Supporting Information

Organocatalytic Mitsunobu Reactions

Tracy Yuen Sze But and Patrick H. Toy*

Department of Chemistry, The University of Hong Kong, Pokfulam Road, Hong Kong,
People's Republic of China

E-mail: phtoy@hku.hk

General Methods. All reactions were carried out under nitrogen in oven-dried or flame-dried glassware. Tetrahydrofuran was purified using a Solv-Tek purification system employing activated Al_2O_3 . Triphenylphosphine was recrystallized from hexane prior to use. Other commercially available reagents were used as received. All reactions were monitored by TLC analysis using GF254 silica gel coated plates. Column chromatography was carried out using silica gel (300-400 mesh) at increased pressure. ^1H - and ^{13}C -NMR spectra were recorded in CDCl_3 on a Bruker DRX-400 spectrometer operating at 400 MHz for ^1H analyses and at 75 MHz for ^{13}C analyses. Chemical shift data is expressed in ppm with reference to TMS. HR EI-MS data was recorded on a Finnigan MAT 96 mass spectrometer. Optical rotation data were recorded in CHCl_3 or EtOH on a Perkin Elmer Polarimeter 343 operating at 20 °C for 589 nm. HPLC analyses were carried out using a Waters Delta Prep 4000 chromatography system with the indicated columns.

General procedure for catalytic Mitsunobu reactions. Procedure A. To 4-nitrobenzoic acid (0.33 g, 1.98 mmol) and 2-phenylethanol (0.21 mL, 1.8 mmol) in anhydrous THF (15 mL), was added triphenylphosphine (0.94 g, 3.6 mmol), diethyl azodicarboxylate (0.028 mL, 0.18 mmol), and iodosobenzene diacetate (1.16 g, 3.6 mmol). The reaction was stirred at rt for 16 h and then the reaction mixture was diluted with diethyl ether (30 mL). The organic phase was washed with saturated aq. NaHCO₃ (2 x 20 mL) and brine (20 mL). The organic layer was dried with Na₂SO₄, filtered and evaporated. The residue was purified by column chromatography to afford 2-phenylethyl 4-nitrobenzoate¹ as yellow solid: TLC (1:4 EA/hexane) R_f 0.55; ¹H-NMR (400 MHz, CDCl₃) δ 3.10 (t, 2H, *J* = 6.9 Hz), 4.59 (t, 2H, *J* = 6.9 Hz), 7.20-7.36 (m, 5H), 8.16 (d, 2H, *J* = 8.8 Hz), 8.28 (d, 2H, *J* = 8.8 Hz); ¹³C-NMR (100 MHz, CDCl₃) δ 55.2, 67.5, 114.0, 123.4, 127.3, 130.3, 130.7, 135.6, 150.5, 164.5. The yield for the background reaction was determined by ¹H-NMR since the desired product was mixed with 2-phenylethyl acetate.

General procedure for slow addition of PPh₃ catalytic Mitsunobu reactions. Procedure B. To 4-nitrobenzoic acid (0.33 g, 1.98 mmol) and 2-phenylethanol (0.21 mL, 1.8 mmol) in anhydrous THF (11 mL), was added diethyl azodicarboxylate (0.028 mL, 0.18 mmol), and iodosobenzene diacetate (1.16 g, 3.6 mmol), then triphenylphosphine (0.94 g, 3.6 mmol) in 4 mL anhydrous THF was added slowly by syringe pump. The reaction was stirred at rt for 16 h and then the reaction mixture was diluted with diethyl ether (30 mL). The organic phase was washed with saturated aq. NaHCO₃ (2 x 20 mL) and brine (20 mL). The organic layer was dried with Na₂SO₄, filtered and evaporated. The residue was purified by column chromatography.

