A Nitrene-Transfer Reaction Catalyzed by Gold Complexes

Zigang Li, Xiangyu Ding and Chuan, He

Department of Chemistry, The University of Chicago 5735 S. Ellis Avenue

Chicago, IL 60637

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General method. All reagents were obtained commercially unless otherwise noted. All reactions were performed under a N₂ atmosphere. Acetonitrile was freshly distilled from CaH₂. Organic solutions were concentrated under reduced pressure (ca. 15 mm Hg) by rotary evaporation. Chromatographic purification of products was accomplished by using forced-flow chromatography on silica gel (35-75 μ m). Electrospray mass spectra (ESI-MS) were recorded on freshly prepared samples (ca. 1 mg/mL in CH₃CN) with an *m/z* range of 0-3000. A 5 μ L sample was injected by flow injection with the use of an autosampler. Purified nitrogen was used as both the nebulizing and drying gas. Typical instrumental parameters were: drying gas temperature 350 °C, nebulizer pressure 35 psi, drying gas flow 12.0 L/min, and fragmentor voltage 0 or 70 V. Visualization of the developed chromatogram was accomplished by fluorescence quenching and by staining with ethanolic anisaldehyde, aqueous potassium permanganate, or aqueous ceric ammonium molybdate (CAM) solution. Nuclear magnetic resonance (NMR) spectra were acquired on instruments operating at 500/400 and 125/100 MHz for ¹H and ¹³C, respectively, and are referenced internally according to residual protio solvent signals. Data for ¹H NMR are recorded as follows: chemical shift (ppm), multiplicity (s, singlet; d, doublet; m, multiplet), integration, coupling constant (Hz). Data for ¹³C NMR are reported in terms of chemical shift (δ , ppm).

Preparation of substrates: Substrates 1, 2 were synthesized according to references.¹ (Scheme 1) Other substrates were obtained from commercial sources and used without further purification.

Scheme 1

General procedure for the carbene insertion reaction. The gold(I) catalyst (3 mol %) was dissolved in 4 ml benzene with 65 mg ethyl diazoacetate (~0.5 mmol) under nitrogen. The reaction was stirred overnight at different temperatures. The reaction mixture was filtered through short celite pad, washed with CH_2Cl_2 , concentrated by vacuo, and analyzed by NMR.

New compound.

IR (% T): 3105(w), 3056(w), 2916(w), 2231(m), 1652(w), 1533(s), 1349(s), 1265(s), 1167(s), 1092(m), 737(s). ^1H NMR (δ , ppm): 8.41-8.39 (dd, 2H, J = 6.9, 1.9 Hz), 8.20-8.18 (dd, 2H, J =6.9, 1.9 Hz), 7.63-7.61 (dd, 2H, J =6.7, 1.7 Hz), 7.37-7.35 (dd, 2H, J =6.7, 1.7 Hz), 3.96-3.93 (dd, 1H, J =7.2, 4.4 Hz), 3.15-3.13 (d, 1H, J =7.2 Hz), 2.47-2.46 (d, 1H, J =4.4 Hz). ^{13}C NMR (δ , ppm): 150.8, 143.4, 139.6, 132.6, 129.3, 127.2, 124.5, 118.2, 112.6, 40.5, 37.1. HRMS: MW calculated: 329.3305, MW found($M+1$): 330.3430.

Spectra for carbene insertion: The crude proton NMR spectra of the reactions with $[\text{Au}(\text{tBu}_3\text{tPy})]_2(\text{OTf})_2$ at 80°C and 50°C shown in scheme 2 were attached. The ratios shown in the manuscript were calculated based on the proton integration.

