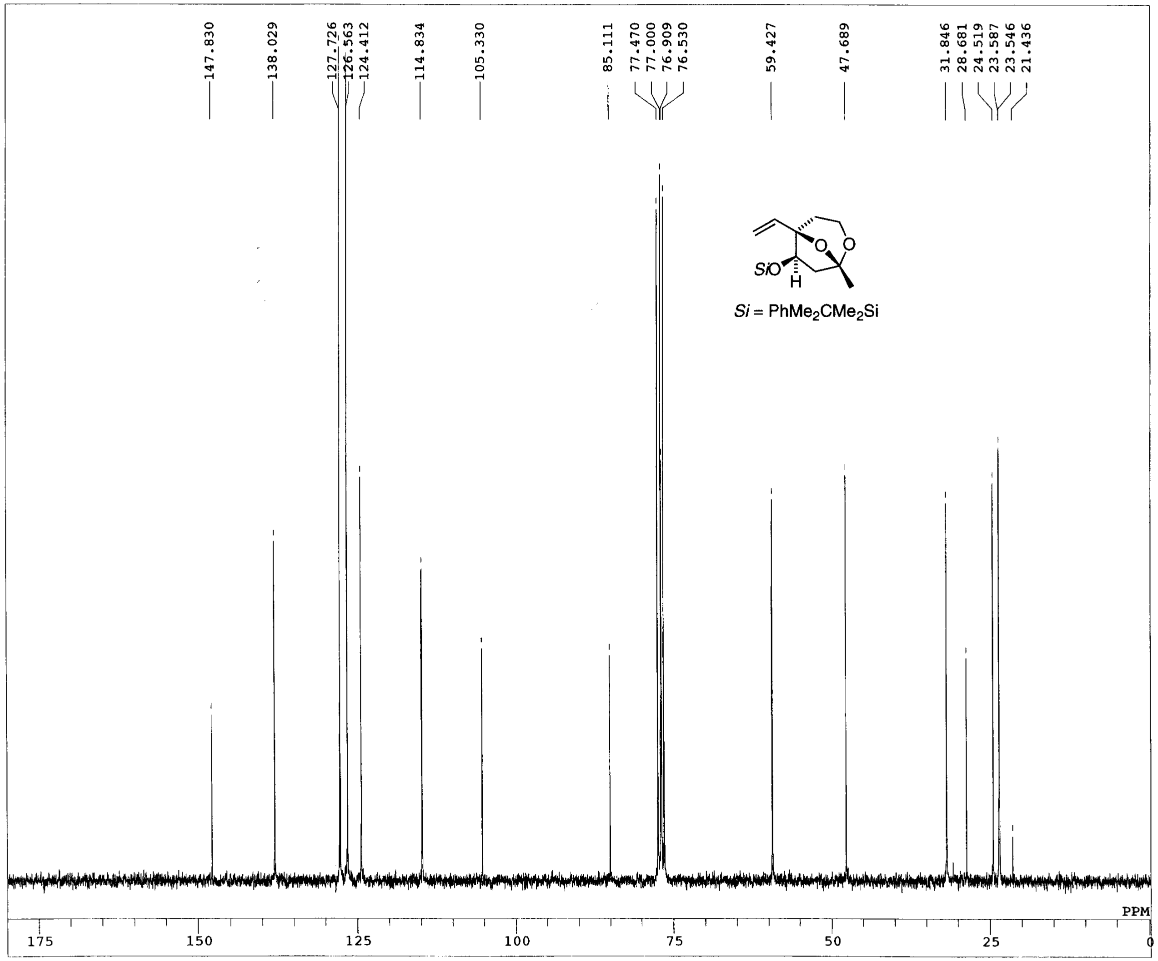


```
SPEED           12 Hz
CSPED           13 Hz
TEMP          80.0 c
CTEMP         22.9 c
TCOMP         1.00
SNO              1
HELEV        85.60
N2LEV       100.00
DATIM  Mon Oct 28 19:47:07 2002
PROBE  TH5
IRCODE         146
PRCODE        1412
DFILE  C:\WINNMR95\DATA\Auto2BCM_E1_FT.al
MENUF  BCM
SHMFL  TH5
COMNT  down
SHMCM  th5/2%age/CDCl3/40mm/17JAN02
SLVNT  C6D6
OBNUC  13C
OBFRQ        67.80 MHz
OBSET       135.00 KHz
OBFIN       5200.0 Hz
OBST2         0.00 KHz
OBFN2          0.0 Hz
OBPHS          0.0 deg
OBATN          511
IRNUC  1H
IRFRQ       270.05 MHz
IRSET       112.00 KHz
IRFIN       5800.0 Hz
IRST2        50.00 KHz
IRFN2       3700.0 Hz
IRPHS          0.0 deg
IRATN          511
IRRPW           40 us
IRRNS            0
TRNUC  1H
TRFRQ       270.05 MHz
TRSET       112.00 KHz
TRFIN       5800.0 Hz
TRST2         0.00 KHz
TRFN2          0.0 Hz
TRPHS          0.0 deg
TRATN          511
TRRPW           70 us
TRRNS            0
PW1            3.7 us
PW2           10.0 us
PW3           10.0 us
PW4            1.0 us
PW5            1.0 us
PI1          1.000 ms
PI2          1.000 ms
PI3           1.00 ms
PI4           1.00 ms
PI5           1.00 ms
LOOP1            1
LOOP2            1
LOOP3            1
LOOP4            1
LOOP5            1
PS1          100.0 us
```

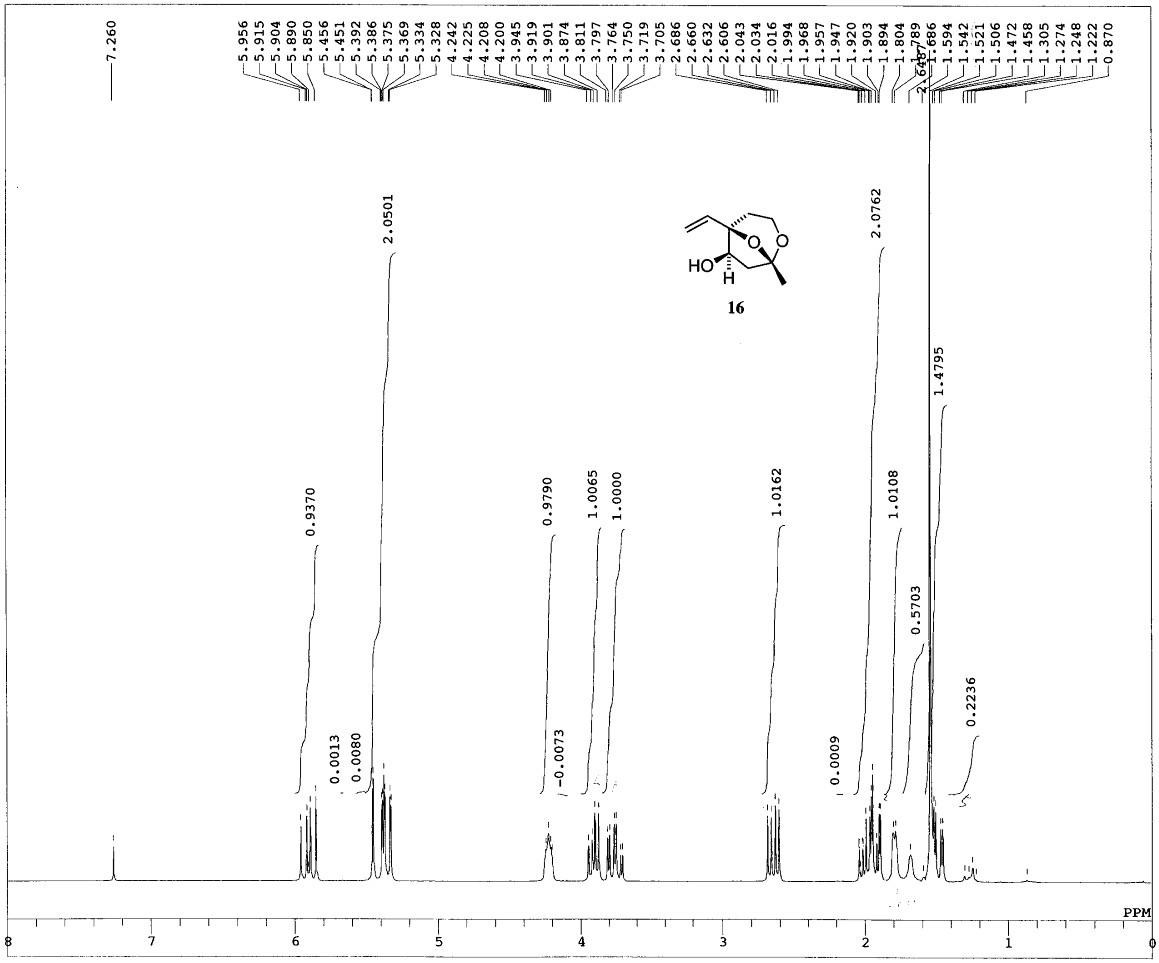


```
SPEED          12 Hz
CSPED          14 Hz
TEMP         80.0 c
CTEMP        23.4 c
TCOMP        1.00
SNO             1
HELEV       88.00
N2LEV       99.60
DATIM  Sat Oct 26 20:36:31 2002
PROBE  TH5
IRCODE        146
PRCODE       1412
DFILE  C:\WINNMR95\DATA\Auto1NON_E1_FT.al
MENUF  NON
SHMFL  TH5
COMNT
SHMCM  th5/2%age/CDCl3/40mm/17JAN02
SLVNT  CDCL3
OBNUC  1H
OBFRQ      270.05 MHz
OBSET      112.00 KHz
