

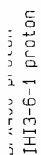
**Diastereoselective Synthesis of α,β' -Disubstituted
Aminomethyl(2-carboxyethyl)phosphinates as Phosphinyl
Dipeptide Isosteres**

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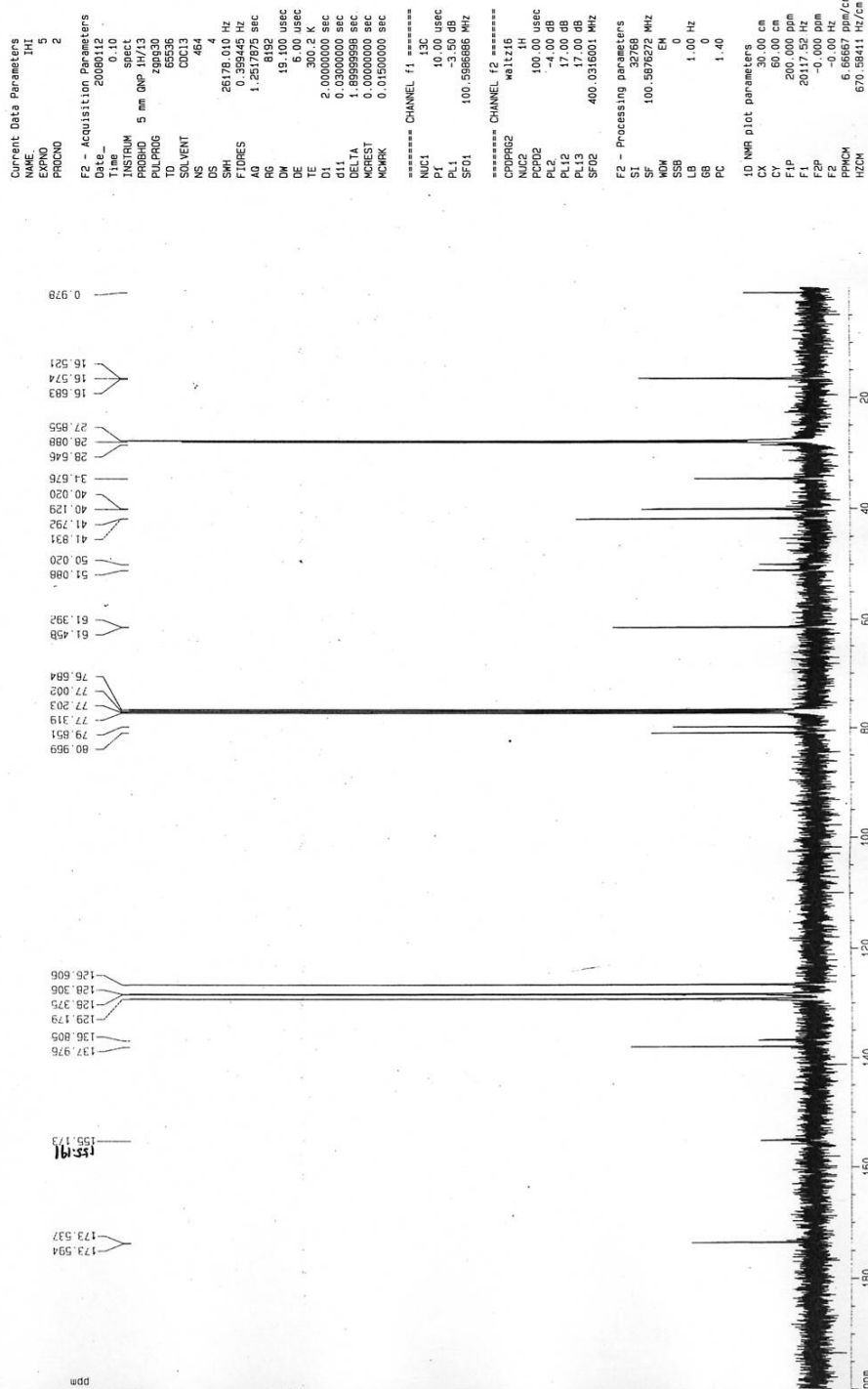
Email: yokomatsu@ps.toyaku.ac.jp

Supporting Information-Part IV: Spectral Data





DPX400 carbon
IH13-6-1 carbon

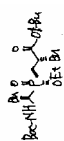


¹³C NMR of 20a

IH13-6-1 phosphorus

91.3566

wdw



Current Data Parameters
NAME IH1
EXPNO 1
PROCNO 3

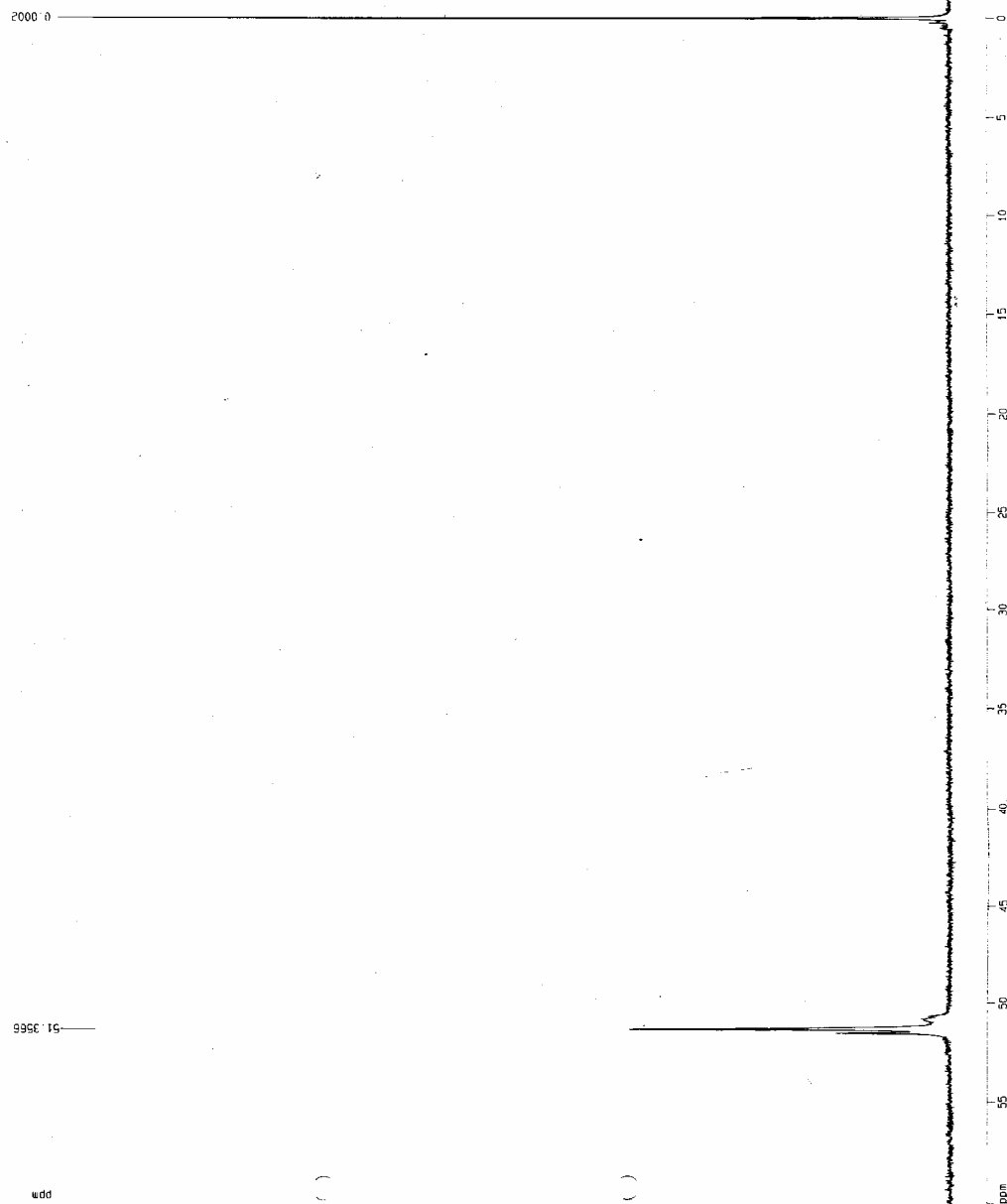
F2 - Acquisition Parameters
Date_ 20001111
Time 20:05
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT D2O
NS 64
DS 2
SWH 64335.065 Hz
FIDRES 0.990830 Hz
AQ 0.5046772 sec
RG 9195.2
DM 7.700 usec
DE 5.00 usec
TE 300.2 K
D1 2.0000000 sec
d11 0.0300000 sec
DELTA 1.8999999 sec
ACQRES 0.0000000 sec
MCHRG 0.0150000 sec

***** CHANNEL f1 *****
NUC1 31P
P1 6.50 usec
PL1 -4.00 dB
SFO1 161.9350920 MHz

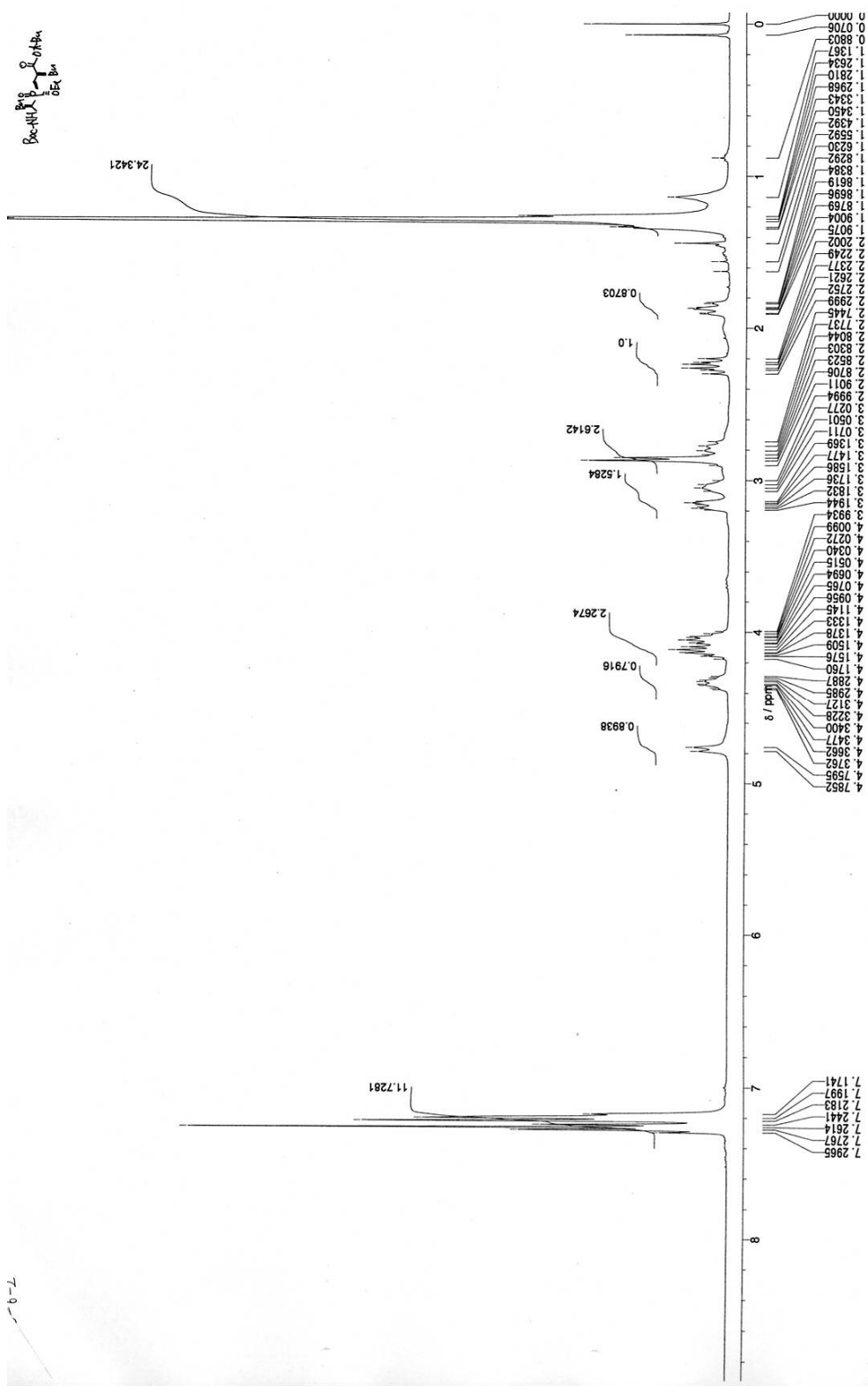
***** CHANNEL f2 *****
CPDPRG2 Waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 400.0316001 MHz