Carbene Insertion 80 ° C

Carbene Insertion at 50° C

Current Data Parameters
 NAME 120-2-001-6
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050609
 Time 15.42
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 TD 65384
 SOLVENT CDCl3
 NS 4
 DS 0
 SHH 4807.682 Hz
 FIDRES 0.293436 Hz
 AQ 1.7039660 sec
 RG 32
 DM 104.000 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.50 usec
 PL1 0.00 dB
 SF01 400.1320007 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300346 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 7.526 ppm
 F1 3011.30 Hz
 F2P -0.651 ppm
 F2 -260.37 Hz
 PPMCH 0.40683 ppm/cm
 HZCM 163.58356 Hz/cm

Copies of product proton and carbon spectra

97/10/17, #5, 122.5 mg

Current Data Parameters
 NAME 1zg-2-80-1-p
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20050720
 Time 11.30
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 TD 16384
 SOLVENT CDCl3
 NS 4
 DS 0
 SWH 4807.582 Hz
 FIDRES 0.293436 Hz
 AQ 1.7039660 sec
 RG 101.6
 DM 104.000 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec

CHANNEL f1

NUC1 1H
 P1 10.50 usec
 PL1 0.00 dB
 SF01 400.1320007 MHz

F2 - Processing parameters

SF 32768
 SF 400.1300097 MHz
 EQ
 SS 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters

CX 20.00 cm
 FIP 10.705 ppm
 F1 4283.41 Hz
 F2 -1.310 ppm
 -524.28 Hz
 PPMCM 0.60077 ppm/cm
 HZCM 240.38454 Hz/cm

3.11210
 3.09411
 2.47994
 2.46852
 2.26282
 2.27972

8.39221
 8.36980
 8.19090
 8.16846
 7.24043
 7.21909
 7.05141
 7.02980

1.0000
 1.0119
 3.0598

1.4760
 1.5117
 1.9659
 1.6675

Integral

ppm

Current Data Parameters
 NAME 1zg-2-p-cn
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060117
 Time 9.59
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 TD 16384
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 4807.692 Hz
 FIDRES 0.293438 Hz
 AQ 1.7039860 sec
 RG 143.7
 DW 104.000 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.50 usec
 PL1 0.00 dB
 SF01 400.1320007 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300096 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 9.749 ppm
 F1 3900.72 Hz
 F2P 0.306 ppm
 F2 122.52 Hz
 PPMH 0.47212 ppm/cm
 HZCM 186.91039 Hz/cm

1.55769
 1.56260
 2.45559
 2.46558
 3.13489
 3.15298
 3.94897

7.26035
 7.26523
 7.35228
 7.37300
 7.61803
 7.62174
 7.63861
 8.18392
 8.18807
 8.20566
 8.40040
 8.40483
 8.42232

ppm

— 37.11
— 40.49

76.69
77.00
77.32

112.61
118.18
124.47
127.19
129.28
132.55
139.58
143.35
150.81

Current Data Parameters
NAME l2g-2-80-2-c
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050726
Time 18.54
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg
TD 121268
SOLVENT CDCl3
NS 92
DS 0
SWH 30303.031 Hz
FIDRES 0.250008 Hz
AQ 1.999820 sec
RG 4096
DM 16.500 usec
DE 7.50 usec
TE 300.0 K
D1 3.00000000 sec
d11 0.03000000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 11.00 usec
PL1 -2.00 dB
SF01 100.6258087 MHz

===== CHANNEL f2 =====
CPOPRG2 waltz16
NUC2 1H
PCPD2 101.00 usec
PL2 120.00 dB
PL12 19.00 dB
SF02 400.1324710 MHz

F2 - Processing parameters
SI 16384
SF 100.612755 MHz
WDM 0
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 157.547 ppm
F1 15851.24 Hz
F2P 18.595 ppm
F2 1870.85 Hz
PPMCM 6.94762 ppm/cm
HZCM 699.01923 Hz/cm

Current Data Parameters
 NAME 125-2-80-3-p
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050720
 Time 11.44
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 TO 16384
 SOLVENT DMSO
 NS 4
 DS 0
 SWH 4807.652 Hz
 FIDRES 0.293438 Hz
 AQ 1.7039860 sec
 RG 71.8
 DN 104.000 usec
 DE 5.00 usec
 TE 300.0 K
 D1 2.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.50 usec
 PL1 0.00 dB
 SF01 400.1320007 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300029 MHz
 NCM EM
 SSF 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 9.039 ppm
 F1 3616.89 Hz
 F2P -0.392 ppm
 F2 -157.02 Hz
 PPMCH 0.47159 ppm/cm
 HZCM 188.69563 Hz/cm