OBFIN      5800.0 Hz
OBST2        0.00 KHz
OBFN2         0.0 Hz
OBPHS         0.0 deg
OBATN         511
IRNUC  1H
IRFRQ      270.05 MHz
IRSET      112.00 KHz
IRFIN      5800.0 Hz
IRST2       50.00 KHz
IRFN2      3700.0 Hz
IRPHS         0.0 deg
IRATN         511
IRRPW          50 us
IRRNS           0
TRNUC  1H
TRFRQ      270.05 MHz
TRSET      112.00 KHz
TRFIN      5800.0 Hz
TRST2        0.00 KHz
TRFN2         0.0 Hz
TRPHS         0.0 deg
TRATN         511
TRRPW          70 us
TRRNS           0
PW1           5.5 us
PW2          10.0 us
PW3          10.0 us
PW4           1.0 us
PW5           1.0 us
PI1         1.000 ms
PI2         1.000 ms
PI3          1.00 ms
PI4          1.00 ms
PI5          1.00 ms
LOOP1           1
LOOP2           1
LOOP3           1
LOOP4           1
LOOP5           1
PS1         100.0 us
```

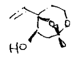


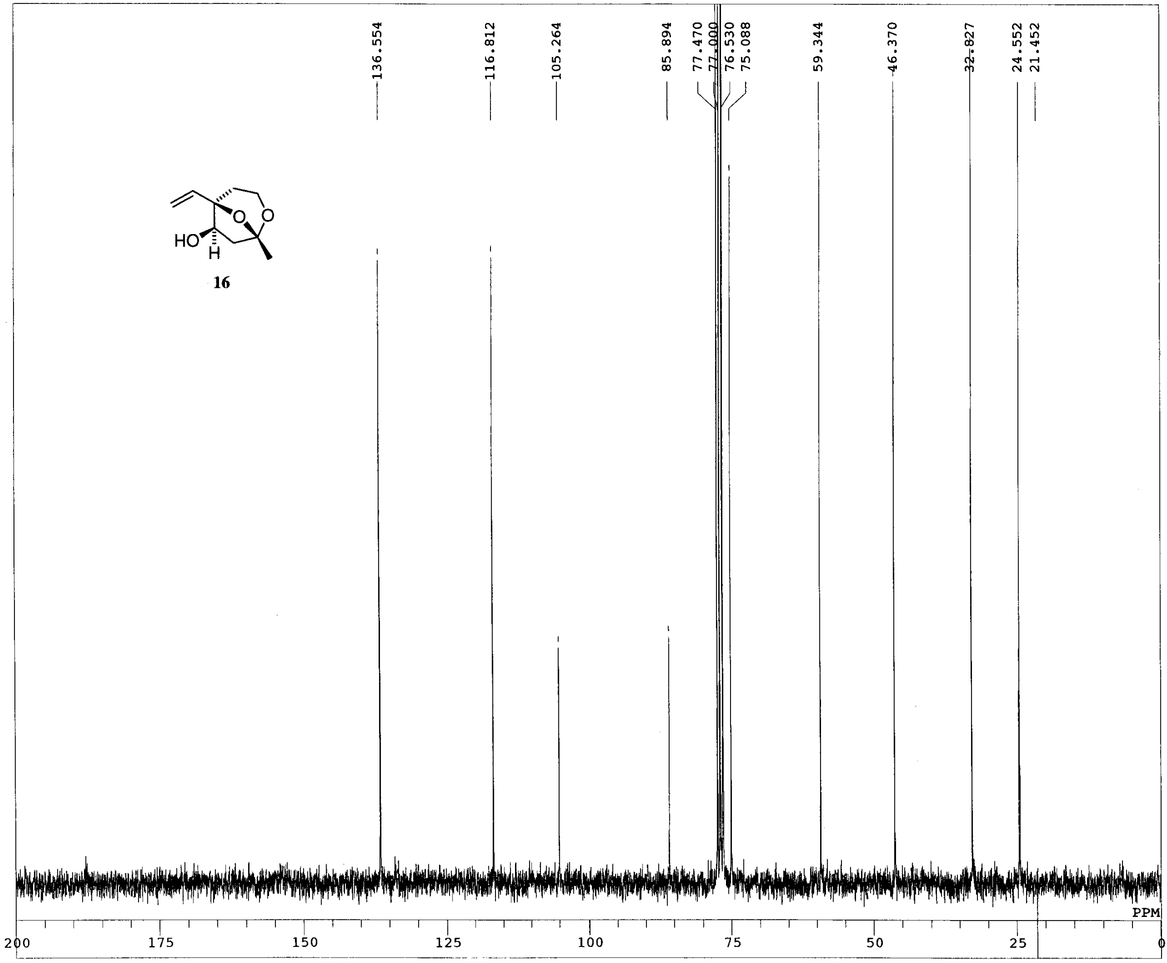


```
SPEED          12 Hz
CSPED          13 Hz
TEMP         80.0 c
CTEMP        23.3 c
TCOMP        1.00
SNO             1
HELEV       82.00
N2LEV       99.60
DATIM  Fri Nov 01 11:15:39 2002
PROBE  TH5
IRCODE        146
PRCODE       1412
DFILE  C:\WINNMR95\DATA\Auto2BCM_E1_FT.al
MENUF  BCM
SHMFL  TH5
COMNT
SHMCM  th5/2%age/CDCl3/40mm/17JAN02
SLVNT  CDCL3
OBNUC  13C
OBFRQ       67.80 MHz
OBSET      135.00 KHz
OBFIN      5200.0 Hz
OBST2        0.00 KHz
OBFN2         0.0 Hz
OBPHS         0.0 deg
OBATN         511
IRNUC  1H
IRFRQ      270.05 MHz
IRSET      112.00 KHz
IRFIN      5800.0 Hz
IRST2       50.00 KHz
IRFN2      3700.0 Hz
IRPHS         0.0 deg
IRATN         511
IRRPW          40 us
IRRNS           0
TRNUC  1H
TRFRQ      270.05 MHz
TRSET      112.00 KHz
TRFIN      5800.0 Hz
TRST2        0.00 KHz
TRFN2         0.0 Hz
TRPHS         0.0 deg
TRATN         511