F2 - Processing parameters
SI 32768
SF 161.9350920 MHz
WDW EM
SSB 0
B 1.00 Hz
S2 0
PC 1.40

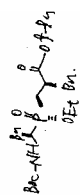
1D NMR plot parameters
CX 32.00 cm
CY 100.00 cm
F1P 60.000 ppm
F1 9716.10 Hz
F2P -1.000 ppm
F2 -161.93 Hz
PPHCH 1.90625 ppm/cm
HZCM 308.68650 Hz/cm



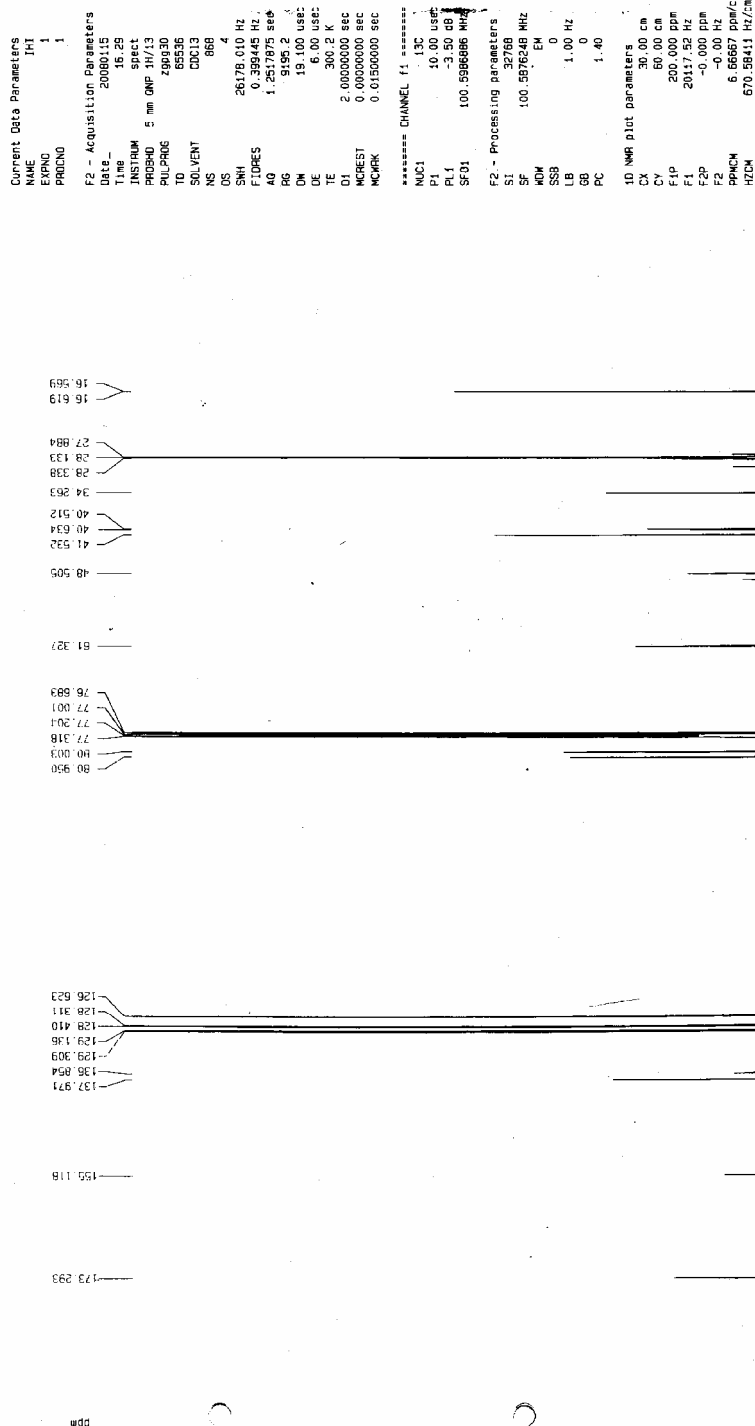
³¹P NMR of 20a



¹H NMR of 20b



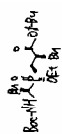
DPX400 carbon
IH13-6-2 carbon



¹³C NMR of 20b

1H13-6-2 phosphorus

ppm 52.5498



Current Data Parameters
 NMR 1H1
 EPRNO 2
 PROCNO 1

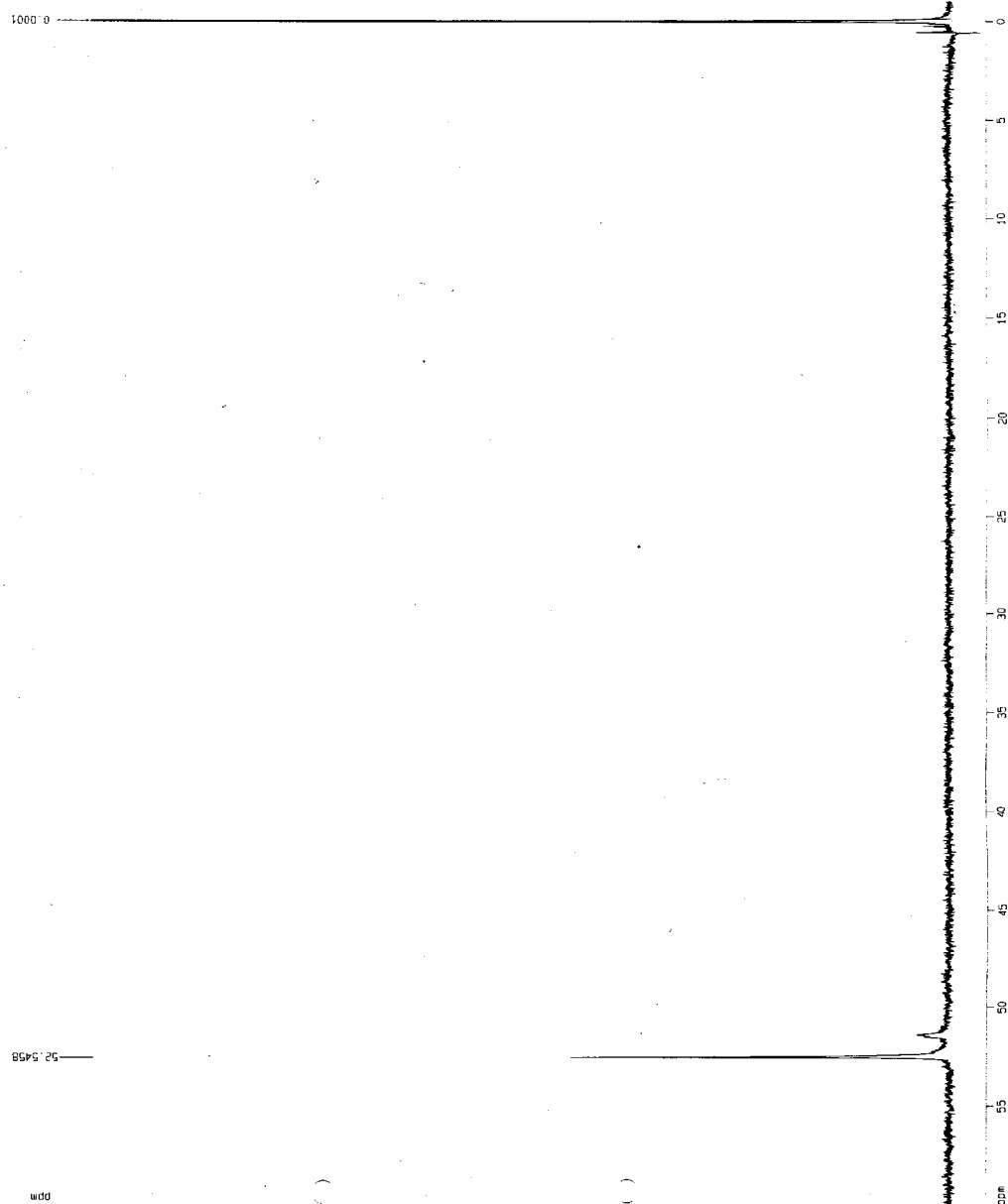
F2 - Acquisition Parameters
 Date_ 20080111
 Time_ 20.11
 INSTRUM spect
 PROBHD 5 mm GNP 1H/13
 PULPROG zgpg30
 TO 65536
 SOLVENT CDCl3
 NS 64
 DS 2
 SWH 64935.065 Hz
 FIDRES 0.990830 Hz
 AQ 0.5046772 sec
 RG 5195.2
 DM 7.700 usec
 DE 6.00 usec
 TE 300.2 K
 D1 2.0000000 sec
 d11 0.0300000 sec
 DELTA 1.6899999 sec
 ACQST 0.0000000 sec
 ACWPR 0.0150000 sec

===== CHANNEL f1 =====
 NUC1 31P
 P1 6.30 usec
 PL1 -4.00 dB
 SF01 161.9250320 MHz

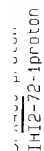
===== CHANNEL f2 =====
 OPBPG2 waltz16
 NUC2 1H
 P2 100.00 usec
 PL2 -4.00 dB
 PL12 17.00 dB
 PL13 17.00 dB
 SF02 400.0316001 MHz

F2 - Processing parameters
 SI 32768
 SF 161.9250320 MHz
 NQW 0
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

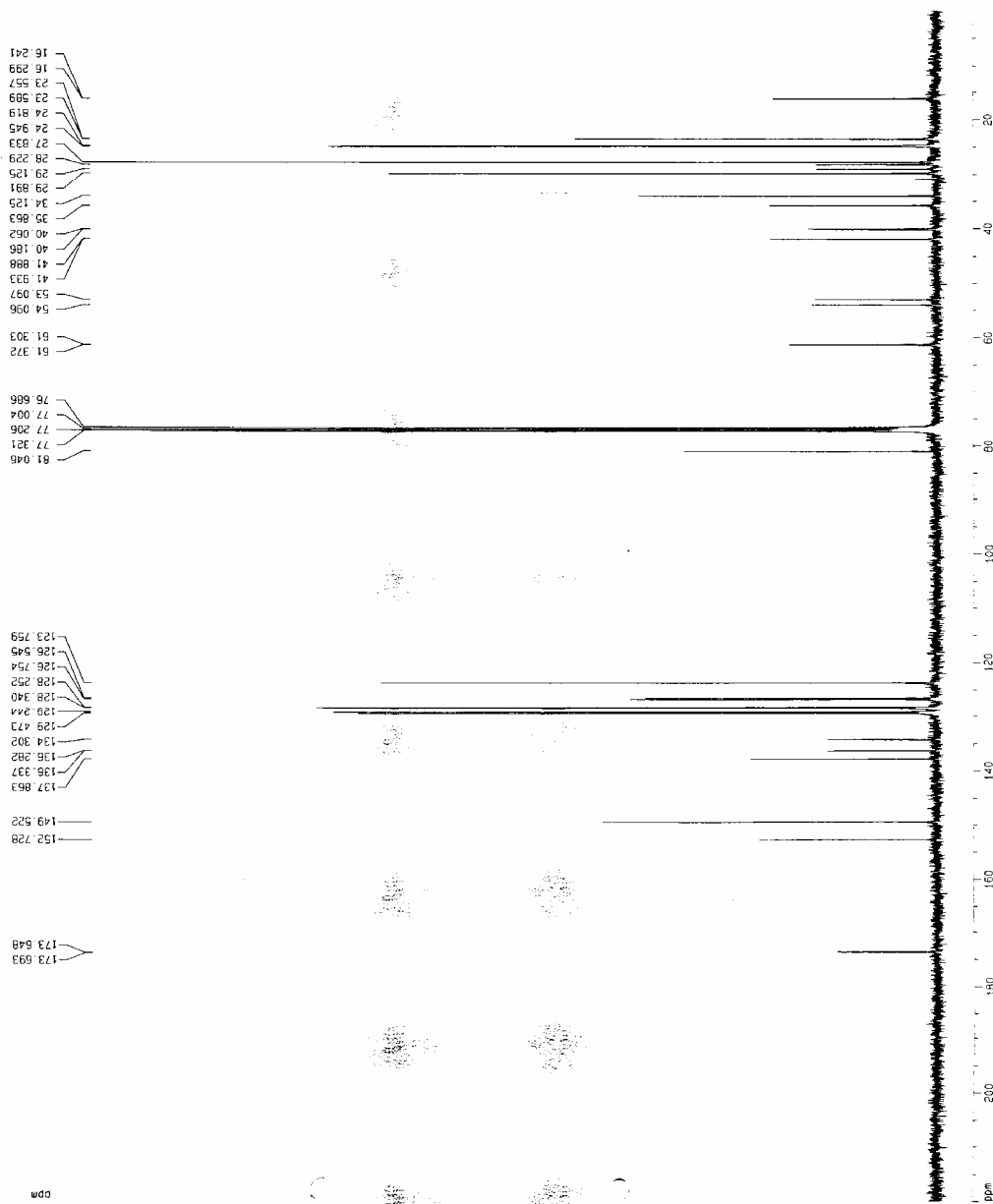
10 NMR plot parameters
 CX 32.00 cm
 CY 277.23 cm
 FIP 50.000 ppm
 F1 9716.10 Hz
 F2 -1.000 ppm
 F2 -161.83 Hz
 PPMCM 1.90625 ppm/cm
 HZCM 308.68860 Hz/cm



