Tandem Pd (II) Catalyzed Vinyl Ether Exchange - Claisen Rearrangement as a Facile Approach to γ,δ - Unsaturated Aldehydes

Xudong Wei,* Jon Lorenz, Suresh Kapadia, Anjan Saha, Nizar Haddad, Carl A.

Busacca and Chris H. Senanayake

Department of Chemical Development, Boehringer Ingelheim Pharmaceuticals, Inc.,
900 Ridgebury Road, Ridgefield, CT 06877

xwei@rdg.boehringer-ingelheim.com

Supporting Information

Table of Contents

Table of compounds and references	S1-S3
Data of new compounds	S3
Copies of NMR spectra	S4-S41

Table 1 List of compounds and references

No.	Product	References
3a		Cresson, P. <i>Bull. Soc. Chim. Fr.</i> 1964 ; 2629-2635. Gajewski, J. J.; Conrad, N. D.; <i>J. Am. Chem. Soc.</i> 1979 , <i>101</i> , 6693-6704.
3b		Harkes, P. D.; <i>Recl. Trav. Chim. Pays-Bas</i> , 1966 , <i>85</i> , 757-764. Clive, D. J.; Murthy, K. S. K.; George, R.; Poznansky, M. J. <i>J. Chem. Soc. Perkin Trans. 1</i> , 1990 , (7), 2099-2108.
3c		Collins, I.; Nadin, A.; Holmes, A. B.; Long, M. E.; Man, J.; Baker, Raymond; <i>J. Chem. Soc. Perkin Trans. 1</i> , 1994 , <i>16</i> , 2205-2216.
3d		Laird, T.; Ollis, W. D.; Sutherland, I. O. <i>J. Chem. Soc. Perkin Trans. 1</i> , 1980 , 1477-1486.
3e		Padwa, A.; Kulkarni, Y. S.; Zhang, Z. <i>J. Org. Chem.</i> , 1990 , <i>55</i> , 4144-4153.

3f		Crandall, J. K.; Mayer, C. F.; <i>J. Org. Chem.</i> 1970 , <i>35</i> , 3049-3053. Laroack, R. C.; Oertle, K.; Potter, G. F. <i>J. Am. Chem. Soc.</i> 1980 , <i>102</i> , 190-197.
3g		Ryu, I.; Kusano, K.; Ogawa, A.; Kambe, N.; Sonoda, N.; <i>J. Am. Chem. Soc.</i> , 1990 , <i>112</i> , 1295-1297. Vig, O. P.; Sharma, M. L.; Gakhar, M.; Malik, N.; <i>Indian J. Chem. Sect. B</i> ; 1980 , <i>19B</i> , 356-357.
3h		Jacobson <i>J. Am. Chem. Soc.</i> 1950 , <i>72</i> , 1489-1491. Bestman, H. J.; Koschatzky, K. H.; Schaetzke, W.; Suess, J.; Vostrowsky, O. <i>Liebigs Ann. Chem.</i> 1981 , (8), 1705-1720.
3i		Morizur, J.-P. <i>et al.</i> ; <i>Bull. Soc. Chim. Fr.</i> , 1970 , 1959-1967.
3j		Awai, N.; Kuwahara, S.; Kodama, O.; Santisopasri, V. <i>Biosci. Bioelectron. Biochem.</i> 1995 , <i>10</i> , 1999-2000.
3k		Burgstahler, A. W.; <i>J. Am. Chem. Soc.</i> 1960 , <i>82</i> , 4681-4685.
3l		Khanjin, N. A.; Snyder, J. P.; Menger, F. M. <i>J. Am. Chem. Soc.</i> 1999 , <i>121</i> , 11831-11846. Cossy, J.; Tresnard, L.; Pardo, D. G. <i>Eur. J. Org. Chem.</i> , 1999 , 1925-1934.
3m		Burgstahler, A. W.; Nordin, I. C. <i>J. Am. Chem. Soc.</i> 1961 , <i>83</i> , 198-206. Agosta, W. C.; Wolff, S. <i>J. Am. Chem. Soc.</i> 1976 , <i>98</i> , 4182-4188. Büchi, G.; Vogel, D. E. <i>J. Org. Chem.</i> 1983 , <i>48</i> (26) 5406-5408.
3n		Takahashi, S.; Oritani, T.; Kuzuhara, H.; Yamashita, K. <i>Agric. Biol. Chem.</i> 1991 , <i>55</i> , 891-892.
2o		Grieco, P. A.; Clark, J. D.; Jagoe, C. T. <i>J. Am. Chem. Soc.</i> 1991 , <i>113</i> (14), 5488-5489. Reetz, M. T.; Gansaeuer, A. <i>Tetrahedron</i> , 1993 , <i>49</i> (23), 6025-6030.
3p		Nathan J.; Gajewski, J. J. <i>J. Am. Chem. Soc.</i> 1994 , <i>116</i> , 6121-6129.
3q		Baekstroem, P.; Li, L.; <i>Tetrahedron</i> , 1991 , <i>32</i> , 6521-6532. Jin, Q.; Coates, R. M. <i>Collect. Czech. Chem. Commun.</i> 2002 , <i>67</i> , 55-57.
Pre-4	Used as crude, not isolated	Jin, Q.; Coates, R. M. <i>Collect. Czech. Chem. Commun.</i> 2002 , <i>67</i> , 55-74.