TRRPW          70 us
TRRNS           0
PW1           3.7 us
PW2          10.0 us
PW3          10.0 us
PW4           1.0 us
PW5           1.0 us
PI1         1.000 ms
PI2         1.000 ms
PI3          1.00 ms
PI4          1.00 ms
PI5          1.00 ms
LOOP1           1
LOOP2           1
LOOP3           1
LOOP4           1
LOOP5           1
PS1         100.0 us
```

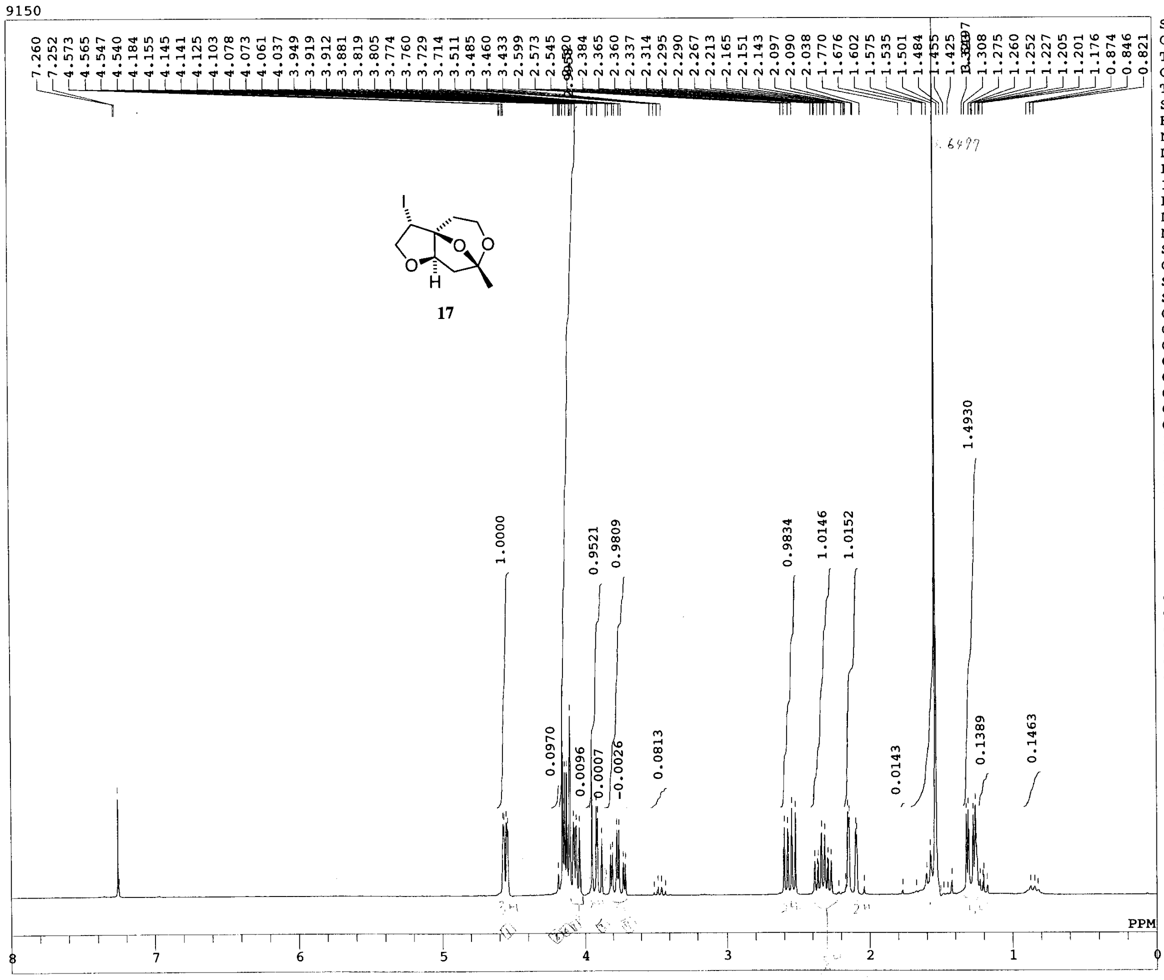


```
SPEED         12 Hz
CSPED         14 Hz
TEMP        80.0 c
CTEMP       24.5 c
TCOMP       1.00
SNO            1
HELEV      77.60
N2LEV     100.00
DATIM  Tue Nov 05 09:21:43 2002
PROBE  TH5
IRCODE       146
PRCODE      1412
DFILE  C:\WINNMR95\DATA\Auto1NON_E1_FT.al
MENUF  NON
SHMFL  TH5
COMNT
SHMCM  th5/2%age/CDCl3/40mm/17JAN02
SLVNT  CDCL3
OBNUC  1H
OBFRQ     270.05 MHz
OBSET     112.00 KHz
OBFIN     5800.0 Hz
OBST2       0.00 KHz
OBFN2        0.0 Hz
OBPHS        0.0 deg
OBATN        511
IRNUC  1H
IRFRQ     270.05 MHz
IRSET     112.00 KHz
IRFIN     5800.0 Hz
IRST2      50.00 KHz
IRFN2     3700.0 Hz
IRPHS        0.0 deg
IRATN        511
IRRPW         50 us
IRRNS          0
TRNUC  1H
TRFRQ     270.05 MHz
TRSET     112.00 KHz
TRFIN     5800.0 Hz
TRST2       0.00 KHz
TRFN2        0.0 Hz
TRPHS        0.0 deg
TRATN        511
TRRPW         70 us
TRRNS          0
PW1          5.5 us
PW2         10.0 us
PW3         10.0 us
PW4          1.0 us
PW5          1.0 us
PI1        1.000 ms
PI2        1.000 ms
PI3         1.00 ms
PI4         1.00 ms
PI5         1.00 ms
LOOP1          1
LOOP2          1
LOOP3          1
LOOP4          1
LOOP5          1
PS1        100.0 us
```





```
SPEED          12 Hz
CSPED          14 Hz
TEMP         80.0 c
CTEMP        24.2 c
TCOMP        1.00
SNO             1
HELEV       77.60
N2LEV      100.00
DATIM  Tue Nov 05 10:13:22 2002