³¹P NMR of 20b



DPX400 carbon
341.2-72.1



Current Data Parameters
NAME: 1H
EXPNO: 1
PROCNO: 2

F2 - Acquisition Parameters
Date_: 20070703
Time: 21.29
INSTRUM: spect
PROBHD: 5 mm GNP 1H/13
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 1024
DS: 2
SWH: 26178.010 MHz
FIDRES: 0.390445 Hz
AQ: 1.2857875 sec
RG: 3251
DM: 19.100 usec
DE: 5.00 usec
TE: 300.2 K
CNS12: 145.000000
C1: 2.0000000 sec
C2: 0.0034629 sec
d12: 0.0002000 sec
d12: 0.0001019 sec
K1RES1: 0.0000000 sec
K1WRK: 0.0100000 sec

===== CHANNEL f1 =====
NUC1: 13C
P1: 8.00 usec
D2: 15.00 usec
PL1: -5.00 dB
SFO1: 100.6258401 MHz

===== CHANNEL f2 =====
CPDPRG2: waltz16
NUC2: 1H
P3: 7.40 usec
P4: 14.80 usec
P5: 100.00 usec
PL2: -5.00 dB
PL3: -5.00 dB
PL4: -5.00 dB
PL5: -5.00 dB
SFO2: 400.135005 MHz

F2 - Processing parameters
SI: 32768
SF: 100.612728 MHz
W0: 0
W1: 0
W2: 0
W3: 0
W4: 0
W5: 0
W6: 0
W7: 0
W8: 0
W9: 0
W10: 0
W11: 0
W12: 0
W13: 0
W14: 0
W15: 0
W16: 0
W17: 0
W18: 0
W19: 0
W20: 0
W21: 0
W22: 0
W23: 0
W24: 0
W25: 0
W26: 0
W27: 0
W28: 0
W29: 0
W30: 0
W31: 0
W32: 0
W33: 0
W34: 0
W35: 0
W36: 0
W37: 0
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W40: 0
W41: 0
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W51: 0
W52: 0
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W60: 0
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W90: 0
W91: 0
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W94: 0
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W96: 0
W97: 0
W98: 0
W99: 0
W100: 0

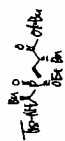
1D IMF plot parameters
CK: 32.00 cm
CY: 34.30 cm
F1: 255.000 ppm
F2: 255.000 ppm
F3: 255.000 ppm
F4: 255.000 ppm
F5: 255.000 ppm
F6: 255.000 ppm
F7: 255.000 ppm
F8: 255.000 ppm
F9: 255.000 ppm
F10: 255.000 ppm
F11: 255.000 ppm
F12: 255.000 ppm
F13: 255.000 ppm
F14: 255.000 ppm
F15: 255.000 ppm
F16: 255.000 ppm
F17: 255.000 ppm
F18: 255.000 ppm
F19: 255.000 ppm
F20: 255.000 ppm
F21: 255.000 ppm
F22: 255.000 ppm
F23: 255.000 ppm
F24: 255.000 ppm
F25: 255.000 ppm
F26: 255.000 ppm
F27: 255.000 ppm
F28: 255.000 ppm
F29: 255.000 ppm
F30: 255.000 ppm
F31: 255.000 ppm
F32: 255.000 ppm
F33: 255.000 ppm
F34: 255.000 ppm
F35: 255.000 ppm
F36: 255.000 ppm
F37: 255.000 ppm
F38: 255.000 ppm
F39: 255.000 ppm
F40: 255.000 ppm
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F47: 255.000 ppm
F48: 255.000 ppm
F49: 255.000 ppm
F50: 255.000 ppm
F51: 255.000 ppm
F52: 255.000 ppm
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F65: 255.000 ppm
F66: 255.000 ppm
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F80: 255.000 ppm
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F86: 255.000 ppm
F87: 255.000 ppm
F88: 255.000 ppm
F89: 255.000 ppm
F90: 255.000 ppm
F91: 255.000 ppm
F92: 255.000 ppm
F93: 255.000 ppm
F94: 255.000 ppm
F95: 255.000 ppm
F96: 255.000 ppm
F97: 255.000 ppm
F98: 255.000 ppm
F99: 255.000 ppm
F100: 255.000 ppm

¹³C NMR of 22a

1H12-72-1 phosphorus

50.8502

ppm



Current Data Parameters
NAME: 1H1
EXPNO: 2
PROCNO: 2

F2 - Acquisition Parameters
Date_: 20071210
Time: 20.19
INSTRUM: spect
PROBHD: 5 mm GNP 1H/13
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 64
DS: 2
SWH: 64935.066 Hz
FIDRES: 0.990630 Hz
AQ: 0.5946772 sec
RG: 1150.72
CW: 17.700 usec
DE: 6.00 usec
TE: 300.2 K
D1: 2.00000000 sec
d11: 0.03000000 sec
DELTA: 1.89999998 sec
MCREST: 0.00000000 sec
MCMRK: 0.01500000 sec

===== CHANNEL f1 =====
NUC1: 31P
P1: 6.90 usec
PL1: -4.00 dB
SFO1: 161.935040 MHz

===== CHANNEL f2 =====
CPDPRG2: waltz16
NUC2: 1H
PCPD2: 100.00 usec
PL2: -4.00 dB
PL12: 17.00 dB
PL13: 17.00 dB
SFO2: 400.0316001 MHz

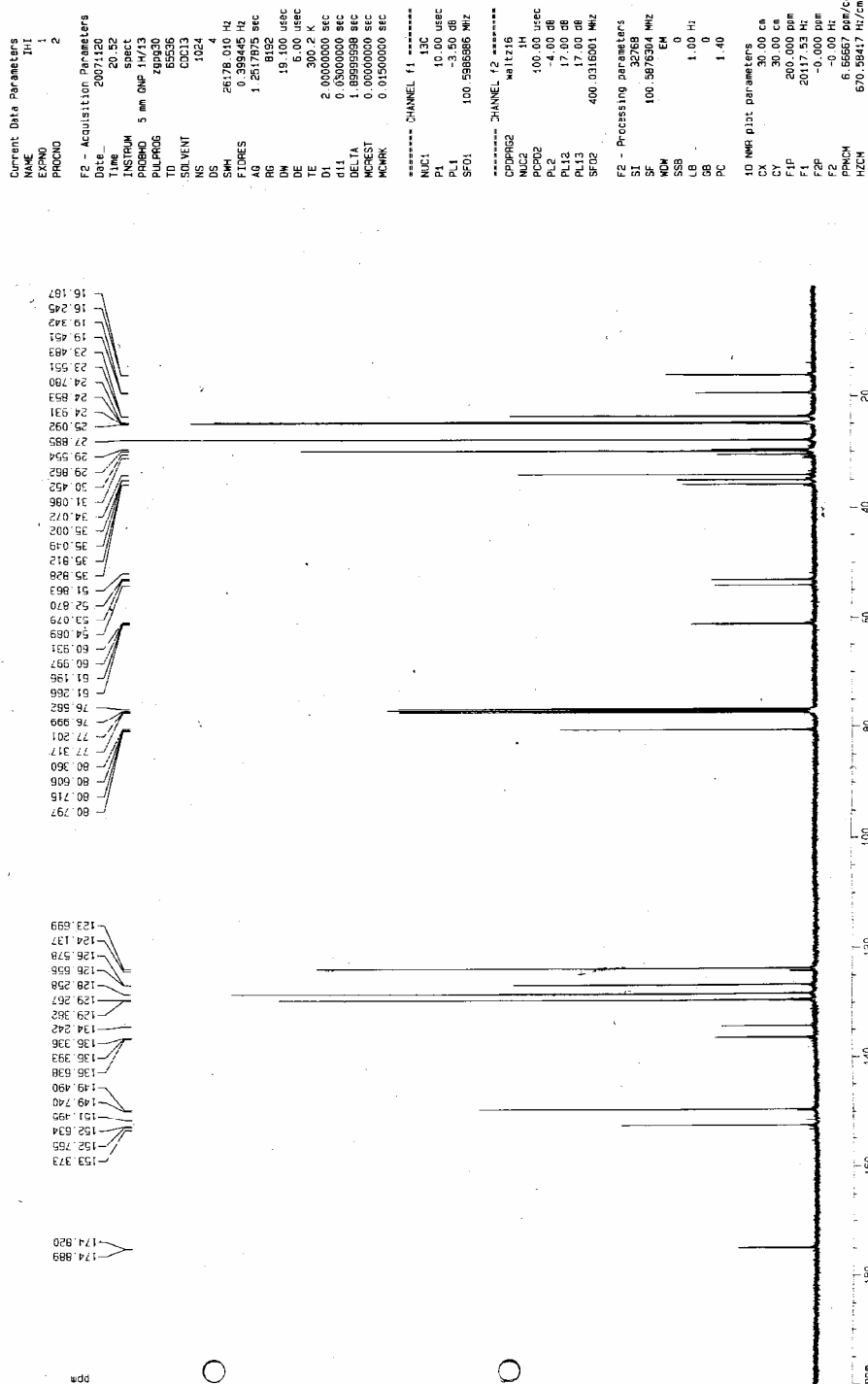
F2 - Processing parameters
SI: 32768
SF: 161.935040 MHz
WDW: EM
SSB: 0
LB: 1.00 Hz
GB: 0
PC: 1.40

1D NMR plot parameters
CX: 32.00 cm
CY: 120.00 cm
FIP: 60.000 ppm
F1: 9716.10 Hz
F2P: -1.000 ppm
F2: -161.93 Hz
PPMCM: 1.90625 ppm/cm
HZCM: 308.68660 Hz/cm

³¹P NMR of 22a

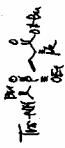


DPX400 carbon
 IH¹⁴-B-1 carbon



¹³C NMR of 23

1H14-8-1 phosphorus



```

Current Data Parameters
NAME      IH1
EXPNO     3
PROCNO    2

F2 - Acquisition Parameters
Date_     20071210
Time      19:30
INSTRUM   spect
PROBHD    5 mm QNP 1H/13
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         64
DS         2
SWH        64395.066 Hz
FIDRES     0.590630 Hz
AQ          0.5046772 sec
RG          9195.2
DN          7.700 usec
DE          6.00 usec
TE         300.2 K
D1         2.80000000 sec
d11        0.63000000 sec
DELTA      1.89999998 sec
MCREST     0.80000000 sec
MCMRK      0.01500000 sec

===== CHANNEL f1 =====
NUC1       31P
P1         6.30 usec
PL1        -4.00 dB
SFO1       161.935026 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
P2         100.00 usec
PL2        -4.00 dB
PL12       17.00 dB
PL13       17.00 dB
SFO2       400.0316001 MHz

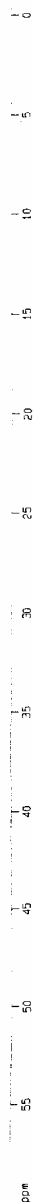
F2 - Processing parameters
SI         32768
SF         161.935026 MHz
WDW         EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40

1D NMR plot parameters
CX          32.00 cm
CY          20.00 cm
F1P         60.000 ppm
F1          9716.10 Hz
F2P         -1.000 ppm
F2          -161.93 Hz
PPHOM       1.90625 ppm/cm
HZCM        368.6860 Hz/cm
  