4		New compound.
Pre-5		New compound.
5		New compound.

Characterization data of new compounds:

Compound 4: Obtained as colorless oil in 70% yield by silica gel column chromatography eluted with hexanes. ^1H NMR (400 MHz, CDCl_3): 1.60 (3H, s), 1.70 (3H, s), 1.98-2.05 (4H, m), 2.34 (2H, q, $J = 7.2$ Hz), 2.50 (2H, dt, $J = 1.0, 7.2$ Hz), 5.07-5.13 (1H, m), 5.35-5.52 (2H, m), 9.77 (1H, t, $J = 1.0$ Hz) ppm; ^{13}C NMR (100 MHz, CDCl_3): 17.7, 25.2, 25.7, 28.0, 32.7, 43.5, 123.9, 127.9, 131.8, 132.0, 202.5 ppm. HRMS m/z : 166.1355 (M^+) (Calcd, 166.1358).

Compound Pre-5: Obtained as colorless oil in 95% yield by silica gel column chromatography eluted with hexanes-EtOAc (95:5 to 80:20). ^1H NMR (400 MHz, CDCl_3): 1.49-1.56 (2H, m), 1.53 (3H, s), 1.62 (3H, s), 1.92-1.97 (4H, m), 1.97-2.06 (2H, m), 4.05 (1H, q, $J = 6.0$ Hz), 5.01-5.06 (2H, m), 5.16 (1H, dt, $J = 17.0, 1.2$ Hz), 5.31-5.42 (2H, m), 5.80 (1H, ddd, $J = 6.0, 10.4, 17.0$ Hz) ppm; ^{13}C NMR (100 MHz, CDCl_3): 17.8, 25.7, 28.1, 28.5, 32.8, 36.7, 72.7, 114.6, 124.1, 129.7, 130.9, 131.7, 141.1 ppm. HRMS m/z : 194.1667 (M^+) (Calcd, 194.1671).

Compound 5: Obtained as colorless oil in 72% yield by silica gel column chromatography eluted with hexanes. ^1H NMR (400 MHz, CDCl_3): 1.53 (3H, s), 1.62 (3H, s), 1.92-1.99 (8H, m), 2.26 (2H, q, $J = 7.2$ Hz), 2.42 (2H, dt, $J = 1.0, 7.2$ Hz), 5.01-5.06 (1H, m), 5.26-5.45 (4H, m), 9.70 (1H, t, $J = 1.0$ Hz) ppm; ^{13}C NMR (100 MHz, CDCl_3): 17.7, 25.2, 25.7, 28.2, 32.5, 32.6, 32.8, 43.5, 124.1, 128.0, 129.6, 130.5, 131.4, 131.6, 202.4 ppm. HRMS m/z : 220.1824 (M^+) (Calcd, 220.1825).