PROBE  TH5
IRCODE        146
PRCODE       1412
DFILE  C:\WINNMR95\TEMPDATA\AUTO2BCM_E1_F
MENUF  BCM
SHMFL  TH5
COMNT
SHMCM  th5/2%age/CDCl3/40mm/17JAN02
SLVNT  CDCL3
OBNUC  13C
OBFRQ       67.80 MHz
OBSET      135.00 KHz
OBFIN      5200.0 Hz
OBST2        0.00 KHz
OBFN2         0.0 Hz
OBPHS         0.0 deg
OBATN         511
IRNUC  1H
IRFRQ      270.05 MHz
IRSET      112.00 KHz
IRFIN      5800.0 Hz
IRST2       50.00 KHz
IRFN2      3700.0 Hz
IRPHS         0.0 deg
IRATN         511
IRRPW          40 us
IRRNS           0
TRNUC  1H
TRFRQ      270.05 MHz
TRSET      112.00 KHz
TRFIN      5800.0 Hz
TRST2        0.00 KHz
TRFN2         0.0 Hz
TRPHS         0.0 deg
TRATN         511
TRRPW          70 us
TRRNS           0
PW1           3.7 us
PW2          10.0 us
PW3          10.0 us
PW4           1.0 us
PW5           1.0 us
PI1         1.000 ms
PI2         1.000 ms
PI3          1.00 ms
PI4          1.00 ms
PI5          1.00 ms
LOOP1           1
LOOP2           1
LOOP3           1
LOOP4           1
LOOP5           1
PS1         100.0 us
```



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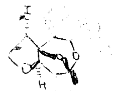
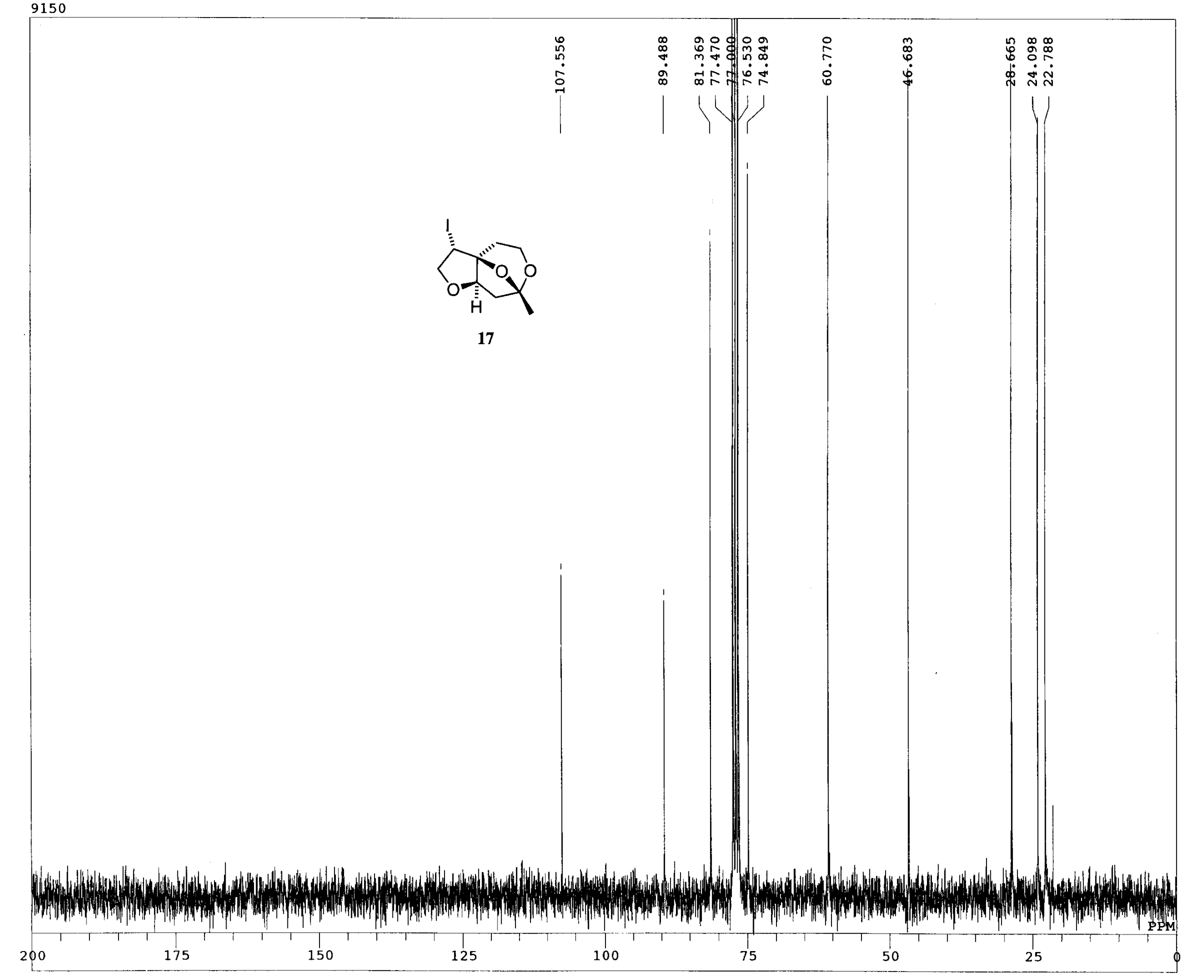
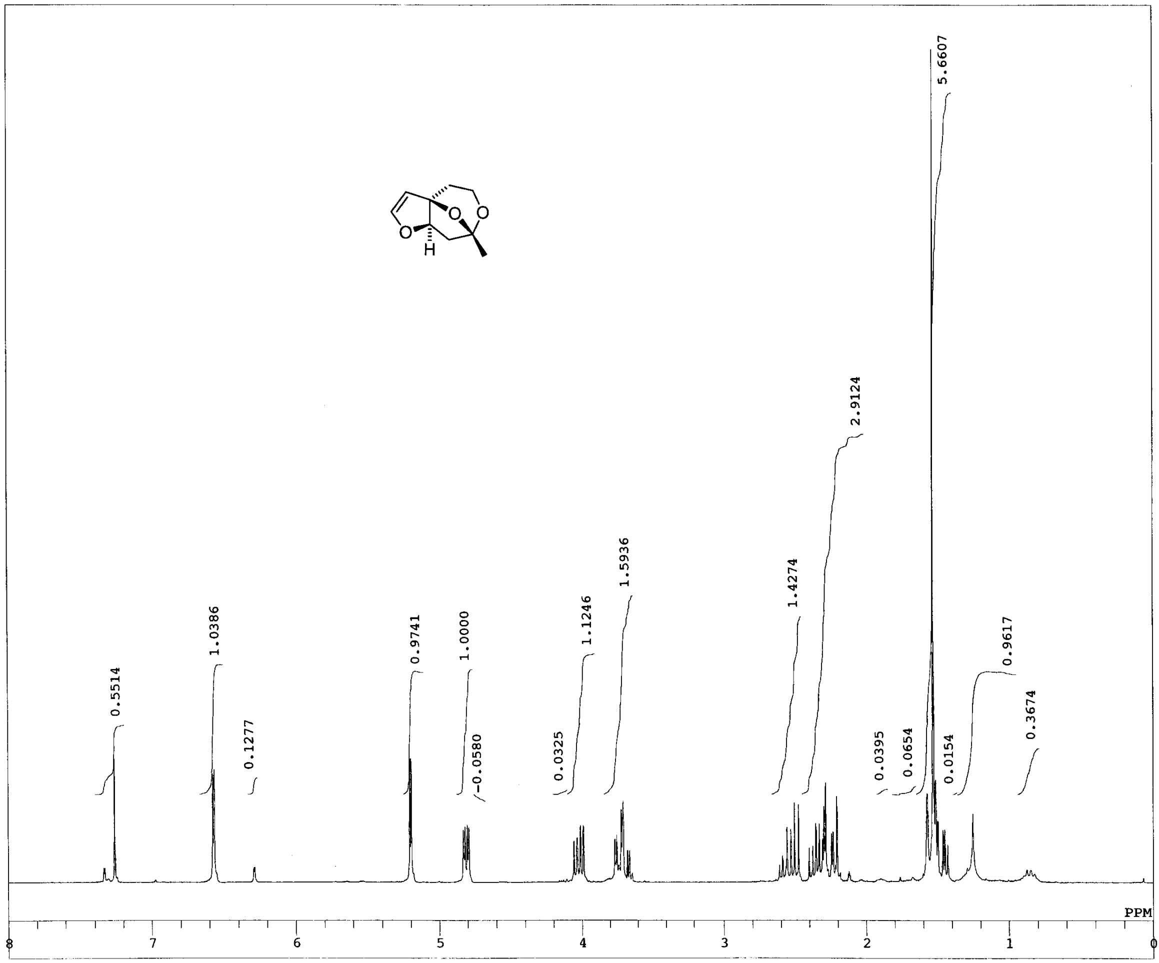
```
SPEED          12 Hz
CSPED          13 Hz
TEMP         80.0 c
CTEMP        23.2 c
TCOMP        1.00
SNO             1
HELEV       75.60
N2LEV      100.00
DATIM  Thu Nov 07 11:04:39 2002
PROBE  TH5
IRCODE        146
PRCODE       1412
DFILE  C:\WINNMR95\DATA\Auto1NON_E1_FT.al
MENUF  NON
SHMFL  TH5
COMNT  9150
SHMCM  th5/2%age/CDCl3/40mm/17JAN02
SLVNT  CDCL3
OBNUC  1H
OBFRQ      270.05 MHz
OBSET      112.00 KHz
OBFIN      5800.0 Hz
OBST2        0.00 KHz
OBFN2         0.0 Hz
OBPHS         0.0 deg
OBATN         511
IRNUC  1H
IRFRQ      270.05 MHz