```

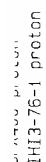
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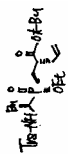
51.1272

ppm

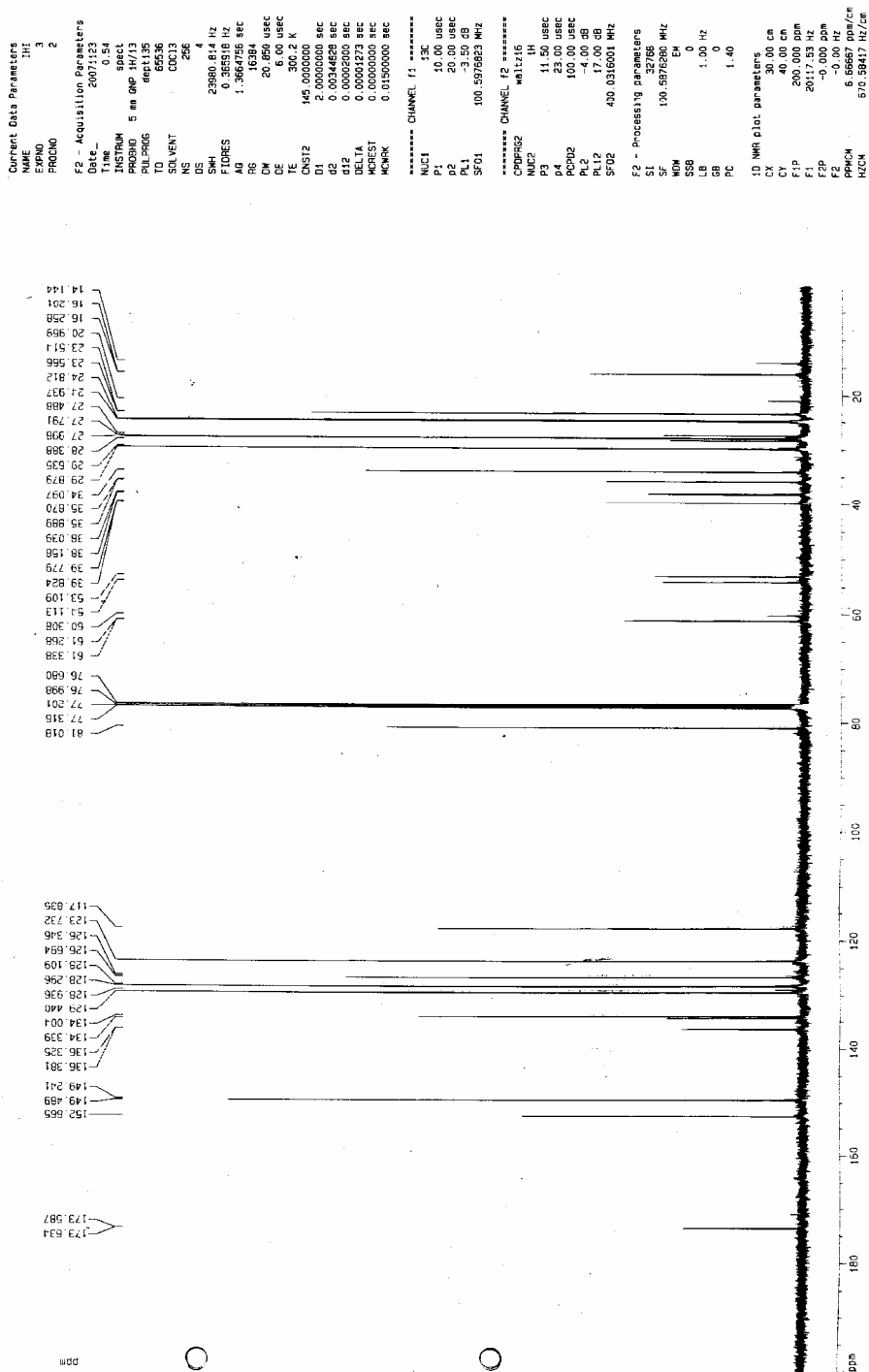


³¹P NMR of 23

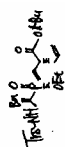




DPX400 carbon
IH14-76-1 carbon



¹³C NMR of 24



Current Data Parameters
 NAME IHI
 EXPNO 2
 PROCNO 3

F2 - Acquisition Parameters
 Date_ 20071210
 Time_ 20:29
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT D2O
 NS 64
 DS 2
 SWH 64635.066 Hz
 FIDRES 0.990830 Hz
 AQ 0.5046772 sec
 RG 4096
 DM 7.700 usec
 DE 5.00 usec
 TE 300.2 K
 D1 2.00000000 sec
 d11 0.00000000 sec
 DELTA 1.00000000 sec
 ACQRES 0.00000000 sec
 WREST 0.00000000 sec
 WCNK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 31P
 P1 6.90 usec
 PL1 -4.00 dB
 SFO1 161.9350920 MHz

===== CHANNEL f2 =====
 CPOPRG2 waltz16
 NUC2 1H
 P2 100.00 usec
 PL2 -4.00 dB
 PL12 17.00 dB
 PL13 17.00 dB
 SFO2 400.0316001 MHz

F2 - Processing parameters
 SI 32768
 SF 161.9350925 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 32.00 cm
 CY 240.00 cm
 FIP 60.000 ppm
 F1 9716.10 Hz
 F2 -1.000 ppm
 F2 161.93 Hz
 FWHM 1.000 ppm/cm
 HZCM 308.66660 Hz/cm

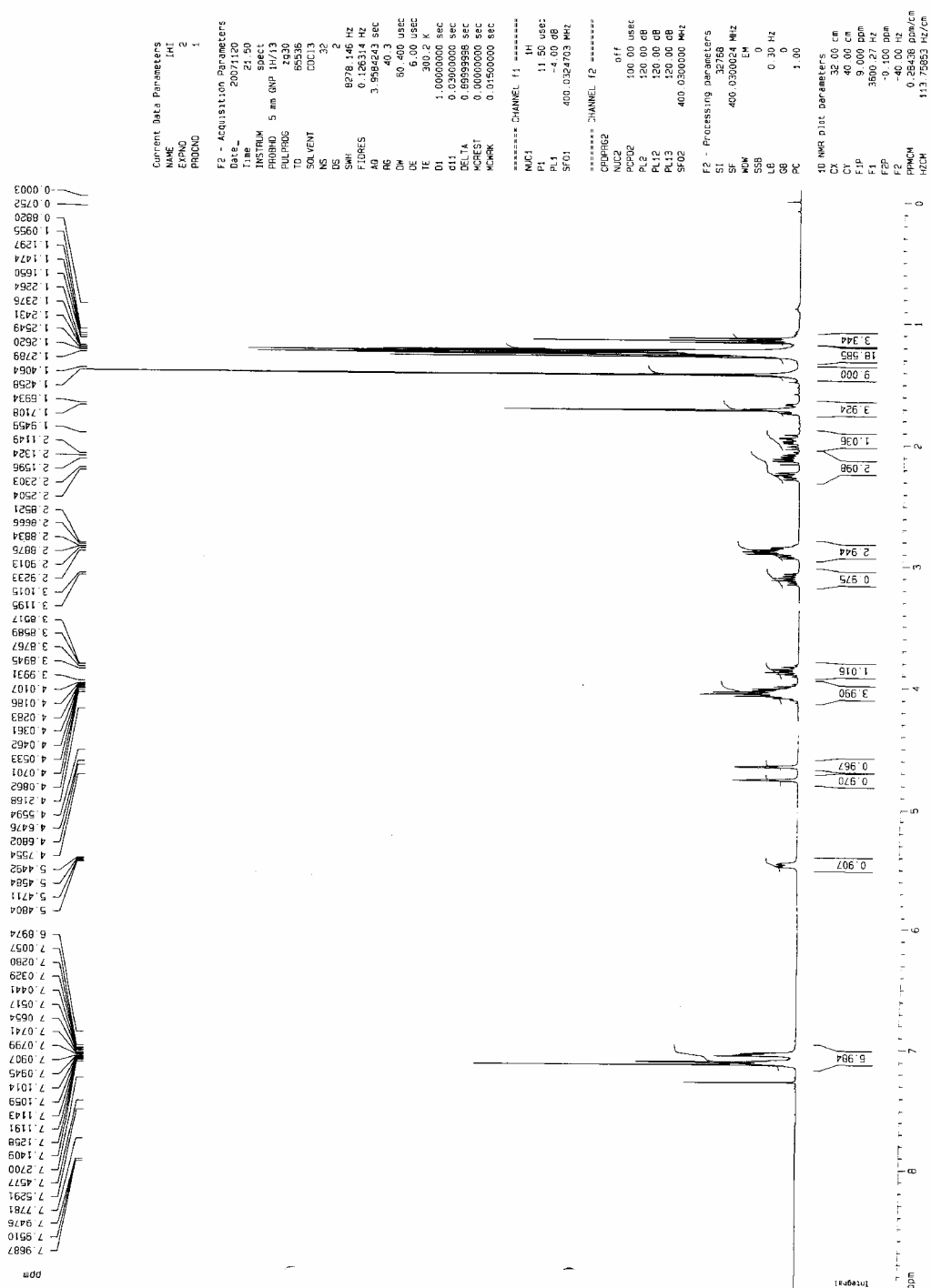
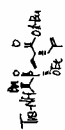
1000 0

51.1401

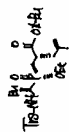
ppm

³¹P NMR of 24

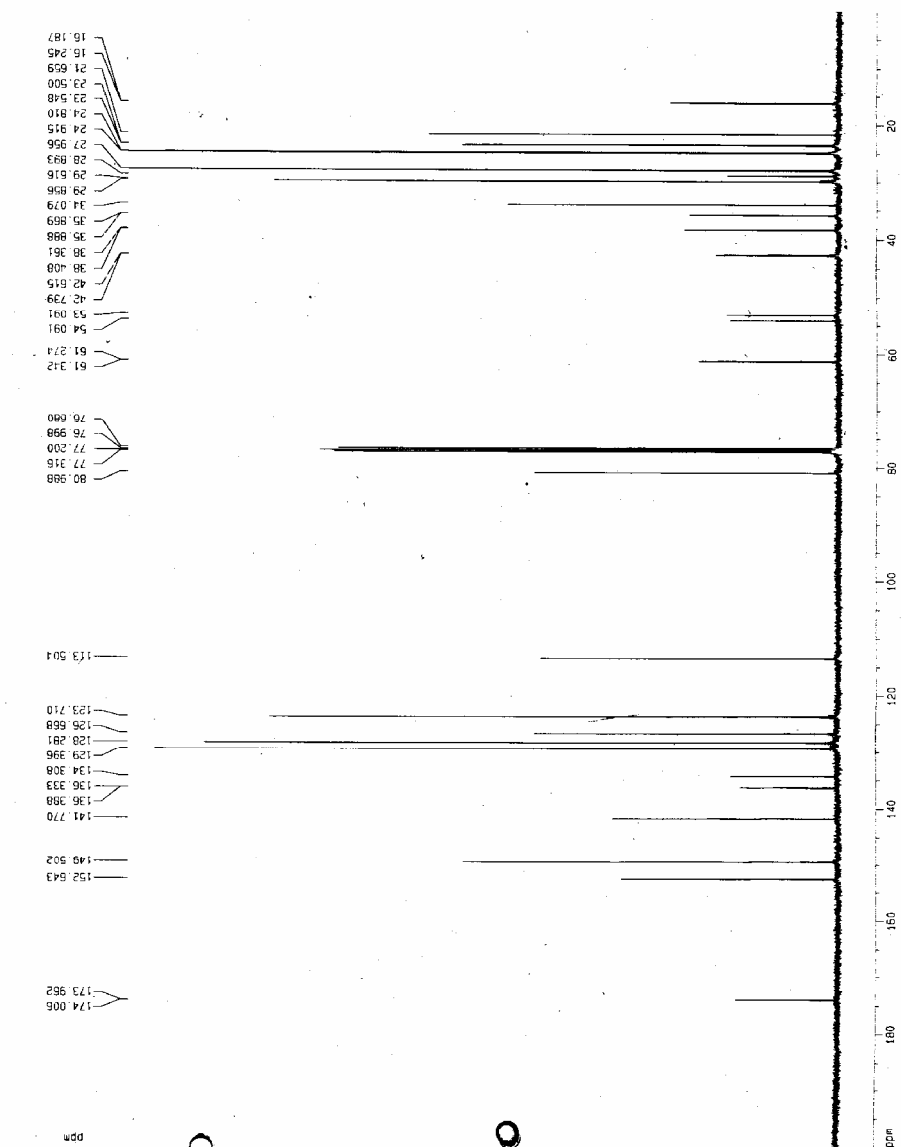
1H4-Z-1 proton



¹H NMR of 25



DPX400 carbon
175 471

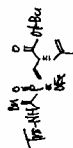


Current Data Parameters
NAME 171
EXPNO 2
PROCNO 2
F2 - Acquisition Parameters
Date_ 20071120
Time 22:25
INSTRUM 5 mm QNP 1H/13
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT DMS-D6
NS 1024
DS 4
SWH 26170.012 Hz
FIDRES 0.38045 Hz
AQ 1.2517573 SEC
RG 81.92
DB 19.103 usec
DE 6.00 usec
TE 300.2 K
DMSI2 145.000000 SEC
D1 2.00000000 SEC
D2 0.03000000 SEC
DELTA 0.00000000 SEC
DELT 0.00001273 SEC
MORST 0.00000000 SEC
MORR 0.03500000 SEC
***** CHANNEL f1 *****
NUC1 13C
P1 10.00 usec
PL1 20.00 dB
PL2 -3.50 dB
SFO1 100.598886 MHz
***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
P2 11.50 usec
PL2 23.00 dB
PCPD2 100.00 usec
PL2 -4.00 dB
SFO2 400.031600 MHz
F2 - Processing parameters
SI 32768
SF 100.5876236 MHz
WDW EN
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 30.00 cm
CY 30.00 cm
FJP 200.000 ppm
F1 20117.53 Hz
F2P -0.000 ppm
P1 0.00000000 Hz
P2 6.66666667 Hz
HZCM 670.58417 Hz/cm