Copies of NMR spectra for all of the new and known compounds.

7717-038-c
NMR_1H_32_scans CDCl3 opt/xwinmr guest 49

Current Data Parameters
NAME Sep06-2006
EXPNO 460
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060906
Time 18.54

INSTRUM spect

PROBHD 5 mm QNP 1H/13

PULPROG zg30

TD 65536

SOLVENT CDCl3

NS 32

DS 0

SWH 8278.146 Hz

FIDRES 0.126314 Hz

AQ 3.9584243 sec

RG 128

DW 60.400 usec

DE 6.00 usec

TE 297.2 K

TDI 1.5000000 sec

MCREST 0.0000000 sec

MCNMR 0.0150000 sec

===== CHANNEL f1 =====

NUC1 1H

P1 12.00 usec

PL1 0.00 dB

SFO1 400.1324710 MHz

F2 - Processing parameters

SI 32768

SP 400.1300028 MHz

WDW EM

SSB 0

LB 0.30 Hz

GB 0

PC 1.00

7298-89-b
NMR_1H_16_scans CDCl3 opt/xwinnmr guest 31

Current Data Parameters
NAME Mar10-2006
EXPNO 400
PROCNO 1

F2 - Acquisition Parameters

Date_ 20060310

Time 18.09

INSTRUM spect

PROBHD 5 mm QNP 1H/13

PULPROG zg30

TD 65536

SOLVENT CDCl3

NS 16

DS 0

SWH 8278.146 Hz

FIDRES 0.126314 Hz

AQ 3.9584243 sec

RG 322.5

TM 60.400 usec

DE 6.00 usec

TE 326.2 K

D1 1.50000000 sec

MCREST 0.00000000 sec

MCWRR 0.01500000 sec

===== CHANNEL f1 =====

NUC1 1H

PL 12.00 usec

PL1 0.00 dB

SFO1 400.1324710 MHz

F2 - Processing parameters

SI 32768

SF 400.130078 MHz

WDW EM

SSB 0

LB 0.30 Hz

GB 0

PC 1.00

8215-b-aldehyde

9.729
9.722
9.714

5.953
5.925
5.910
5.882

5.043
5.015
4.999

2.349
2.342

1.157

Current Data Parameters
NAME Oct11-2006
EXPNO 340
PROCNO 1

F2 - Acquisition Parameters
Date_ 20061011
Time 16.12

INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0

SWH 8278.146 Hz
FIDRES 0.252629 Hz
AQ 1.9792372 sec
RG 50.8

DW 60.400 usec
DE 6.00 usec
TE 295.2 K
D1 1.50000000 sec
MCREST 0.00000000 sec
MCMRK 0.01500000 sec

CHANNEL f1
NUC1 1H
P1 9.06 usec
PL1 -2.00 dB
SFO1 400.3332026 MHz

F2 - Processing parameters
SI 32768
SF 400.329981 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

8215-b-aldehyde

7298-102-p3

Current Data Parameters
 NAME Nov18-2005
 EXPNO 260
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20051118
 Time 15.17

INSTRUM spect
 PROBD 5 mm BBO BB-1H
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3

NS 16

DS 0

SWH 8278.146 Hz

FIDRES 0.252629 Hz

AQ 1.9792372 sec

RG 57

DW 60.400 usec

DE 6.00 usec

TE 295.2 K

DL 1.50000000 sec

MCRES* 0.00000000 sec

MCWRK 0.01500000 sec

===== CHANNEL f1 =====

NUC1 1H

P1 9.06 usec

PL -2.00 dB

SFO1 400.3332026 MHz

F2 - Processing Parameters

SI 32768

SF 400.3300005 MHz

WDW EM

SSB 0

LB 0.30 Hz

GB 0

PC 1.00

8215-7-p2
NMR_1H_32_scans CDCl3 opt/xwinmr guest 12

Current Data Parameters
NAME Sep27-2006
EXPNO 230
PROCNO 1

F2 - Acquisition Parameters

Date_ 20060927
Time 16.59
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 0
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 512
DE 60.400 usec
DM 6.00 usec
TE 297.2 K
D1 1.50000000 sec
MCREST 0.00000000 sec
MCWRR 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing Parameters
SI 32768
SF 400.1300076 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