IRSET      112.00 KHz
IRFIN      5800.0 Hz
IRST2       50.00 KHz
IRFN2      3700.0 Hz
IRPHS         0.0 deg
IRATN         511
IRRPW          50 us
IRRNS           0
TRNUC  1H
TRFRQ      270.05 MHz
TRSET      112.00 KHz
TRFIN      5800.0 Hz
TRST2        0.00 KHz
TRFN2         0.0 Hz
TRPHS         0.0 deg
TRATN         511
TRRPW          70 us
TRRNS           0
PW1           5.5 us
PW2          10.0 us
PW3          10.0 us
PW4           1.0 us
PW5           1.0 us
PI1         1.000 ms
PI2         1.000 ms
PI3          1.00 ms
PI4          1.00 ms
PI5          1.00 ms
LOOP1           1
LOOP2           1
LOOP3           1
LOOP4           1
LOOP5           1
PS1         100.0 us
```

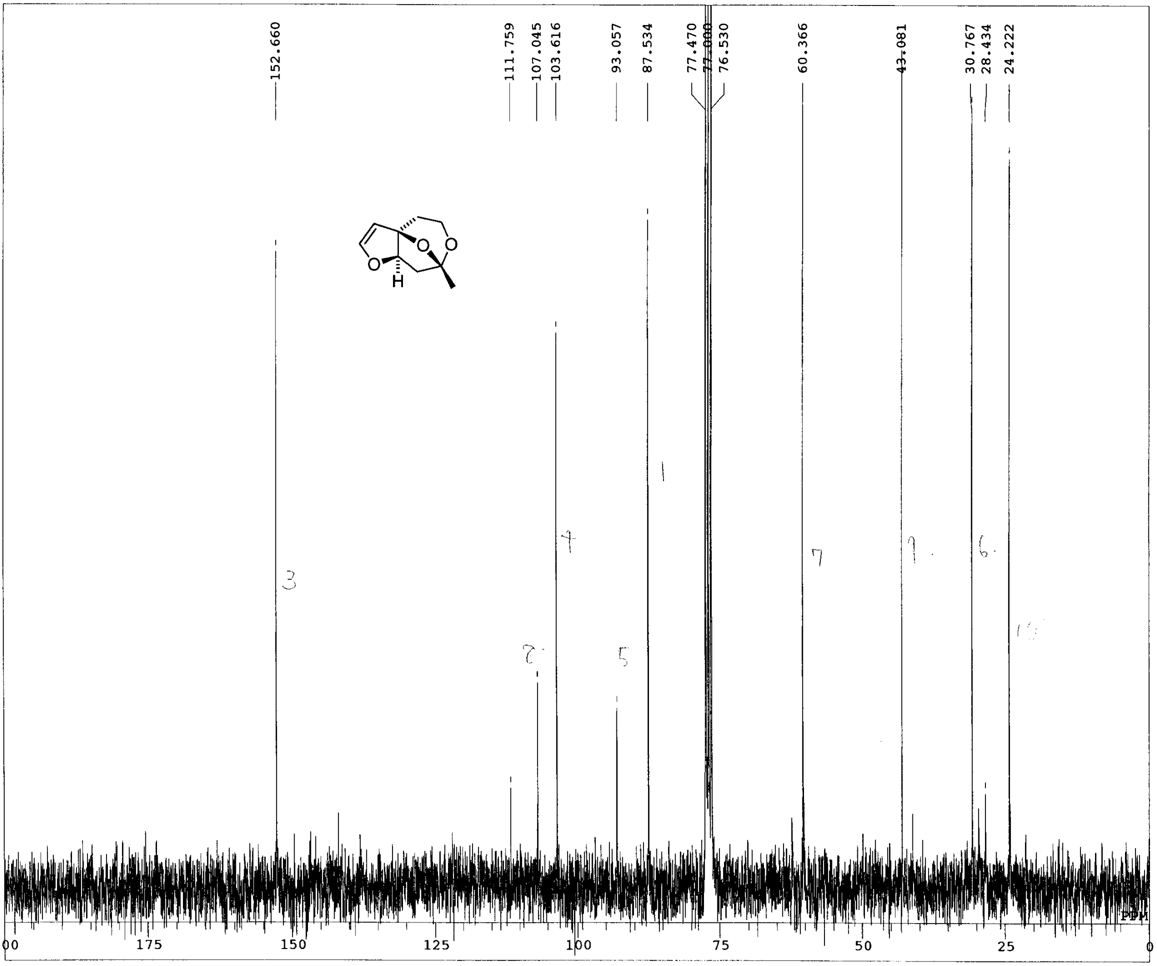




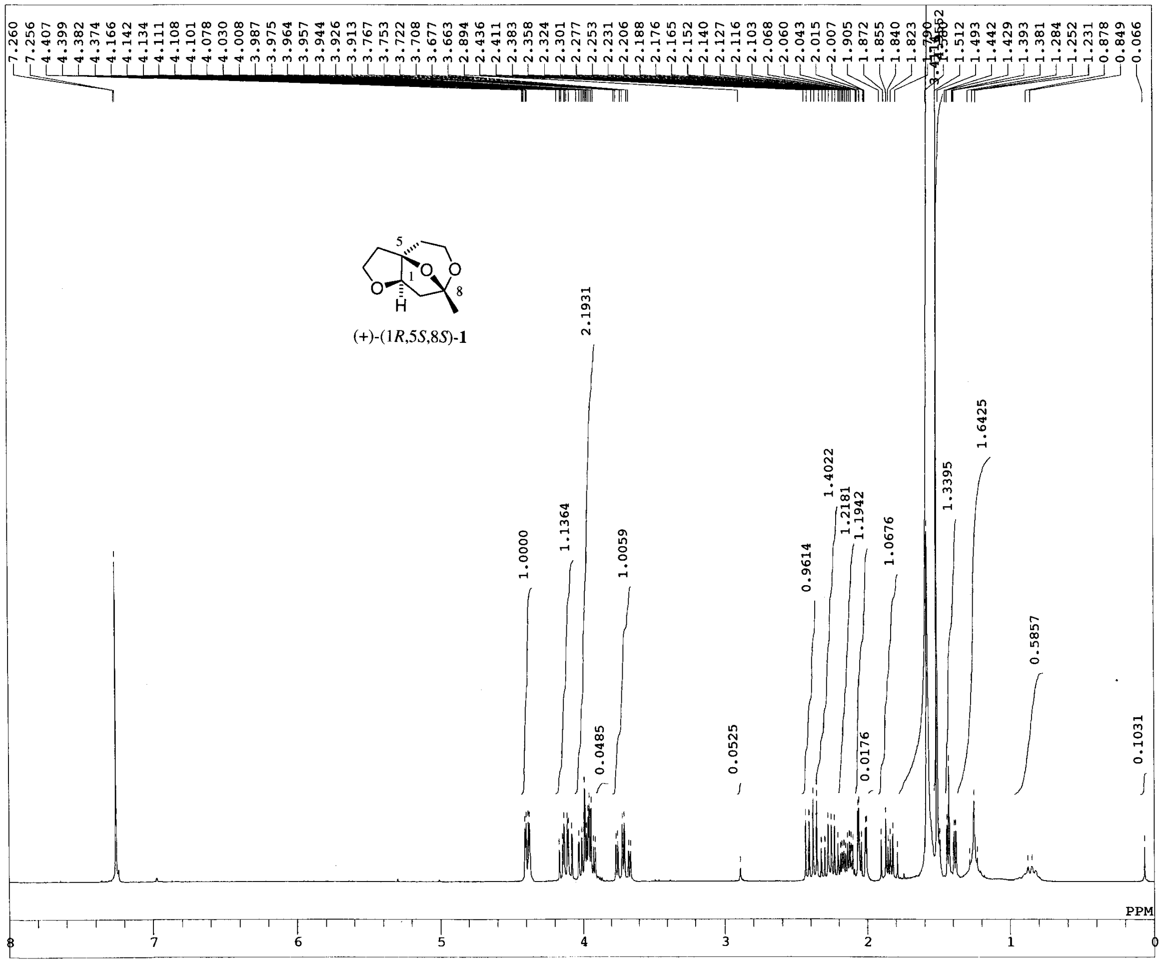
```
SPEED           12 Hz
CSPED           14 Hz
TEMP          80.0 c
CTEMP         24.2 c
TCOMP         1.00
SNO              1
HELEV        75.60
N2LEV       100.00
DATIM  Thu Nov 07 12:21:54 2002
PROBE  TH5
IRCODE         146
PRCODE        1412
DFILE  C:\WINNMR95\TEMPDATA\AUTO2BCM_E1_F
MENUF  BCM
SHMFL  TH5
COMNT  9150
SHMCM  th5/2%age/CDCl3/40mm/17JAN02
SLVNT  CDCL3
OBNUC  13C
OBFRQ        67.80 MHz
OBSET       135.00 KHz
OBFIN       5200.0 Hz
OBST2         0.00 KHz
OBFN2          0.0 Hz
OBPHS          0.0 deg
OBATN          511
IRNUC  1H
IRFRQ       270.05 MHz
IRSET       112.00 KHz
IRFIN       5800.0 Hz
IRST2        50.00 KHz
IRFN2       3700.0 Hz
IRPHS          0.0 deg
IRATN          511
IRRPW           40 us
IRRNS            0
TRNUC  1H
TRFRQ       270.05 MHz
TRSET       112.00 KHz
TRFIN       5800.0 Hz
TRST2         0.00 KHz
TRFN2          0.0 Hz
TRPHS          0.0 deg
TRATN          511
TRRPW           70 us
TRRNS            0
PW1            3.7 us
PW2           10.0 us
PW3           10.0 us