¹³C NMR of 25

IH14-7-; phosphorus

51.1304
ppm



Current Data Parameters
NAME IH1
EXPNO 3
PROCNO 3

F2 - Acquisition Parameters
Date_ 20071210
Time 19.38
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT D2O
NS 64
DS 2
SWH 64035.065 Hz
FIDRES 0.590930 Hz
AQ 0.5046772 sec
RG 5195.2
DM 7.700 usec
DE 6.00 usec
TE 300.2 K
D1 2.0000000 sec
d11 0.0300000 sec
DELTA 1.8995998 sec
MORST 0.0000000 sec
MCNRK 0.0150000 sec

===== CHANNEL f1 =====
NUC1 31P
P1 6.90 usec
PL1 -4.00 dB
SFO1 161.9350920 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
P2 100.00 usec
PL2 -4.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 400.0316001 MHz

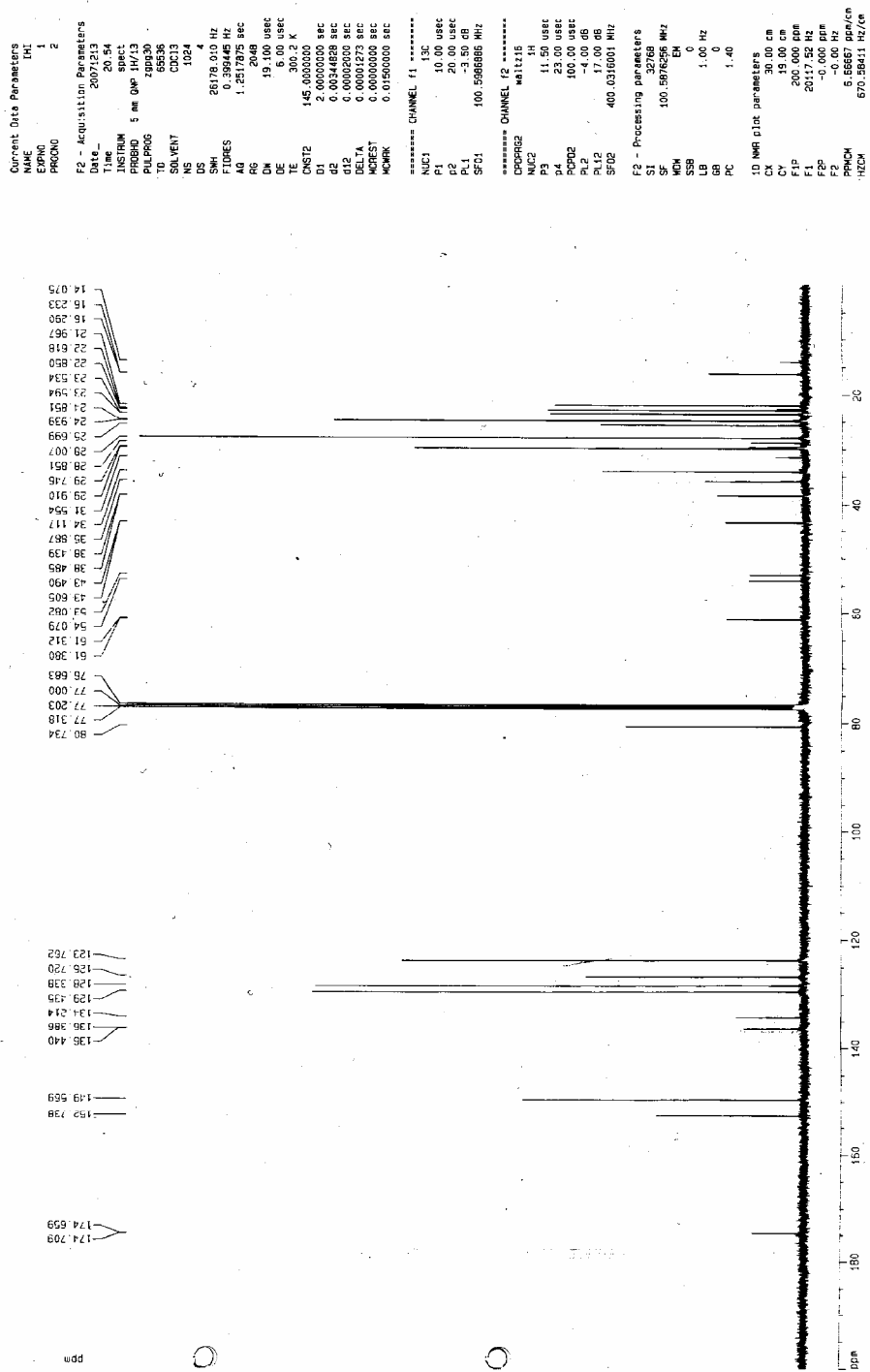
F2 - Processing parameters
SI 32768
SF 161.9350920 MHz
WDW EM
SS 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 32.00 cm
CY 60.00 cm
FIP 60.000 ppm
F1 9716.10 Hz
F2 -1.000 ppm
F2 -161.93 Hz
PPHM 1.90625 ppm/cm
HZCM 309.68660 Hz/cm

³¹P NMR of 25

CC1=CC=C(C=C1)C2=CC=CC=C2
 100%
 100%

DPX400 carbon
 125.76 MHz

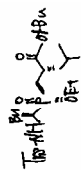


¹³C NMR of 26

THE 4-15-1 phosphorus

ppm 0 50 100

0.542
0.288
0.000



Current Data Parameters
NAME IHI
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters

Date_ 20071220
Time 22:24
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT D2O
NS 64
DS 2
SWH 64935.066 Hz
FIDRES 0.990830 Hz
AQ 0.5446772 sec
RG 9195.2
DM 7.700 usec
DE 5.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89599998 sec
ACQRES 0.00000000 sec
PCPRG2 0.01500000 sec

***** CHANNEL f1 *****

NIC1 31P
PL1 8.30 usec
PL2 4.00 dB
SFO1 161.935048 MHz

***** CHANNEL f2 *****

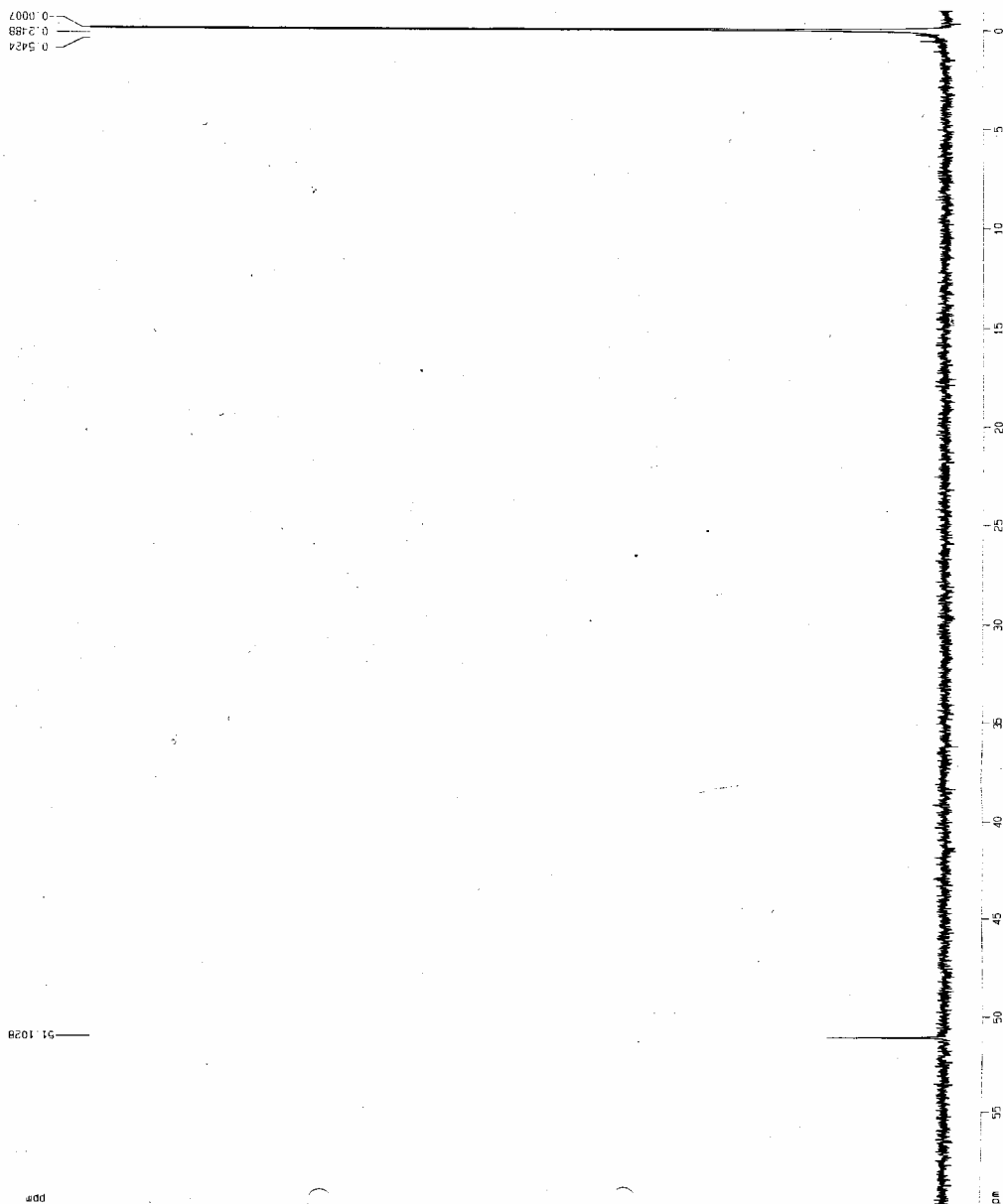
OPDPRG2 waltz16
NIC2 1H
PL2 100.00 usec
PL2 -4.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 400.0315001 MHz