Current Data Parameters
 NAME 12g-2-m-me
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060117
 Time 11.42
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 TO 16384
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 4807.692 Hz
 FIDRES 0.293438 Hz
 AQ 1.7039860 sec
 RG 90.5
 DM 104.000 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.0000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.50 usec
 PL1 0.00 dB
 SF01 400.132007 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300098 MHz
 NQW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 10.301 ppm
 F1 4121.92 Hz
 F2P 0.319 ppm
 F2 127.52 Hz
 PPM0H 0.45914 ppm/cm
 HZ0H 199.71999 Hz/cm

97/10/17, #5, 122.5 mg

Current Data Parameters
NAME 1zg-2-82-2-c
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060720
Time 15.44
INSTRUM spect
PROBHD 5 mm GNP 1H
PULPROG zgpg30
TD 268090
SOLVENT Aceton
NS 25
DS 0
SWH 35682.538 Hz
FIDRES 0.166570 Hz
AQ 2.9999840 sec
RG 1024
DM 12.600 usec
DE 7.50 usec
TE 300.0 K
D1 4.00000000 sec
d11 0.03000000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 8.20 usec
PL1 0.00 dB
SF01 125.7736214 MHz

===== CHANNEL f2 =====
CPDPRG2 Meltz16
NUC2 1H
PCPD2 50.00 usec
PL2 120.00 dB
PL12 19.00 dB
SF02 500.1338000 MHz

F2 - Processing parameters
SI 32768
SF 125.7577946 MHz
WDM EN
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

ID NMR plot parameters
CX 20.00 cm
F1P 163.153 ppm
F1 20522.84 Hz
F2P -14.717 ppm
F2 -1650.84 Hz
PPHCH 8.89554 ppm/cm
HZCH 1118.68384 Hz/cm

Current Data Parameters
NAME jzg-2-p-cl
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080117
Time 9.53
INSTRUM spect
PROBHD 5 mm QNP 4H
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 8
DS 0
SWH 4807.692 Hz
FIDRES 0.293438 Hz
AQ 1.7039860 sec
RG 161.3
DM 104.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.0000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 10.50 usec
PL1 0.00 dB
SF01 400.1320007 MHz

F2 - Processing parameters
SI 32768
SF 400.1300096 MHz
WDW EN
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
FJP 8.874 ppm
F1 3550.70 Hz
F2P 0.266 ppm
F2 107.07 Hz
PQNCM 0.43031 ppm/cm
HZCM 172.18126 Hz/cm

1.54918
1.55484

2.45763
2.46892

3.09839
3.11642

3.86019
3.87154
3.87785
3.88944

7.14980
7.15425
7.17106
7.26011
7.26575
7.26618
7.29088
7.30742
8.17304
8.17719
8.19463
8.19980
8.38367
8.38843
8.40074
8.40567

ppm

Current Data Parameters
 NAME 12g-2-transmet
 EXPNO 1
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20060117
 Time 10.04
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 TO 16364
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 4807.622 Hz
 FIDRES 0.233438 Hz
 AQ 1.7039860 sec
 RG 101.5
 DM 104.000 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.0000000 sec
 ===== CHANNEL f1 =====
 NUC1 1H
 P1 10.50 usec
 PL1 0.00 dB
 SF01 400.1320007 MHz
 F2 - Processing parameters
 SI 32768
 SF 400.1300095 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00
 1D NMR plot parameters
 CX 20.00 cm
 F1P 10.572 ppm
 F1 4230.16 Hz
 F2P 0.409 ppm
 F2 163.69 Hz
 PPMCM 0.50814 ppm/cm
 HZCM 203.32317 Hz/cm