PW4            1.0 us
PW5            1.0 us
PI1          1.000 ms
PI2          1.000 ms
PI3           1.00 ms
PI4           1.00 ms
PI5           1.00 ms
LOOP1            1
LOOP2            1
LOOP3            1
LOOP4            1
LOOP5            1
PS1          100.0 us
```



```
SPEED           12 Hz
CSPED           14 Hz
TEMP          80.0 c
CTEMP         23.4 c
TCOMP         1.00
SNO              1
HELEV        72.80
N2LEV        99.60
DATIM  Sat Nov 09 09:09:45 2002
PROBE  TH5
IRCODE         146
PRCODE        1412
DFILE  C:\WINNMR95\DATA\ V,μ,¢ftƒHƒ‹ƒ_\Au
MENUF  NON
SHMFL  TH5
COMNT
SHMCM  th5/2%age/CDCl3/40mm/17JAN02
SLVNT  CDCL3
OBNUC  1H
OBFRQ       270.05 MHz
OBSET       112.00 KHz
OBFIN       5800.0 Hz
OBST2         0.00 KHz
OBFN2          0.0 Hz
OBPHS          0.0 deg
OBATN          511
IRNUC  1H
IRFRQ       270.05 MHz
IRSET       112.00 KHz
IRFIN       5800.0 Hz
IRST2        50.00 KHz
IRFN2       3700.0 Hz
IRPHS          0.0 deg
IRATN          511
IRRPW           50 us
IRRNS            0
TRNUC  1H
TRFRQ       270.05 MHz
TRSET       112.00 KHz
TRFIN       5800.0 Hz
TRST2         0.00 KHz
TRFN2          0.0 Hz
TRPHS          0.0 deg
TRATN          511
TRRPW           70 us
TRRNS            0
PW1            5.5 us
PW2           10.0 us
PW3           10.0 us
PW4            1.0 us
PW5            1.0 us
PI1          1.000 ms
PI2          1.000 ms
PI3           1.00 ms
PI4           1.00 ms
PI5           1.00 ms
LOOP1            1
LOOP2            1
LOOP3            1
LOOP4            1
LOOP5            1
PS1          100.0 us
```



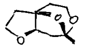
```
SPEED          12 Hz
CSPED          14 Hz
TEMP         80.0 c
CTEMP        24.1 c
TCOMP        1.00
SNO             1
HELEV       72.40
N2LEV      100.00
DATIM  Sat Nov 09 11:05:12 2002