F2 - Processing parameters

SI 32768
SF 161.935048 MHz
WDW EN
SSB 0
LB 0
GB 0
PC 1.40

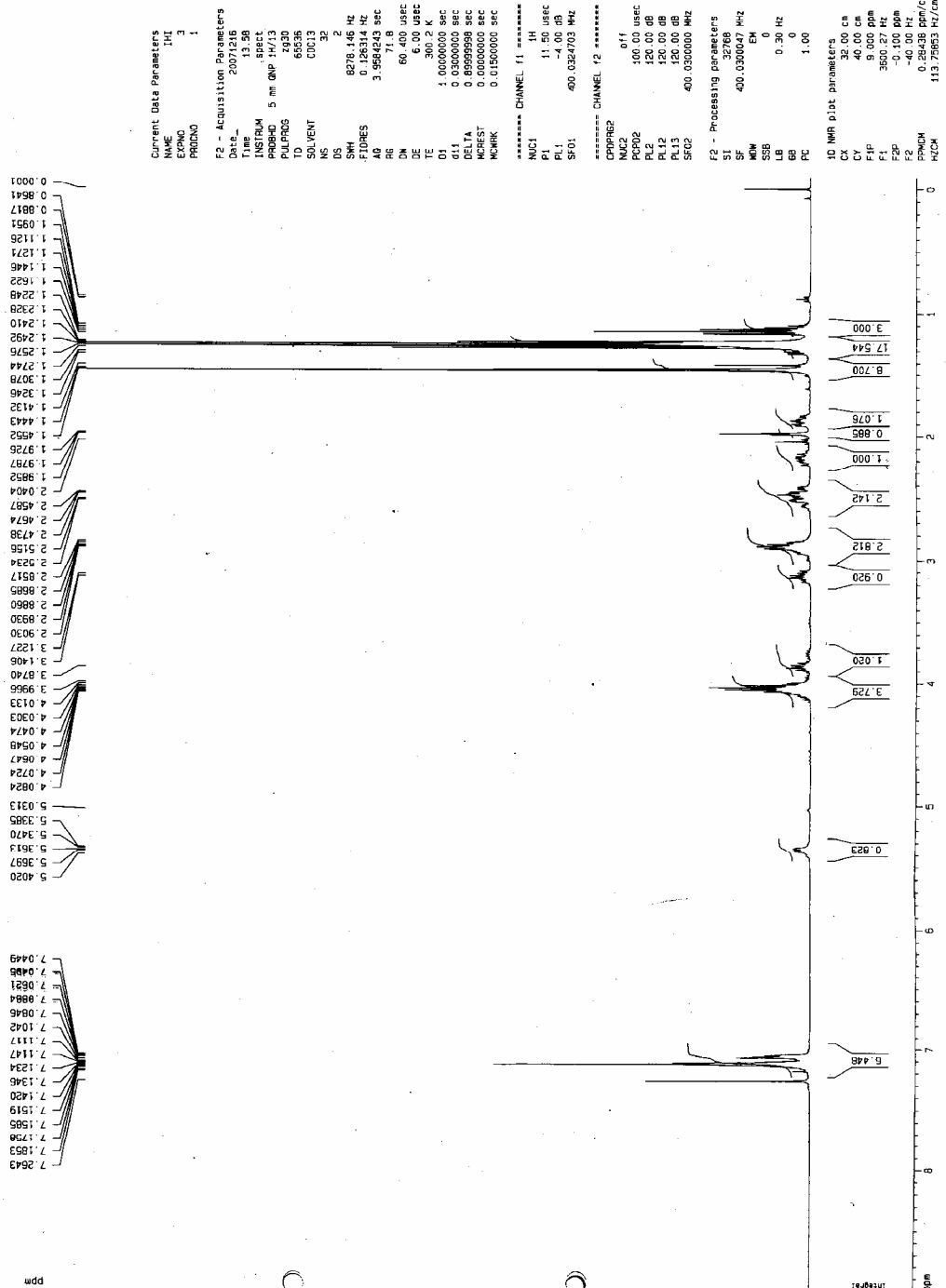
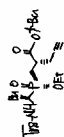
10 MHz plot parameters

CX 32.00 cm
CY 280.00 cm
F1P 80.000 ppm
F1 9716.10 Hz
F2P -1.000 ppm
F2 -161.93 Hz
PPMCH 1.90625 ppm/cm
HZCM 308.68860 Hz/cm

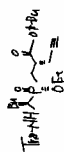


³¹P NMR of 26

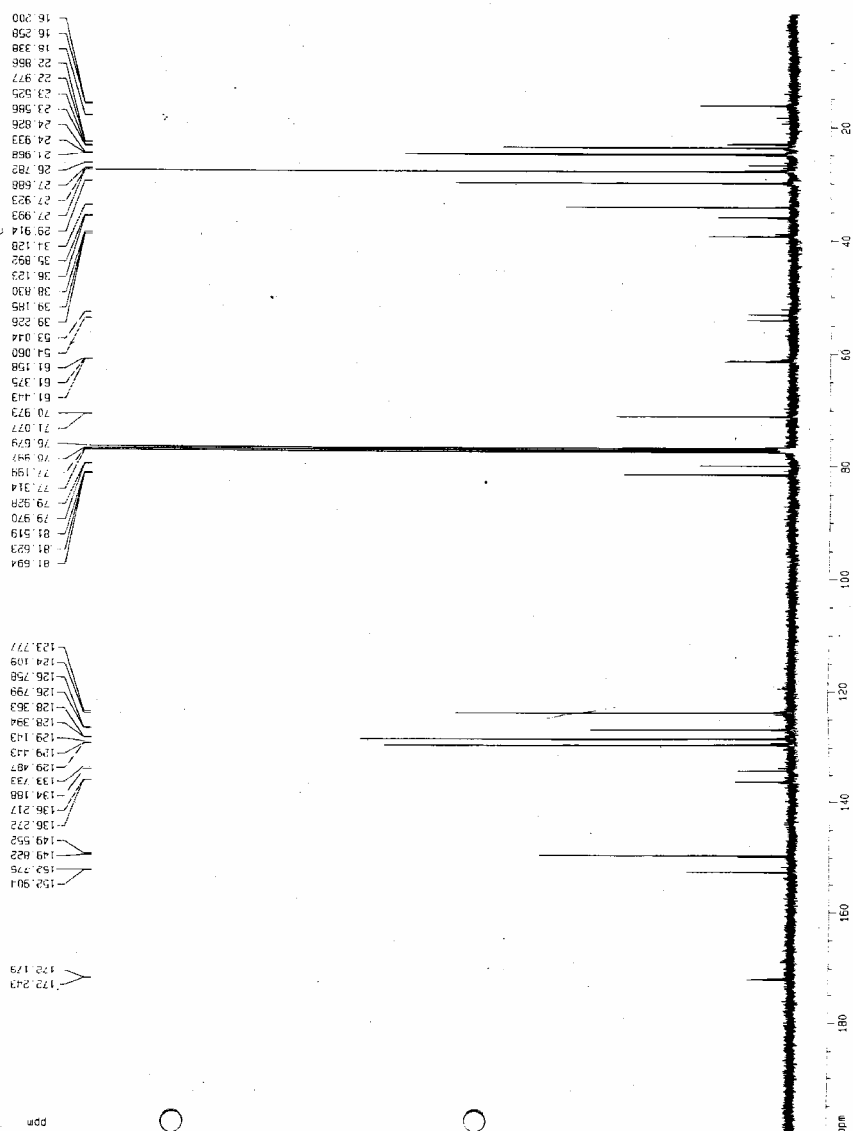
1H14-6-1 proton



¹H NMR of 27



DPX400 carbon
IH14-6-1 carbon

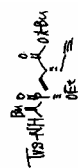


Current Data Parameters

NAME	INH
EXPNO	3
PROCNO	2
F2 - Acquisition Parameters	
Date_	20071215
Time	14.57
INSTRUM	spect
PROBHD	5 mm GNP 1H/13
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
DS	1024
OS	4
SMH	26178.010 Hz
FIDRES	0.399445 Hz
AQ	1.2517875 sec
RG	6192
DM	19.100 usec
DE	3.000 usec
TE	300.2 K
DNS12	14E:00000000
D1	2.00000000 sec
d2	0.00344828 sec
d12	0.00002000 sec
DELTA	0.0001273 sec
NUC1	13C
NUC2	1H
NUC1	13C
P1	10.00 usec
DE	20.00 usec
PL1	0.00 dB
PL2	0.00 dB
SFO1	100.596886 MHz
***** CHANNEL f2 *****	
CPDPRG2	waltz16
NUC2	1H
P2	11.50 usec
DE	20.00 usec
PL2	0.00 dB
PL12	17.00 dB
SFO2	400.0316001 MHz
F2 - Processing parameters	
SI	32768
SF	100.5976260 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40
10 MHz plot parameters	
CX	30.00 cm
CY	19.00 cm
FIP	200.000 cm
F1	20117.52 Hz
F2P	-0.000 ppm
PRNCH	6.666 Hz
HZCM	670.58411 Hz/cm

¹³C NMR of 27

0.14500
IH14-G-1 phosphorus



Current Data Parameters
NAME IH1
EXPNO 3
PROCNO 1

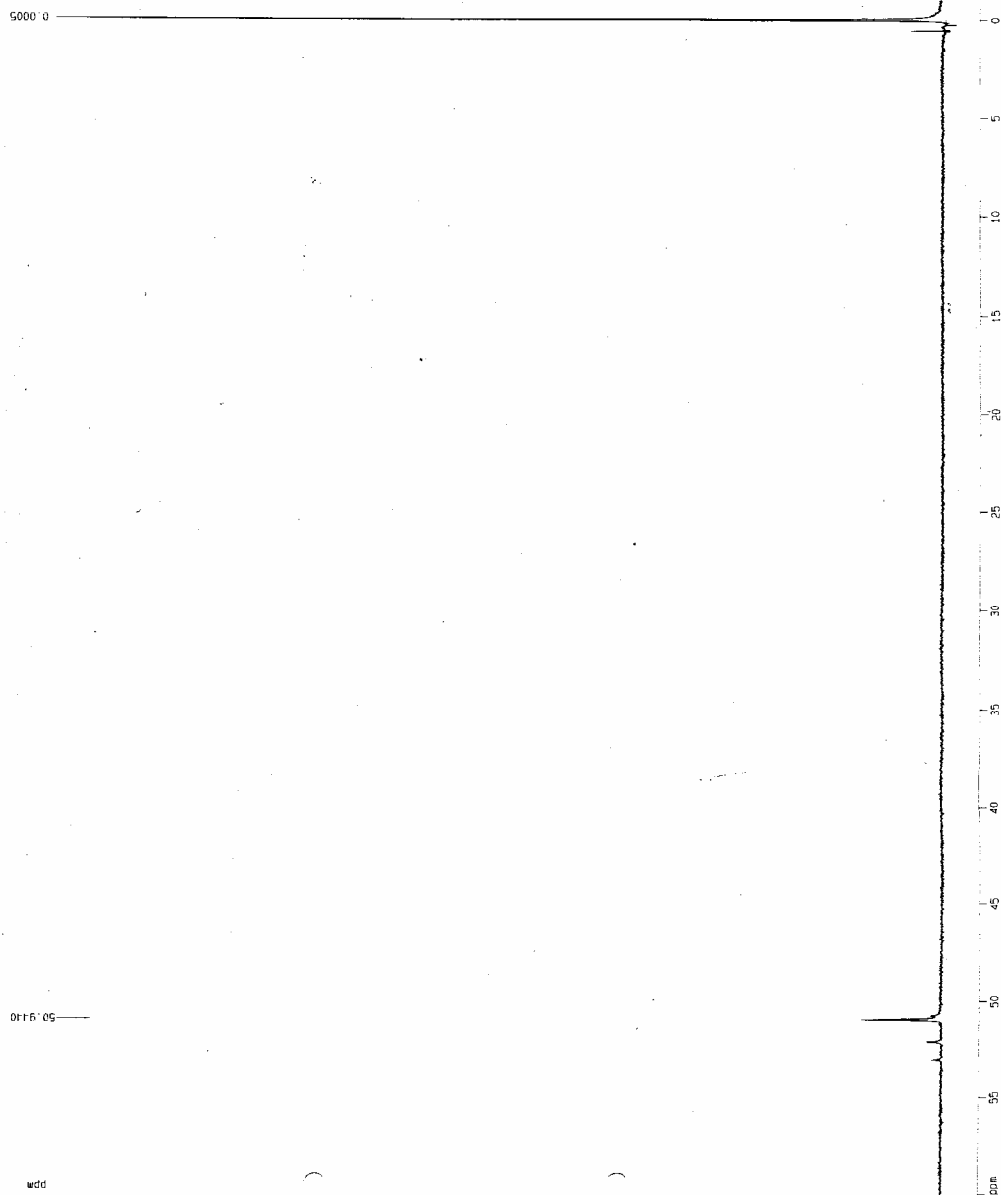
F2 - Acquisition Parameters
Date_ 20071220
Time 22.00
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 654
DS 2
SWH 64535.066 Hz
FIDRES 0.990830 Hz
AQ 0.5646772 sec
RG 9195.2
DM 7.700 usec
DE 6.00 usec
TE 300.2 K
O1 2.0000000 sec
C11 0.0300000 sec
DELTA 1.8999998 sec
ACQRES 0.0000000 sec
MCHRG 0.0150000 sec