8215-2-p1
NMR_1H_32_scans CDCl3 opt/xwinmr guest 15

Current Data Parameters
NAME Sep11-2006
EXPNO 171
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060912
Time 1.30
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 161.3
RW 20.850 usec
DE 6.00 usec
TE 298.2 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRR 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 9.50 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPCPRG2 waltz16
NUC2 1H
PCPD2 85.00 usec
PL2 -0.00 dB
PL12 17.00 dB
PL13 10.79 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

7298-107-p4
NMR_1H_32_scans CDCl3 opt/xwinmr guest 51

Current Data Parameters
NAME Nov23-2005
EXPNO 270
PROCNO 1

F2 - Acquisition Parameters
Date_ 20051123
Time 18.31
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
FIDRES 0.126314 Hz
RG 322.5
DE 60.400 usec
TE 297.2 K
D1 1.50000000 sec
MCREST 0.00000000 sec
MCNMRK 0.01500000 sec

==== CHANNEL f1 =====
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300062 MHz
WDW EM
SSB 0
GB 0
PC 1.00

Current Data Parameters	
NAME	Jan20-2006
EXPNO	290
PROCNO	1

```
F2 - Acquisition Parameters
Date_      20060120
Time       15.08
```

INSTRUM		spect
PROBHD	5 mm	QNP 1H/13
PULPROG		zg30
TD		65536
SOLVENT		CDCl3
NS		32

MS 2
DS 0

SWH	82/8.146 HZ
FIDRES	0.126314 HZ

AQ	3.9584243 sec
RG	57

DW	60.400 use
DE	6.00 use

```
TE      297.2 K
D1      1.50000000 sec
```

MCREST	0.00000000 sec
MCNRK	0.01500000 sec

CHANNEL, F1

NUC1	1H
P1	12 00 use

PL1	400 1334710 MHz
PL1	0.00 dB
PL1	44.00 dB

400.1324/10 PWZ

P2 - Processing parameters	32768
SI	

SP 400.1300000 MHz
WDM FM

SSB 0
LB 0.30 Hz

GB
PC

3

3

Current Data Parameters
 NAME Jan20-2006
 EXPNO 291
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20060120
 Time 15.38
 INSTRUM spect
 PROBD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 512
 DS 2
 SWH 23980.814 Hz
 FIDRES 0.365918 Hz
 AQ 1.3664756 sec
 RG 143.7
 DW 20.850 usec
 DE 6.00 usec
 TE 298.2 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 MCREST 0.00000000 sec
 MCWRR 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 9.50 usec
 PL1 -2.00 dB
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 85.00 usec
 PL2 -0.00 dB
 PL12 17.00 dB
 PL13 10.79 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127690 MHz
 EM
 SSB 0
 LB 3.00 Hz
 GB 0
 PC 1.40

7298-132-product1
NMR_1H_16_scans CDCl3 opt/xwinmr guest 78

E:Z = 8:1

Current Data Parameters
NAME Jan18-2006
EXPNO 430
PROCNO 1

F2 - Acquisition Parameters

Date_ 20060118
Time 19.04
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0

SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec

RG 128
RW 60.400 usec
DE 6.00 usec

TE 297.2 K
DI 1.50000000 sec
MCREST 0.00000000 sec

MCWRR 0.01500000 sec

===== CHANNEL f1 =====

NUC1 1H
P1 12.00 usec
PL1 0.00 dB

SFO1 400.1324710 MHz

F2 - Processing parameters

SI 32768
SF 400.1300044 MHz

WDW EM
SSB 0
LB 0.30 Hz

GB 0
PC 1.00

7298-151-p
NMR_1H_32_scans CDCl3 opt/xwinmr guest 30

Current Data Parameters
NAME Mar07-2006
EXPTNO 350
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060307
Time 18.00

INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 0

SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 101.6

DW 60.400 usec
DE 6.00 usec
TE 297.2 K
DL 1.50000000 sec
MCPRST 0.00000000 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300016 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

7298-151-p
NMR 13C_512_scans CDCl3 opt 30

Current Data Parameters
NAME Mar07-2006
EXENO 351
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060308
Time_ 7.54

INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4000
DS 2
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 114
RG 114
TDW 20.850 usec
DE 6.00 usec
TE 298.2 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCMRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 9.50 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 85.00 usec
PL2 -0.00 dB
PL12 17.00 dB
PL13 10.79 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

7712-101f2

Current Data Parameters
 NAME 7712-101f2
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060203
 Time_ 17.57

INSTRUM spect
 PROBD 5 mm BBI 1H-BB
 PULPROG zg30

TD 32768
 SOLVENT CDCl3

NS 16
 DS 4

SWH 7374.631 Hz
 FIDRES 0.225056 Hz

RG 35.9
 ACQ 2.2217205 sec

DW 67.800 usec
 DE 4.50 usec

TE 303.0 K
 D1 2.00000000 sec

===== CHANNEL f1 =====
 NUCL 1H

P1 7.40 usec
 PL1 -3.50 dB

SFO1 500.1334009 MHz

F2 - Processing parameters
 SI 65536

SF 500.1300087 MHz
 WDW EM

SSB 0
 LB 0.05 Hz

GB 0
 PC 1.00

7712-101-f2

202.23

129.76
128.39

50.01

30.08
28.94
24.83
20.94

Current Data Parameters
NAME 7712-101f2
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060203
Time_ 20.16

INSTRUM spect
PROBHD 5 mm BBI 1H-BB
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 2048

DS 8
SWH 30303.031 Hz
FIDRES 0.924775 Hz
AQ 0.5407220 sec

RG 812.7
DE 16.500 usec
TE 303.0 K
D1 0.50000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

CHANNEL f1
NUC1 13C
P1 12.00 usec
PL1 -5.50 dB
SFO1 125.772011 MHz

CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
P12 -3.50 dB
PL12 16.02 dB
PL13 120.00 dB
SFO2 500.1324000 MHz

F2 - Processing parameters
SI 65536
SF 125.7577941 MHz
SSB 0
EM 0
LB 1.00 Hz
GB 0
PC 1.00

8037-056-4

Current Data Parameters
 NAME Nov05-2006
 EXENO 60
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20061105
 Time_ 17.34
 INSTRUM spect
 PROBD 5 mm BBO BB-1H
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 2048
 DS 4
 SWH 23980.814 Hz
 FIDRES 0.365918 Hz
 AQ 1.3664796 sec
 RG 8192
 DW 20.850 usec
 DE 6.00 usec
 TE 295.2 K
 D1 0.50000000 sec
 d11 0.03000000 sec
 MCREST 0.00000000 sec
 MCRRK 0.01500000 sec

CHANNEL f1

NUC1 13C
 P1 8.10 usec
 PL1 -3.00 dB
 SFO1 100.6731249 MHz

CHANNEL f2

CEPDERG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 -2.00 dB
 PL12 18.86 dB
 PL13 18.86 dB
 SFO2 400.3316013 MHz

F2 - Processing parameters

SI 32768
 SF 100.6630590 MHz
 MDW EM
 SSB 0
 LB 3.00 Hz
 GB 0
 PR 1.40

8037-058-3
NMR_13C_512_scans CDCl3 opt/xwinmr guest 18

8037-057
NMR_13C_512_scans CDCl3 opt/xwinmr guest 77

Current Data Parameters
NAME Oct31-2006
EXNO 320
PROCNO 1

F2 - Acquisition Parameters
Date_ 20061031
Time_ 20.06
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 2
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664796 sec
RG 161.3
DW 20.890 usec
DE 6.00 usec
TE 298.2 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