4-Phenylbutyl 4-nitrobenzoate. General procedure A was

followed using 4-nitrobenzoic acid (0.33 g, 1.98 mmol) and 4-phenylbutanol (0.27 mL, 1.8 mmol), to afford a yellow oil:

TLC (1:4 EA/hexane) R_f = 0.62; $^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 1.81 (m, 4H), 2.70 (t, 2H, J = 7.3 Hz), 4.39 (t, 2H, J = 6.1 Hz), 7.18-7.29 (m, 5H), 8.27 (d, 2H, J = 1.9 Hz), 8.29 (d, 2H, J = 1.9 Hz); $^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 27.6, 28.1, 35.3, 65.7, 123.4, 125.9, 128.3, 130.6, 135.7, 141.7, 150.4, 164.6; HRMS for $\text{C}_{17}\text{H}_{17}\text{NO}_4$: calcd 299.1158, found 299.1158.

Benzyl 4-nitrobenzoate.² General procedure A was followed using

4-nitrobenzoic acid (0.33 g, 1.98 mmol) and benzyl alcohol (0.18 mL, 1.8 mmol), to afford a yellow solid: TLC (1:4 EA/hexane) R_f = 0.58;

$^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 5.40 (s, 2H), 7.26-7.39 (m, 5H), 8.16 (d, 2H, J = 9 Hz), 8.28 (d, 2H, J = 9 Hz); $^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 67.7, 123.5, 128.4, 128.5, 128.6, 128.7, 130.8, 135.2, 135.4, 150.3, 164.5; HRMS for $\text{C}_{14}\text{H}_{11}\text{NO}_4$: calcd 257.0688, found 257.0688.

4-Bromobenzyl 4-nitrobenzoate.³ General procedure A was

followed using 4-nitrobenzoic acid (0.33 g, 1.98 mmol) and 4-bromobenzyl alcohol (0.33 g, 1.8 mmol), to afford a pale yellow

solid: TLC (1:4 EA/hexane) R_f = 0.58; $^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 5.34 (s, 2H), 7.32 (d, 2H, J = 8.1 Hz), 7.53 (d, 2H, J = 8.1 Hz), 8.23 (d, 2H, J = 12 Hz), 8.27 (d, 2H, J = 12 Hz); $^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 66.8, 123.5, 130.1, 130.8, 131.9, 162.3; HRMS for $\text{C}_{14}\text{H}_{10}\text{BrNO}_4$: calcd 334.9793, found 334.9793.

4-Methoxybenzyl 4-nitrobenzoate.³ Procedure A was

followed using 4-nitrobenzoic acid (0.33 g, 1.98 mmol) and 4-methoxybenzyl alcohol (0.22 mL, 1.8 mmol), to afford a pale

yellow solid: TLC (1:4 EA/hexane) R_f = 0.45 ; $^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 3.82 (s, 3H), 5.33 (s, 2H), 6.92 (d, 2H, J = 8.7 Hz), 7.39 (d, 2H, J = 8.7 Hz), 8.22 (d, 2H, J = 9 Hz), 8.25 (d, 2H, J = 9 Hz); $^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 55.2, 67.5, 114.0, 123.4, 127.3, 130.3, 130.7, 135.6, 150.3, 159.9, 164.5; HRMS for $\text{C}_{15}\text{H}_{13}\text{NO}_5$: calcd 287.0794, found 287.0793.

Benzyl phenylacetate.⁴ Procedure A was followed using phenyl acetic

acid (0.33 g, 1.98 mmol) and benzyl alcohol (0.18 mL, 1.8 mmol), to

afford a colorless oil: TLC (1:4 EA/Hexane) R_f 0.61; $^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 3.67 (s, 2H), 5.13 (s, 3H), 7.25-7.35 (m, 10H); $^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 41.2, 66.4, 127.0, 128.0, 128.1, 128.43, 128.47, 129.2, 133.8, 135.7, 171.2; HRMS for $\text{C}_{15}\text{H}_{14}\text{O}_2$: calcd 226.0993, found 226.0994.

2-Phenylethyl N-Boc-2-aminoacetate.⁵ Procedure A was

followed using Boc-glycine (0.34 g, 1.98 mmol) and

2-phenylethanol (0.21 mL, 1.8 mmol), to afford a colorless oil: TLC (1:4 EA/Hexane) R_f = 0.33; $^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 1.45 (s, 9H), 2.95 (t, 2H, J = 7.2 Hz), 3.89 (d, 2H, J = 5.2 Hz), 4.35 (t, 2H, J = 7.2 Hz), 4.93 (s, 1H) 7.19-7.30 (m, 5H); $^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 82.2, 34.9, 42.3, 65.5, 79.8, 126.5, 128.4, 128.7, 137.3, 155.6, 170.2.