===== CHANNEL f1 =====
NUC1 31P
P1 630.0 usec
PL1 -4.00 dB
SFO1 161.950020 MHz

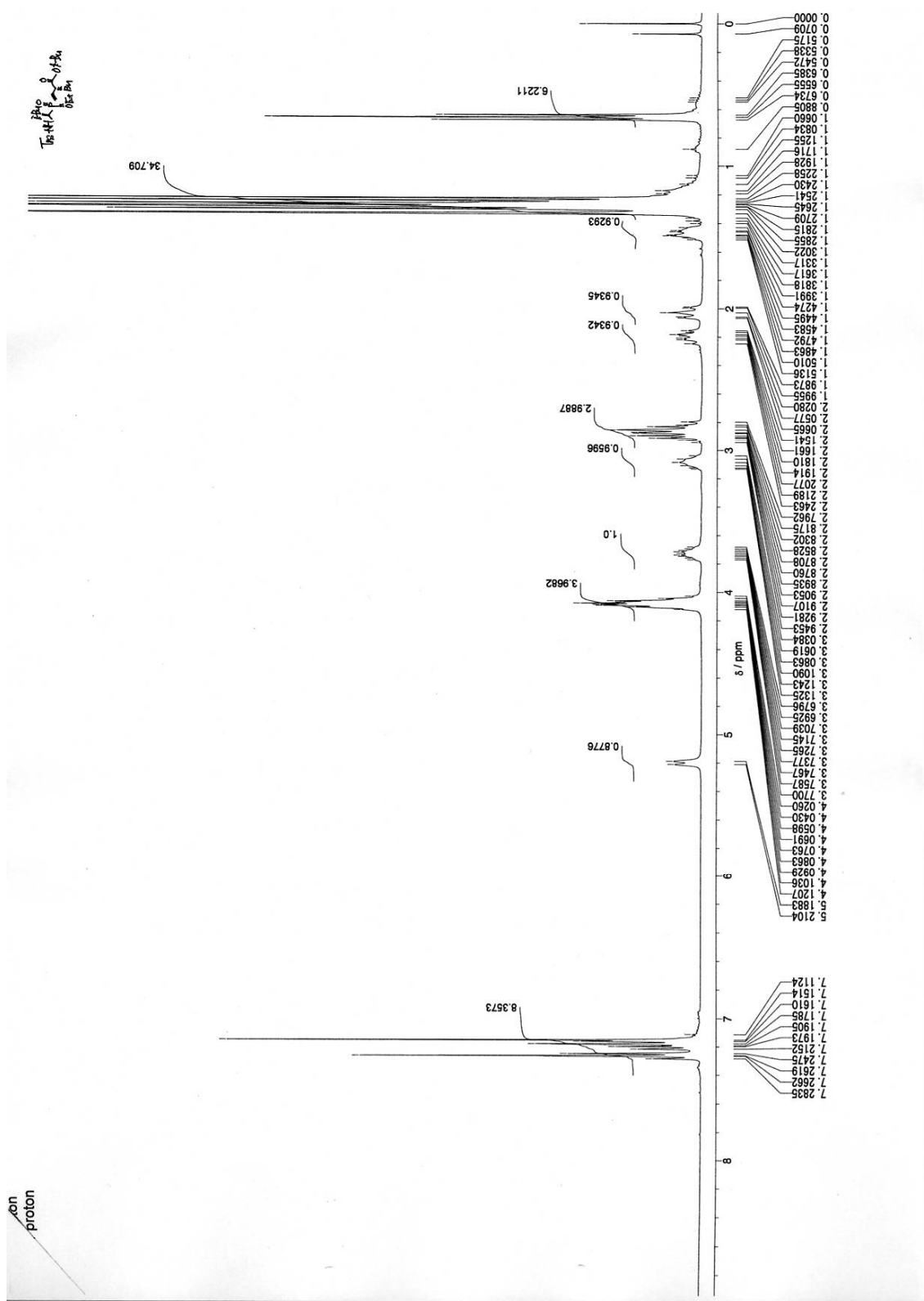
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 400.0315001 MHz

F2 - Processing parameters
SI 32768
SF 161.950020 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

3D NMR plot parameters
CX 32.00 cm
CY 100.00 cm
FIP 90.000 ppm
F1 9716.10 Hz
F2 -1.000 ppm
F2 -161.93 Hz
PPHM 1.90625 ppm/cm
HZCM 398.66660 Hz/cm



³¹P NMR of 27



^1H NMR of 28

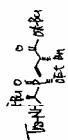
DPX400 carbon
IHI4-25-1 carbon



TH14-25-1 phosphorus

51.5206

ppm



Current Data Parameters
 NAME IHI
 EXPNO 3
 PROCNO 1

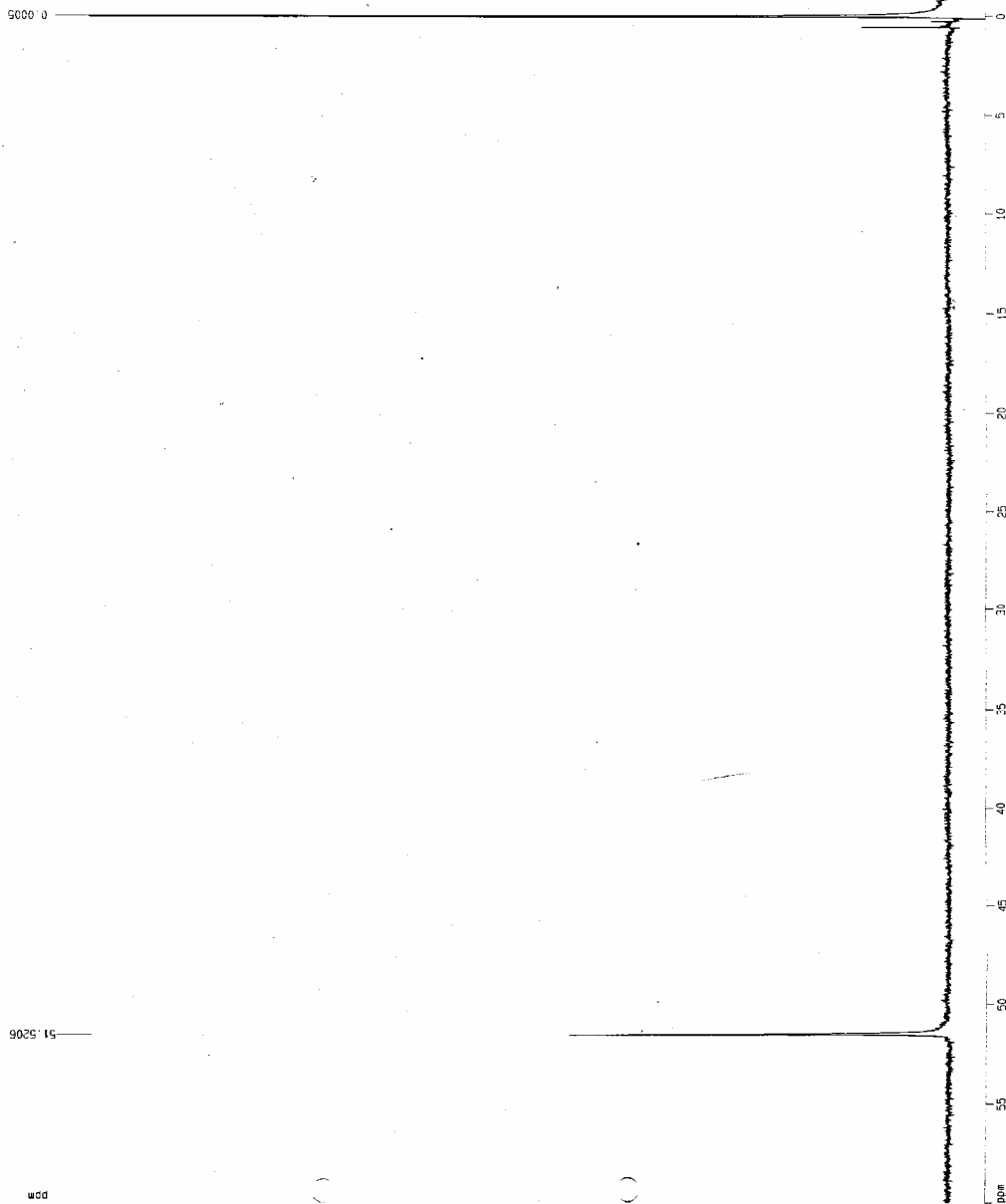
F2 - Acquisition Parameters
 Date_ 20081111
 Time 20:33
 INSTRUM spect
 PROBU 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT D2O
 NS 64
 DS 2
 SWH 64935.066 Hz
 FIDRES 0.990630 Hz
 AQ 0.5046772 sec
 RG 9195.2
 DW 7.700 usec
 DE 5.00 usec
 TE 300.2 K
 D1 2.00000000 sec
 d11 0.43000000 sec
 DELTA 1.69999998 sec
 MCREST 0.00000000 sec
 MCWRR 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 31P
 P1 6.90 usec
 PL1 -4.00 dB
 SF01 161.9350920 MHz

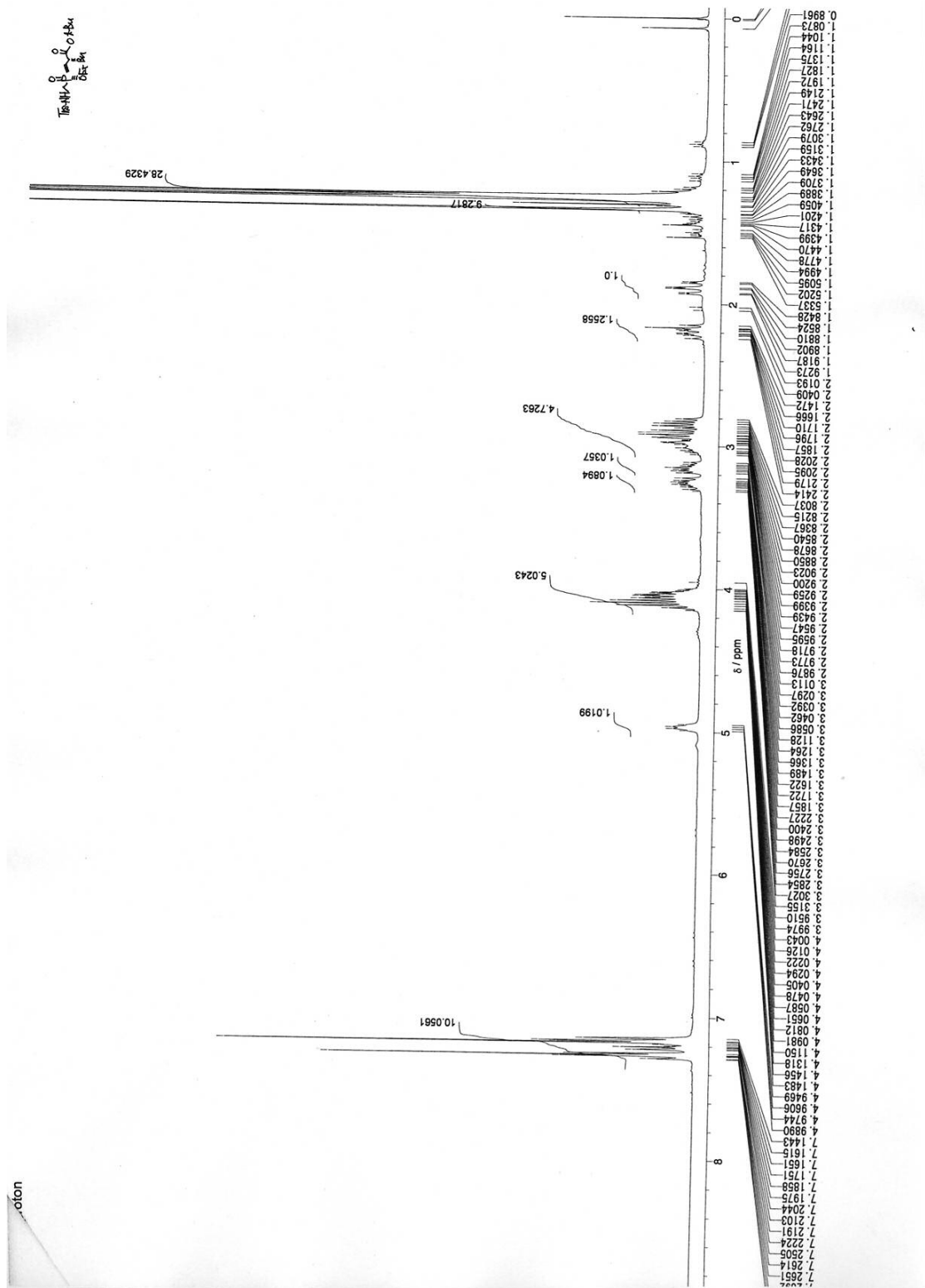
===== CHANNEL f2 =====
 CROPRG2 waltz16
 NUC2 1H
 P2 100.00 usec
 PL2 -4.00 dB
 PL12 17.00 dB
 PL13 17.00 dB
 SF02 400.0316001 MHz

F2 - Processing parameters
 SI 32768
 SF 161.9350920 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

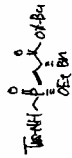
1D NMR plot parameters
 CX 32.00 cm
 CY 234.83 cm
 FIP 60.000 ppm
 F1 9716.10 Hz
 F2P -1.000 ppm
 F2 -161.93 Hz
 PPMCM 1.90625 ppm/cm
 HZCM 308.68660 Hz/cm