PROBE  TH5
IRCODE        146
PRCODE       1412
DFILE  C:\WINNMR95\DATA\ V,μ,¢ƒtƒHƒ‹ƒ_\Au
MENUF  BCM
SHMFL  TH5
COMNT
SHMCM  th5/2%age/CDCl3/40mm/17JAN02
SLVNT  CDCL3
OBNUC  13C
OBFRQ       67.80 MHz
OBSET      135.00 KHz
OBFIN      5200.0 Hz
OBST2        0.00 KHz
OBFN2         0.0 Hz
OBPHS         0.0 deg
OBATN         511
IRNUC  1H
IRFRQ      270.05 MHz
IRSET      112.00 KHz
IRFIN      5800.0 Hz
IRST2       50.00 KHz
IRFN2      3700.0 Hz
IRPHS         0.0 deg
IRATN         511
IRRPW          40 us
IRRNS           0
TRNUC  1H
TRFRQ      270.05 MHz
TRSET      112.00 KHz
TRFIN      5800.0 Hz
TRST2        0.00 KHz
TRFN2         0.0 Hz
TRPHS         0.0 deg
TRATN         511
TRRPW          70 us
TRRNS           0
PW1           3.7 us
PW2          10.0 us
PW3          10.0 us
PW4           1.0 us
PW5           1.0 us
PI1         1.000 ms
PI2         1.000 ms
PI3          1.00 ms
PI4          1.00 ms
PI5          1.00 ms
LOOP1           1
LOOP2           1
LOOP3           1
LOOP4           1
LOOP5           1
PS1         100.0 us
```

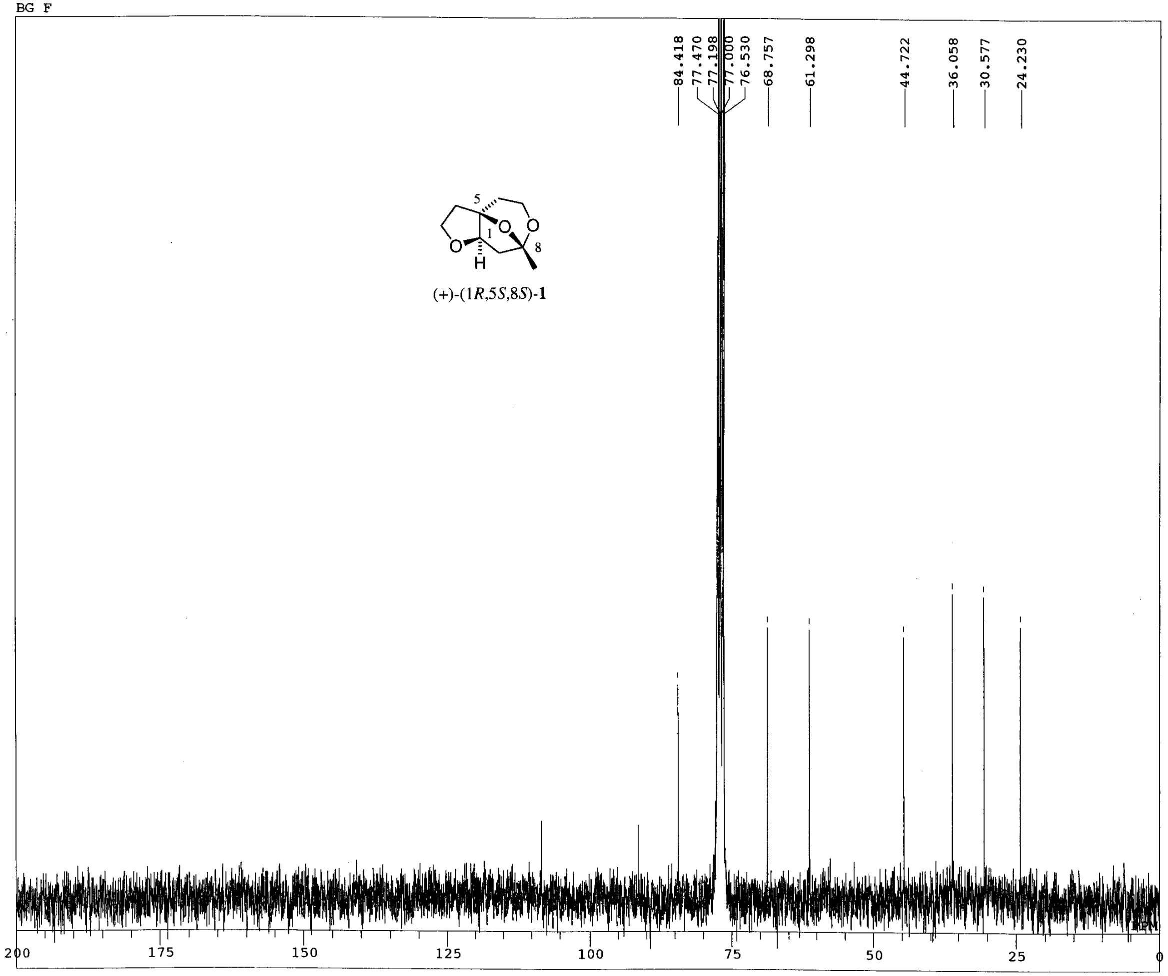


(+)-(1*R*,5*S*,8*S*)-**1**

```
SPEED         12 Hz
CSPED         14 Hz
TEMP        80.0 c
CTEMP       23.3 c
TCOMP       1.00
SNO            1
HELEV      70.80
N2LEV     100.00
DATIM  Mon Nov 11 16:48:46 2002
PROBE  TH5
IRCODE       146
PRCODE      1412
DFILE  C:\WINNMR95\DATA\Auto1NON_E1_FT.al
MENUF  NON
SHMFL  TH5
COMNT
SHMCM  th5/2%age/CDCl3/40mm/17JAN02
SLVNT  CDCL3
OBNUC  1H
OBFRQ     270.05 MHz
OBSET     112.00 KHz
OBFIN     5800.0 Hz
OBST2       0.00 KHz
OBFN2        0.0 Hz
OBPHS        0.0 deg
OBATN        511
IRNUC  1H
IRFRQ     270.05 MHz
IRSET     112.00 KHz
IRFIN     5800.0 Hz
IRST2      50.00 KHz
IRFN2     3700.0 Hz
IRPHS        0.0 deg
IRATN        511