1.55133
1.54830

4.36325

7.25993
7.26294
7.37117
7.38008
7.38793
7.41293
7.41569
7.41932
7.42286
7.42838
7.43692
7.93115
7.93560
7.94834
7.95339
8.24772
8.25212
8.25500
8.26995

ppm

Integral

1.985
1.933
10.049

2.000

Current Data Parameters
NAME 129-2-tpn
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050118
Time 9.10
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 8
DS 0
SWH 4807.592 Hz
FIDRES 0.293438 Hz
AQ 1.7039860 sec
RG 101.5
DM 104.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.0000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 10.50 usec
PL1 0.00 dB
SFO1 400.1320007 MHz

F2 - Processing parameters
SI 32768
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

10 NMR plot parameters
CX 20.00 cm
F1P 10.611 ppm
F1 4245.60 Hz
F2P 0.293 ppm
F2 117.37 Hz
PPMCM 0.51586 ppm/cm
HZCM 206.41164 Hz/cm

S22

97/10/17, #5, 122.5 mg

51.327

76.741
76.994
77.249

124.040
128.096
128.671
129.140
132.218

145.485
150.182

Current Data Parameters
NAME 12g-2-78-6-c
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050718
Time 14.57
INSTRUM spect
PROBHD 5 mm QNP-1H
PULPROG zgpgc
TD 238080
SOLVENT Aceton
NS 16
DS 0
SWH 39882.539 Hz
FIDRES 0.166570 Hz
AQ 2.9999840 sec
RG 2048
DM 12.600 usec
DE 7.50 usec
TE 300.0 K
D1 4.00000000 sec
d11 0.03000000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 8.20 usec
PL1 0.00 dB
SF01 125.7738214 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 120.00 dB
PL12 19.00 dB
SF02 500.1330000 MHz

F2 - Processing parameters
S1 32768
SF 125.7577970 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CA 20.00 cm
F1P 157.062 ppm
F1 19754.32 Hz
F2P -7.932 ppm
F2 -597.54 Hz
PPHOM 8.25072 ppm/cm
HZOM 1037.59302 Hz/cm

97/10/17, #5, 122.5 mg

42.960
35.841
30.750
30.500
28.137
25.432

77.255
77.001
76.748

128.840
124.234
118.268
117.851

148.974
144.736

Current Data Parameters
NAME 17g-2-62-1-c
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050720
Time 15.41
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 238050
SOLVENT Aceton
NS 50
DS 0
SWH 39692.539 Hz
FIDRES 0.166670 Hz
AQ 2.9998840 sec
RG 1024
DW 12.600 usec
DE 7.50 usec
TE 300.0 K
D1 4.00000000 sec
d11 0.03000000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 8.20 usec
PL1 0.00 dB
SF01 125.7735214 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 120.00 dB
PL12 19.00 dB
SF02 500.1338000 MHz

F2 - Processing parameters
SI 32768
SF 125.7577934 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
FIP 153.037 ppm
F1 19245.56 Hz
F2P 5.625 ppm
F2 707.37 Hz
PPMCM 7.37059 ppm/cm
HZCM 526.90942 Hz/cm

Current Data Parameters
 NAME 129-2-oct
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060326
 Time 16.56
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 TD 16384
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 4807.692 Hz
 FIDRES 0.293438 Hz
 AQ 1.7039860 sec
 RG 203.2
 DM 104.000 usec
 DE 5.00 usec
 TE 300.0 K
 D1 2.0000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.50 usec
 PL1 0.00 dB
 SFO1 400.1320007 MHz

F2 - Processing parameters
 SI 32768
 SF 400.130002 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1 9.888 ppm
 F2 -0.438 ppm
 F2 -174.61 Hz
 PPMCM 0.51523 ppm/cm
 HZCM 206.15765 Hz/cm

Current Data Parameters
NAME 1zg-1-235-13-p
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050418
Time 10.30
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 5208.333 Hz
FIDRES 0.158946 Hz
AQ 3.1457779 sec
RG 128
DN 96.000 usec
DE 4.50 usec
TE 300.0 K
D1 2.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 10.00 usec
PL1 0.00 dB
SF01 500.1320118 MHz

F2 - Processing parameters
SI 16384
SF 500.1300167 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 9.195 ppm
F1 4599.25 Hz
F2P -1.218 ppm
F2 -609.08 Hz
PPMCM 0.52070 ppm/cm
HZCM 260.41665 Hz/cm