(R)-1-Phenylethyl 4-nitrobenzoate.⁶ Procedure B was followed, using 4-nitrobenzoic acid (0.33 g, 1.98 mmol) and (*S*)-1-phenylethanol (0.21 mL, 1.8 mmol), to afford a yellow oil: TLC (1:4 EA/Hexane) R_f = 0.61; $^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 1.71 (d, 3H, J = 6.8 Hz), 6.15 (q, 1H, J = 6.8 Hz), 7.32-7.45 (m, 5H) 8.22 (d, 2H, J = 8.8 Hz), 8.26 (d, 2H, J = 8.8 Hz); $^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 22.1, 74.1, 123.4, 126.0, 128.2, 128.6, 130.6, 135.8, 140.9, 150.4, 163.8; HRMS for $\text{C}_{15}\text{H}_{13}\text{NO}_4$: calcd 271.0844, found 271.0844.

Ethyl (*R*)-2-((4-nitrobenzoyl)oxy)propionate.⁷ Procedure B was followed, using 4-nitrobenzoic acid (0.33 g, 1.98 mmol) and L-ethyl lactate (0.20 mL, 1.8 mmol), to afford a yellow solid: TLC (1:4

EA/Hexane) R_f = 0.46; $^1\text{H-NMR}$ (300 MHz, CDCl_3) δ 1.29 (t, 3H, J = 7.1 Hz), 1.66 (d, 3H, J = 7.0 Hz), 4.23 (q, 2H, J = 7.1 Hz), 5.35 (q, 1H, J = 7.0 Hz), 8.27 (d, 2H, J = 1.2 Hz), 8.29 (d, 2H, J = 1.2 Hz); $^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 14.0, 16.9, 61.6, 69.9, 123.5, 130.9, 134.8, 150.7, 164.0, 170.1; HRMS for $\text{C}_{12}\text{H}_{13}\text{NO}_6$: calcd 267.0743, found 267.0743.

(S)-2-Butyl 4-nitrobenzoate.⁸ Procedure B was followed, using 4-nitrobenzoic acid (0.33 g, 1.98 mmol) and (*R*)-2-butanol (0.27 mL, 1.8 mmol), to afford a yellow liquid: TLC (1:4 EA/Hexane) R_f = 0.64; $^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 0.98 (t, 3H, J = 7.4 Hz), 1.36 (d, 3H, J = 6.2 Hz), 1.75 (m, 1H), 5.13 (m, 1H), 8.21 (d, 2H, J = 8.9 Hz), 8.29 (d, 2H, J = 8.9 Hz); $^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 9.6, 19.3, 28.7, 74.1, 123.4, 130.5, 136.2, 150.3, 164.2; HRMS for $\text{C}_{11}\text{H}_{13}\text{NO}_4$: calcd 223.0844, found 223.0844.

Characterization data of the products prepared from chiral secondary alcohols:

		Yield (%)	Optical Rotation		HPLC	
			Cat.	Stoic.	Cat.	Stoic.
First Set	65	-24.3°	-51.4°	ee = 65% (graph A-2)	ee = 86% (graph A-1)
	Second Set	59	-25.0°	-52.0°	ee = 65% (graph A-3)	
	Third Set	56	-36.2°	-53.3°	ee = 72% (graph A-4)	
	Fourth Set	55	-39.8°	-54.4°	ee = 72% (graph A-5)	
						Average ee = 68%

HPLC traces:

OJ column, 0.5mL/min, 50% IPA in hexane

Graph A-1, ee = $93 - 7 = 86\%$

Graph A-2, ee = $82.71 - 17.29 = 65\%$

Graph A-3, ee = $82.62 - 17.38 = 65\%$

Graph A-4, ee = $85.92 - 14.08 = 72\%$

Graph A-5, ee = $86.03 - 13.97 = 72\%$

		Yield (%)	Optical Rotation		HPLC	
			Cat.	Stoic.	Cat.	Stoic.
First Set	61	-12.7° (EtOH)	-13.5° (EtOH)	For this substrate, Daicel OD, OJ, OT columns were used with different flow rates. However, all of the attempted separations afforded only a single peak, including when the conditions used to separate the enantiomers of the other two chiral products were used.	
	Second Set	75	-12.2° (EtOH)	-12.3° (EtOH)		
	Third Set	60	-11.9° (EtOH)	-12.8° (EtOH)		

HPLC traces:

OD column, 0.5mL/min, 50% EtOH in hexane

OD column, 0.7mL/min, 50% IPA in hexane

OD column, 0.4mL/min, 50% IPA in hexane

OT column, 0.5mL/min, 10% MeOH in CH₂Cl₂

OJ column, 0.7mL/min, 50% IPA in hexane

OJ column, 0.5mL/min, 50% IPA in hexane

OJ column, 0.5mL/min, 30% IPA in hexane

OJ column, 0.7mL/min, 4% IPA in hexane

OJ column, 0.7mL/min, 2% IPA in hexane

OJ column, 0.5mL/min, 1% IPA in hexane

		Yield (%)	Optical Rotation		HPLC	
			Cat.	Stoic.	Cat.	Stoic.
First Set	60	+29.9°	+31.1°	ee = 87% (graph B-2)	ee = 88% (graph B-1)
	Second Set	60	+30.5°	+30.9°	ee = 87% (graph B-3)	
	Third Set	70	+31.4°	+31.7°	ee = 87% (graph B-4)	
					Average ee = 87%	

HPLC traces:

OJ column, 0.5mL/min, 1% IPA in hexane

Graph B-1, $ee = 94 - 6 = 88\%$

Graph B-2, $ee = 93.59 - 6.41 = 87\%$

Graph B-3, $ee = 93.60 - 6.40 = 87\%$

Graph B-4, ee = 93.67 – 6.33 = 87%

References:

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Current Data Parameters
 NAME 330A
 EXPNO 1
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20050308
 Time 17.05
 INSTRUM dm300
 PROGNO DUAL 5mm
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 22
 DS 2
 SWH 4495.483 Hz
 FIDRES 0.137219 Hz
 AQ 3.6438515 sec
 RG 456.1
 DM 111.280 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

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 NUC1 1H
 P1 9.90 usec
 PL1 -6.00 dB
 SFO1 300.1315007 MHz
 F2 - Processing parameters
 SI 32768
 SF 300.1300095 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

ID NMR plot parameters
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 FI 3001.30 Hz
 F2P -1.000 ppm
 F2 -280.13 Hz
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 HZCM 132.95721 Hz/cm

Current Data Parameters
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 PROCNO 1
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 Time 13.49
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 PROGNO 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 282
 DS 0
 SWH 25125.629 Hz
 FIDRES 0.758773 Hz
 AQ 0.5521332 sec
 RG 2580.3
 DM 19.900 usec
 DE 6.00 usec
 TE 300.0 K
 D1 3.00000000 sec
 D11 0.03000000 sec

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 P1 9.10 usec
 PL1 -6.00 dB
 SFO1 100.6280654 MHz

----- CHANNEL f2 -----
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 PCPD2 80.00 usec
 PL2 -4.00 dB
 PL12 12.00 dB
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F2 - Processing parameters
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 SF 100.6127729 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

ID NMR plot parameters
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 FI 22134.81 Hz
 F2P -10.000 ppm
 F2 -1006.13 Hz
 PPMCH 9.20000 ppm/cm
 HZCM 925.63751 Hz/cm

Current Data Parameters
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 AXCNO 1

2 - Acquisition Parameters
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 Time 10.47
 INSTRUM dpx300
 PROCNO DUAL 5mm

7a PROC 2930
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 SOLVENT CDCl3
 VS 15
 IS 2
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 TE 300.0 K
 SI 1.00000000 sec