IRRPW         50 us
IRRNS          0
TRNUC  1H
TRFRQ     270.05 MHz
TRSET     112.00 KHz
TRFIN     5800.0 Hz
TRST2       0.00 KHz
TRFN2        0.0 Hz
TRPHS        0.0 deg
TRATN        511
TRRPW         70 us
TRRNS          0
PW1          5.5 us
PW2         10.0 us
PW3         10.0 us
PW4          1.0 us
PW5          1.0 us
PI1        1.000 ms
PI2        1.000 ms
PI3         1.00 ms
PI4         1.00 ms
PI5         1.00 ms
LOOP1          1
LOOP2          1
LOOP3          1
LOOP4          1
LOOP5          1
PS1        100.0 us
```

Bo. 7





(+)-(1*R*,5*S*,8*S*)-**1**

```
SPEED          12 Hz
CSPED          13 Hz
TEMP         80.0 c
CTEMP        21.6 c
TCOMP        1.00
SNO             1
HELEV       71.60
N2LEV      100.00
DATIM  Mon May 27 08:26:22 2002
PROBE  TH5
IRCODE        146
PRCODE       1412
DFILE  C:\WINNMR95\TEMPDATA\AUTO1BCM_E1_F
MENUF  BCM
SHMFL  TH5
COMNT  BG F
SHMCM  th5/2%age/CDCl3/40mm/17JAN02
SLVNT  CDCL3
OBNUC  13C
OBFRQ       67.80 MHz
OBSET      135.00 KHz
OBFIN      5200.0 Hz
OBST2        0.00 KHz
OBFN2         0.0 Hz
OBPHS         0.0 deg
OBATN         511
IRNUC  1H
IRFRQ      270.05 MHz
IRSET      112.00 KHz
IRFIN      5800.0 Hz
IRST2       50.00 KHz
IRFN2      3700.0 Hz
IRPHS         0.0 deg
IRATN         511
IRRPW          40 us
IRRNS           0
TRNUC  1H
TRFRQ      270.05 MHz
TRSET      112.00 KHz
TRFIN      5800.0 Hz
TRST2        0.00 KHz
TRFN2         0.0 Hz
TRPHS         0.0 deg
TRATN         511
TRRPW          70 us
TRRNS           0
PW1           3.7 us
PW2          10.0 us
PW3          10.0 us
PW4           1.0 us
PW5           1.0 us
PI1         1.000 ms
PI2         1.000 ms
PI3          1.00 ms
PI4          1.00 ms
PI5          1.00 ms
LOOP1           1
LOOP2           1
LOOP3           1
LOOP4           1
LOOP5           1
```