0.07
0.00
1.26
1.57
2.04
2.62
2.69
3.23
3.24
4.02
4.03
4.03
4.04

7.17
7.29
7.29
7.30
7.31
7.32
7.32
7.32
7.33
7.72
7.73
7.73
7.73
7.74
8.22
8.23

0.9834
0.9887
0.8492

Integral

97/10/17, #5, 122.5 mg

Current Data Parameters
NAME 129-1-235-13-C
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20050416
Time 10.34
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 238990
SOLVENT Aceton
NS 10
DS 0
SWH 39862.539 Hz
FIDRES 0.166570 Hz
AQ 2.9999840 sec
RG 4096
DM 12.800 usec
DE 7.50 usec
TE 300.0 K
D1 4.0000000 sec
d11 0.0300000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 8.20 usec
PL1 0.00 dB
SFO1 125.7736214 MHz

===== CHANNEL f2 =====
CPOPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 120.00 dB
PL12 19.00 dB
SFO2 500.1338000 MHz

F2 - Processing parameters

SF 32768
SF 125.7577970 MHz
WDW EN
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 20.00 cm
F1P 169.305 ppm
F1 21291.36 Hz
F2P -18.825 ppm
F2 -2367.35 Hz
PPMCM 9.40646 ppm/cm
HZCM 1182.93655 Hz/cm

Current Data Parameters
 NAME 129-1-221-1p
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050407
 Time 10.22
 INSTRUM spect
 PROBHD 5 mm GNP 1H
 PULPROG zg
 TD 16384
 SOLVENT CDCl3
 NS 8
 DS 0
 SMH 4807.692 Hz
 FIDRES 0.293438 Hz
 AQ 1.7039860 sec
 RG 57
 DM 104.000 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.50 usec
 PL1 0.00 dB
 SF01 400.1320007 MHz
 F2 - Processing parameters
 SI 32768
 SF 400.1300119 MHz
 MDW EM
 SS9 0
 LB 0.30 Hz
 GB 0
 PC 1.00
 ID NMR plot parameters
 CX 20.00 cm
 FIP 8.333 ppm
 F1 3334.44 Hz
 F2P -0.273 ppm
 F2 -109.24 Hz
 PPKCM 0.43032 ppm/cm
 HZCM 172.16390 Hz/cm

2.99003
 2.97205
 2.42703
 2.39006
 2.37903

7.87841
 7.85792
 7.33712
 7.31647
 7.29184
 7.27849
 7.27626
 7.22481
 7.21833
 7.20561

1.4075
 1.8631
 2.0843
 1.4395

21.63
35.91
40.99

76.69
77.00
77.33

126.52
127.90
128.27
128.53
129.72
135.00
144.53

ppm

Current Data Parameters
NAME 129-1-227-1-PC
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050407
Time 12.48
INSTRUM spect
PROBHD 5 mm QNP
PULPROG zgpg30
TD 121208
SOLVENT CDCl3
NS 65
DS 0
SWH 30303.031 Hz
FIDRES 0.250008 Hz
AQ 1.999820 sec
RG 2580.3
DM 16.503 usec
DE 7.50 usec
TE 300.0 K
D1 3.00000000 sec
d11 0.03000000 sec
611

===== CHANNEL f1 =====
NUC1 13C
P1 11.00 usec
PL1 -2.00 dB
SFO1 100.6250007 MHz

===== CHANNEL f2 =====
CQPRG2 Meltz16
NUC2 1H
PCPD2 101.00 usec
PL2 120.00 dB
PL12 19.00 dB
SFO2 400.1324710 MHz

F2 - Processing parameters
S1 16384
SF 100.6127755 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CA 20.00 cm
F1P 280.168 ppm
F1 28166.51 Hz
F2P -21.016 ppm
F2 -2114.52 Hz
PPHOM 15.05924 ppm/cm
HZCM 1515.15161 Hz/cm