CHANNEL F1 13C
NUC1 13C
P1 9.50 usec
PL1 -2.00 dB
SFO1 100.622828 MHz

CHANNEL F2 1H
CPDPRG2 waltz16
NUC2 1H
PCPD2 85.00 usec
PL2 0.00 dB
PL12 17.00 dB
PL13 10.79 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127704 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

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

7298-104-p3
 NMR_1H_16_scans CDCl3 opt/xwinmr guest 105

Current Data Parameters
 NAME Nov18-2005
 EXPNO 360
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20051118
 Time 18.21

INSTRUM spect
 PROBRD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 16

DS 0
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec

RG 101.6
 DW 60.400 usec
 DE 6.00 usec
 TE 297.2 K
 D1 1.50000000 sec
 MCREST 0.00000000 sec
 MCNMRK 0.01500000 sec

===== CHANNEL f1 =====
 NUCL 1H
 P1 12.00 usec
 PL1 0.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.130022 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

7298-104-p3
NMR_13C_512_scans CDC13 opt/skwi1nmr guest 105

Current Data Parameters
NAME Nov18-2005
EXPNO 361
PROCNO 1

P2 - Acquisition Parameters
Date_ 20051118
Time 20.15

INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2000

DS 2
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 181

DW 20.850 usec
DE 6.00 usec
TE 298.2 K

DI 2.00000000 sec
d11 0.03000000 sec
MCRESST 0.00000000 sec
MCWRRK 0.01500000 sec

===== CHANNEL f1 =====

NUC1 13C
P1 9.50 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16
NUC2 1H
PCPD2 85.00 usec
PL2 -0.00 dB
PL12 17.00 dB
PL13 10.79 dB
SFO2 400.1316005 MHz

P2 - Processing Parameters

SI 32768
SF 100.6127650 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

7298-123-p2
NMR_1H_32_scans CDCl3 opt/xwinmr guest 3

E: Z ~ 60:40

Current Data Parameters
NAME Sep05-2006
EXPNO 300
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060905
Time 19.28

INSTRUM spect

PROBHD 5 mm QNP 1H/13

PULPROG zg30

TD 65536

SOLVENT CDCl3

NS 32

DS 0

SWH 8278.146 Hz

FIDRES 0.126314 Hz

AQ 3.9584243 sec

RG 322.5

DW 60.400 usec

DE 6.00 usec

TE 297.2 K

D1 1.50000000 sec

MCREST 0.00000000 sec

MCWPRK 0.01500000 sec

===== CHANNEL f1 =====

NUC1 1H

P1 12.00 usec

PL1 0.00 dB

SFO1 400.1324710 MHz

F2 - Processing parameters

SI 32768

SF 400.130076 MHz

WDW EM

SSB 0

LB 0.30 Hz

GB 0

PC 1.00

$$\begin{array}{r} 202 \\ 202 \\ \hline \end{array}$$

136
136
131
131
124
124
122
122

- 77.
 - 77.
 - 76.

Current Data Parameters	
NAME	Sep05-2006
EXPNO	301
PROCNO	1

```
F2 - Acquisition Parameters
Date_      20060905
Time       22.19
```

INSTRUM	5 mm QNP	spect
PROBHD	1H/13	
NUC1	13C	
NUC2	1H	
PROBHD	1H/13	
NUC1	13C	
NUC2	1H	

POLYMER	29P950
TD	65536
SOLVENT	CDCl3
	3000

NS	3000
DS	2
SWH	23980.814 Hz

EIDRES	0.365918 Hz
AQ	1.3664756 sec
RG	161.3

DW	20.850 use
DE	6.00 use
TE	298.2 K

D1	2.00000000 sec
d11	0.03000000 sec
MCREST	0.00000000 sec

```
MCWRK      0.01500000 sec
```