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f2 - Processing parameters
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 SF 300.1300663 MHz
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 GB 0
 GC 1.00

10 NMR plot parameters
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 F2P -1.000 ppm
 F2 -300.13 Hz
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Integral

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1.9918
2.0359

1.5548
1.5521

0.0001
0.0035

Current Data Parameters
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 AXCNO 1

2 - Acquisition Parameters
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 PROCNO DUAL 5mm

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 VS 15
 IS 1
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 FIDRES 0.147183 Hz
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 SI 0.03000000 sec

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 SFO1 75.4762004 MHz

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 PL2 120.00 dB
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f2 Processing parameters
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 SF 1.00
 SFO1 0
 GB 1.00 Hz
 GB 0
 GC 1.40

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 F2P -10.000 ppm
 F2 -754.88 Hz
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 FZCM 684.30334 Hz/cm

ppm 200 180 160 140 120 100 80 60 40 20 0

Current Data Parameters
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PROCNO 1

F2 - Acquisition Parameters
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PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 25
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 514.7
RW 52.400 usec
DE 8.00 usec
TE 300.0 K
D1 1.00000000 sec

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F2 - Processing parameters
SI 32768
SF 400.132000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
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CY 12.83 cm
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F1 4001.30 MHz
F2P -1.000 ppm
F2 -1006.13 Hz
PPHCH 0.44000 ppm/cm
HZCN 176.05721 Hz/cm

ppm
7.3546
7.3442
7.3325
7.3256
7.3185
7.3084
7.2947
7.2870
7.2776
7.2672
7.2542

Current Data Parameters
NAME 374-C
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
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INSTRUM spect
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PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 33
DS 0
SWH 25125.629 Hz
FIDRES 0.766773 Hz
AQ 0.6521332 sec
RG 2580.3
RW 19.900 usec
DE 8.00 usec
TE 300.0 K
D1 3.00000000 sec
d11 0.03000000 sec

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NUC1 13C
P1 9.10 usec
PL1 -5.00 dB
SFO1 100.6236364 MHz

***** CHANNEL f2 *****
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NUC2 1H
PCPD2 80.00 usec
PL2 -4.00 dB
PL12 12.90 dB
SFO2 400.1320003 MHz

F2 - Processing parameters
SI 32768
SF 100.6127883 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 25.00 cm
CY 20.87 cm
F1P 200.000 ppm
F1 20134.81 MHz
F2P -10.000 ppm
F2 -1006.13 Hz
PPHCH 9.20000 ppm/cm
HZCN 505.63763 Hz/cm

ppm
171.269

135.767
133.808
129.203
128.473
128.430
128.105
128.017
127.022

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77.003
76.682

66.463
41.224

Current Data Parameters
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 PROCNO 1

F2 - Acquisition Parameters
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 PROBR0 DUAL 5mm
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 10
 DS 2
 SWH 4496.403 Hz
 FIDRES 0.137219 Hz
 AQ 3.6438515 sec
 RG 362
 DM 111.200 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

***** CHANNEL f1 *****
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 PL1 -6.00 dB
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F2 - Processing parameters
 SI 32768
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 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
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 F2P -1.000 ppm
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Current Data Parameters
 NAME 444
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20051201
 Time 9.47
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 PROBR0 DUAL 5mm
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 10
 DS 2
 SWH 4496.403 Hz
 FIDRES 0.137219 Hz
 AQ 3.6438515 sec
 RG 362
 DM 111.200 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

***** CHANNEL f1 *****
 NUC1 13C
 P1 7.00 usec
 PL1 -6.00 dB
 SFO1 75.4762004 MHz

***** CHANNEL f2 *****
 NUC2 1H
 P2 100.00 usec
 PL2 120.00 dB
 SFO2 300.1310005 MHz

F2 - Processing parameters
 SI 32768
 SF 75.4677508 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
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 F1P 200.000 ppm
 F1 16662.91 Hz
 F2P -10.000 ppm
 F2 -75.468 Hz
 PPMCH 9.20000 ppm/cm
 HZCH 694.30334 Hz/cm