Current Data Parameters
 NAME 12g-2-65-2-c
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050722
 Time 15.30
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zgpg30
 TD 238090
 SOLVENT Acetone
 NS 15
 DS 0
 SWH 39562.539 Hz
 FIDRES 0.166670 Hz
 AQ 2.9959940 sec
 RG 2048
 DM 12.600 usec
 DE 7.50 usec
 TE 300.0 K
 D1 4.00000000 sec
 d11 0.03000000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 8.20 usec
 PL1 0.00 dB
 SF01 125.7736214 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 P2 90.00 usec
 PL2 120.00 dB
 PL12 19.00 dB
 SF02 500.1338000 MHz

F2 - Processing parameters
 SI 32768
 SF 125.7578019 MHz
 NQM EN
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 F1P 167.255 ppm
 F1 21032.67 Hz
 F2P -19.872 ppm
 F2 -2499.00 Hz
 PPMCH 9.35535 ppm/cm
 HZCH 1176.63342 Hz/cm

18.537

35.926

41.244

76.709
 76.962
 77.216

122.716
 126.641
 128.270
 128.443
 134.810
 138.087
 138.196

150.727
 153.140

ppm

Current Data Parameters
 NAME 1z9-2-84-6-c
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050721
 Time 16.54
 INSTRUM spect
 PROBHD 5 mm GNP 1H
 PULPROG zgpg
 TD 121208
 SOLVENT CDCl3
 NS 84
 DS 0
 SWH 30303.031 Hz
 FIDRES 0.250008 Hz
 AQ 1.9999820 sec
 RG 4095
 DM 16.500 usec
 DE 7.50 usec
 TE 300.0 K
 D1 3.00000000 sec
 d11 0.03000000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 11.00 usec
 PL1 -2.00 dB
 SF01 100.6258067 MHz

===== CHANNEL f2 =====
 CDP062 wait216
 NUC2 1H
 P0P02 101.00 usec
 PL2 120.00 dB
 PL12 19.00 dB
 SF02 400.1324710 MHz

F2 - Processing parameters
 SI 16384
 SF 100.6127717 MHz
 NCM EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00

ID NMR plot parameters
 CX 20.00 cm
 FIP 145.571 ppm
 F1 14546.35 Hz
 F2P -15.164 ppm
 F2 -1525.73 Hz
 PPMCM 8.03679 ppm/cm
 HZCM 808.60419 Hz/cm

1.02

37.53

42.73

76.71
77.03
77.35
79.67

126.54
128.80
128.93
133.80

ppm

Reference

- (1) (a) Guthikonda, K.; Du Bois, J. *J. Am. Chem. Soc.* **2002**, *124*, 13672. b) Duran, F.; Leman, L.; Ghini, A.; Burton, G.; Dauban, P.; Dodd, R. H. *Org. Lett.* **2002**, *4*, 2481.
- (3) For entry 1, 4, 5, 6, 7 in table 2 cited in Ryan, D.; McMorn, P.; Bethell, D.; Hutchings, G. *Org. Biomol. Chem.* **2004**, *2*, 3566. entry 2, 8, 9 in table 2 cited in Muller, P.; Baud, C.; Jacquier, Y. *Tetrahedron*, **1996**, *52*, 1543. entry 10 in table 2 cited in: Sodergren, M. J.; Alonso, D. A.; Andersson, P. G. *Tetrahedron: Asymmetry* **1997**, *8*, 3563, entry 11, 12 in table 2 cited in Leung, S. K.-Y.; Tsui, W.-M.; Huang, J.-S.; Che, C.-M.; Liang, J.-L.; Zhu, N. *J. Am. Chem. Soc.* **2005**, *127*, 16629. entry 13 in table 2 cited in 2b. entry 1 in table 3 cited in Kim, S. K.; Jacobsen, E. N. *Angew. Chem. Intl. Ed.* **2004**, *43*, 3952. entry 2 of table 3 commercial available, entry 3 in table 3 cited in Han, H; Bae, I.; Yoo, E. J.; Lee, J.; Do, Y.; Chang, S. *Org. Lett.* **2004**, *6*, 4109. entry 4 of table 3, 2a.