NUC1	13C
P1	9.50 use
ex 1	3.00 ap

PLI	-2.00	GD
SFO1	100.6228298	MR:

```
===== CHANNEL 12 =====
CPDPRG2      waltz16
NUC2         1H
```

PCPD2	85.00	use
PL2	-0.00	dB
PL12	17.00	dB

PL13	10.79 dB
SFO2	400.1316005 MHz

F2 - Processing parameters	
SI	32768
SP	100-6127690 MHz

	EM
WDW	0
SSB	0
SSB	0

LB	2.00 m
GB	0
PC	1.40

7662101-1

Current Data Parameters
 NAME Dec20-2005
 EXPNO 120
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20051220
 Time 14.34
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 32
 DS 0
 SWH 8278.146 Hz
 FIDRES 0.252629 Hz
 AQ 1.9792372 sec
 RG 50.8
 IAW 60.400 usec
 DE 6.00 usec
 TE 294.2 K
 DI 1.50000000 sec
 MCREST 0.00000000 sec
 MCMRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 9.06 usec
 PL1 -2.00 dB
 SFO1 400.3332026 MHz

F2 - Processing parameters
 SI 32768
 SF 400.3300000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

```
F2 - Acquisition Parameters
Date_      20051220
Time       15.55
```

	spec	5 mm BBO	RB-1H
INSTRUM			
PROBHD			
FULPROG			zppg30
TD			62536
SOLVENT			CDCl3
NS			2048
DS			4
SWH		23980.814	Hz
FTIDRES		0.365918	Hz
AQ		1.3664756	sec
RG		13004	
DW		20.850	usec
DE		6.00	usec
TE		254.2	K
DI1		0.50000000	sec
d11		0.03000000	sec
MCREST		0.00000000	sec
MCWRST		0.01500000	sec

```
===== CHANNEL F1 =====
NUC1      13C
P1         8.10 usec
PL1        -3.00 dB
SFO1      100.6731249 MHz
```

```
===== CHANNEL 12 =====
CPDRG2      waltz16
NUC2         1H
PCPD2        100.00 usec
PL2          -2.00 dB
PL12         18.86 dB
PL13         18.86 dB
SFO2        400.3316013 MHz
```

I2 -	Processing Parameters
SI	32768
SF	100.6630590 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

7662113-2
NMR_1H_32_scans CDCl3 opt/xwinmr guest 56

Current Data Parameters
NAME Jan23-2006
EXPNO 140
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060123
Time 13.38

INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 0
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 322.5
RW 60.400 usec
DE 6.00 usec
TE 297.2 K
D1 1.50000000 sec
MCREST 0.00000000 sec
MCWRR 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300366 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

7662113-2
NMR_13C_512_scans CDCl₃ 59

Current Data Parameters
NAME Jan23-2006
EXPNO 170
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060123
Time 15.12

INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl₃
NS 1024

DS 2
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 114

DE 20.850 usec
TE 6.00 usec
T1 298.2 K
d11 2.00000000 sec
d11 0.03000000 sec
d11 0.00000000 sec
MCWRR 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 9.50 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 85.00 usec
PL2 -0.00 dB
PL12 17.00 dB
PL13 10.79 dB
SFO2 400.1316005 MHz

7662127-3
NMR_1H_64_scans CDCl3 opt/xwinnmr guest 35

Current Data Parameters
NAME Feb15-2006
EXPNO 80
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060215
Time 10.08

INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 0

SWH 8278.146 Hz
FIDRES 0.12614 Hz
AQ 3.9584243 sec
RG 203.2

DW 60.400 usec
DE 6.00 usec
TE 297.2 K
DI 1.50000000 sec
HCREST 0.00000000 sec
MCMRK 0.01500000 sec

===== CHANNEL f1 =====

NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters

SI 32768
SF 400.1300357 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00