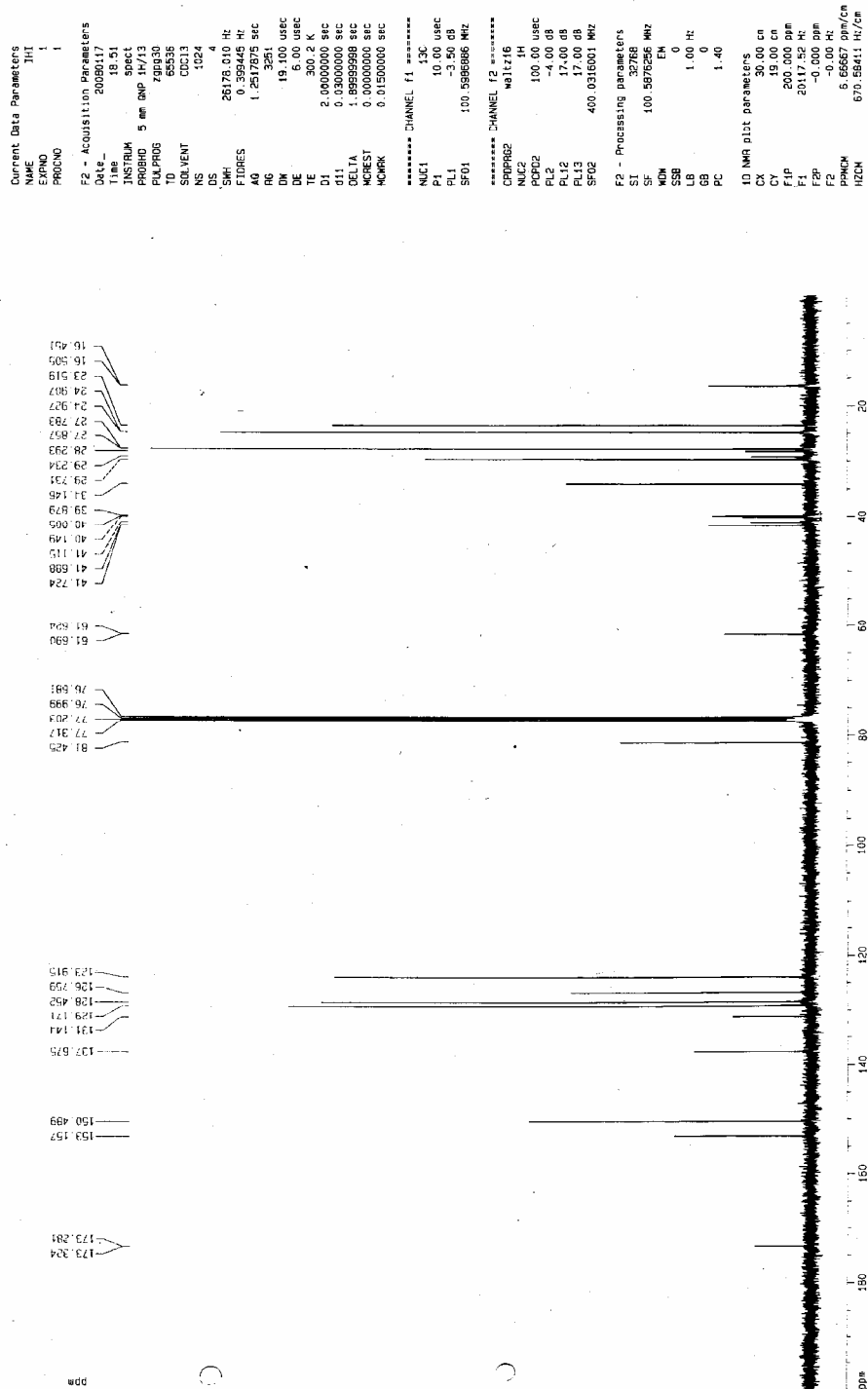
³¹P NMR of 28



¹H NMR of 29

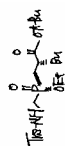


DPX400 carbon
IH14-24-3 carbon



¹³C NMR of 29

IHI4-24-3 phosphorus



0.000

47.5141

Current Data Parameters
NAME IHI
EXPNO 2
PROCNO 3

F2 - Acquisition Parameters
Date_ 20080111
Time 20:26
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT D2O
NS 64
DS 2
SWH 64935.066 Hz
FIDRES 0.590830 Hz
AQ 0.5646772 sec
RG 9195.2
DM 7.700 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.68999998 sec
MCHST 0.00000000 sec
MCHRK 0.01900000 sec

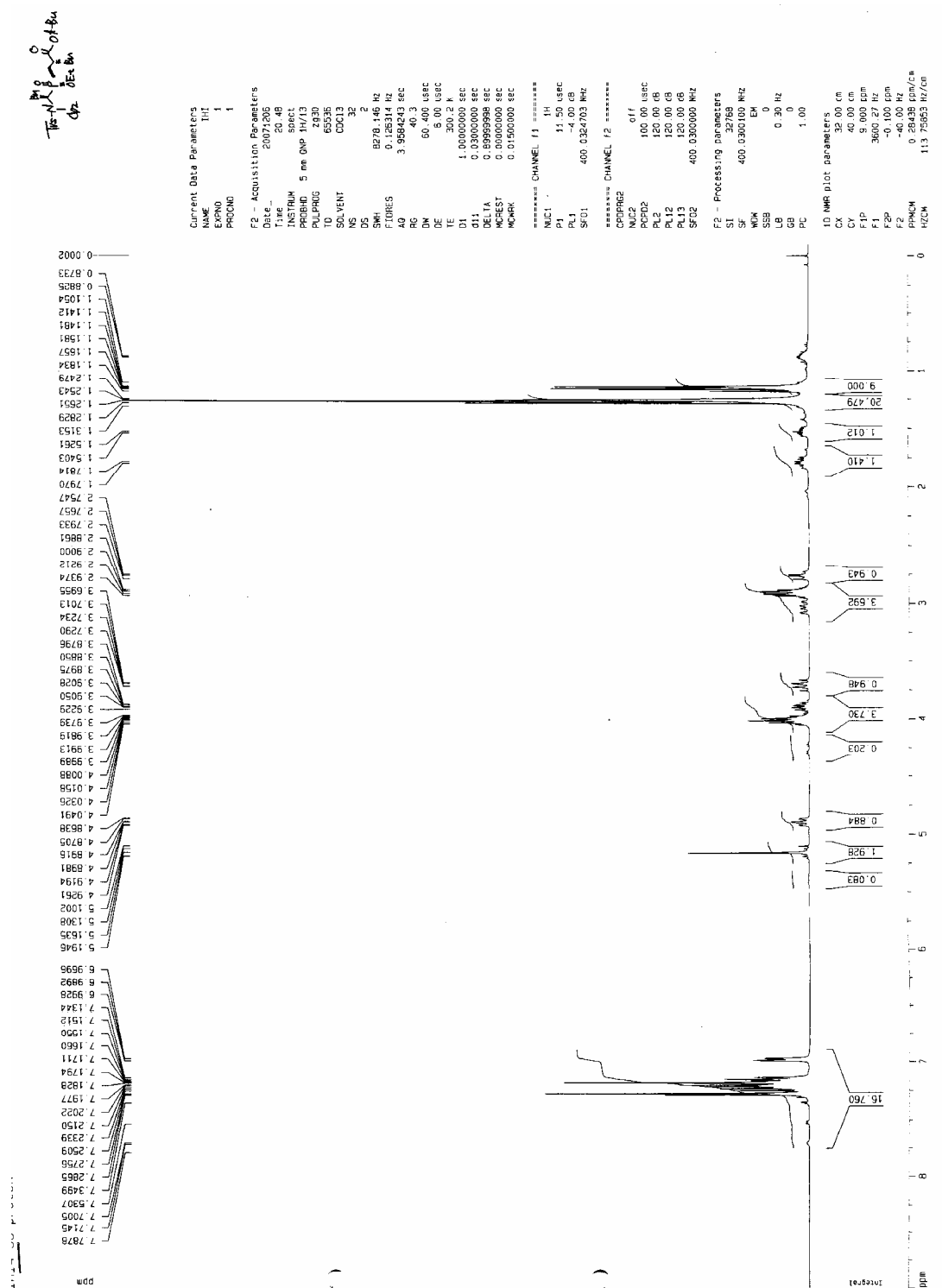
===== CHANNEL f1 =====
NUC1 31P
P1 6.90 usec
PL1 -4.00 dB
SFO1 161.935046 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
P2 100.00 usec
PL2 -4.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 400.0315001 MHz

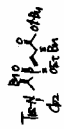
F2 - Processing parameters
SI 32768
SF 161.935046 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

10 NMR plot parameters
CX 32768 cm
CY 263.71 cm
FID 70.000 ppm
F1 11335.46 Hz
F2 -1.000 ppm
F3 -161.94 Hz
P1MCM 2.21875 ppm/cm
HZCM 359.29327 Hz/cm

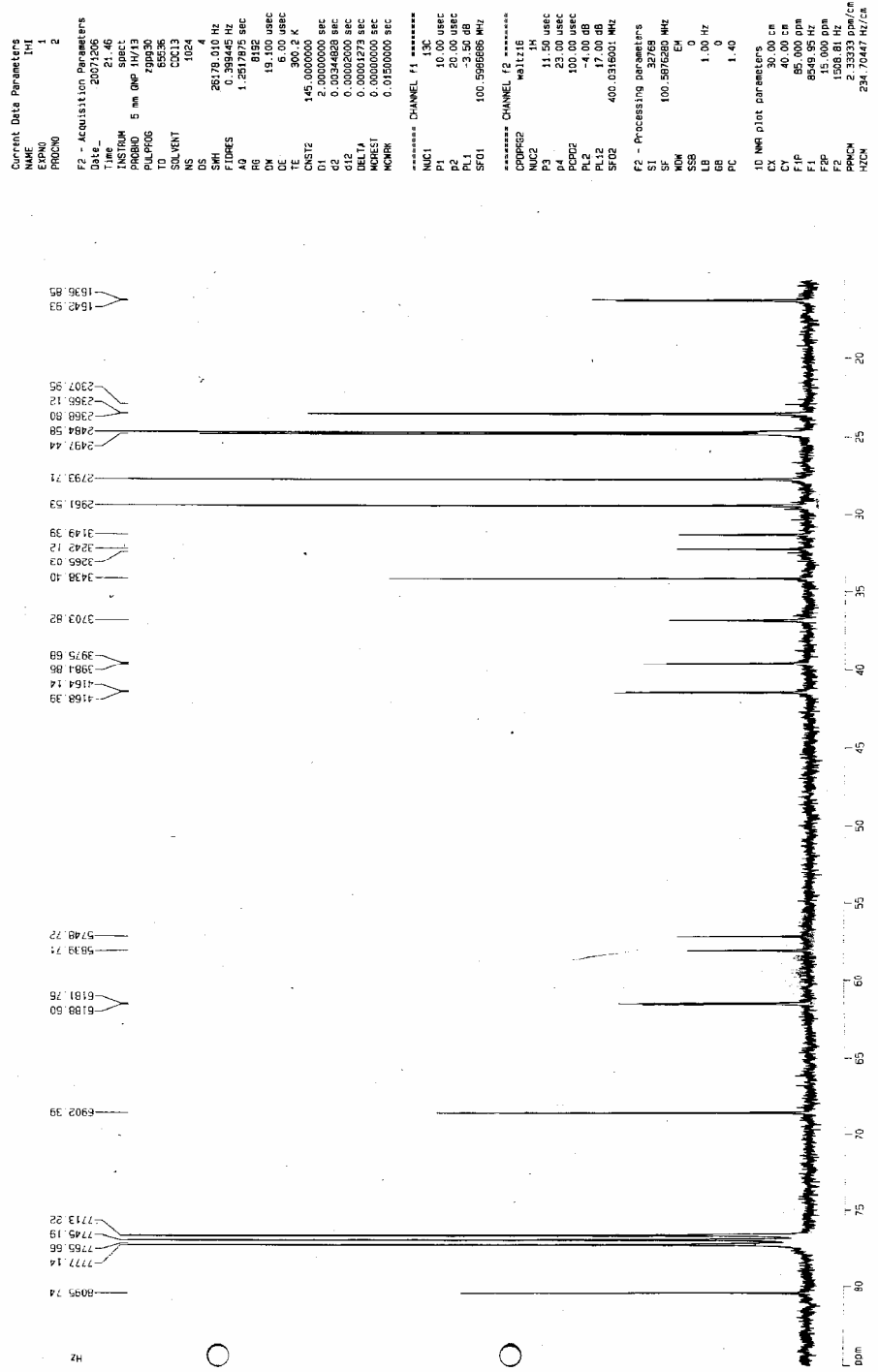
³¹P NMR of 29



¹H NMR of 30

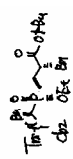


DPX400 carbon
IH14-56 carbon



¹³C NMR of 30

1H14-56 phosphorus



Current Data Parameters
 NAME IHI
 EXPNO 1
 PROCNO 1

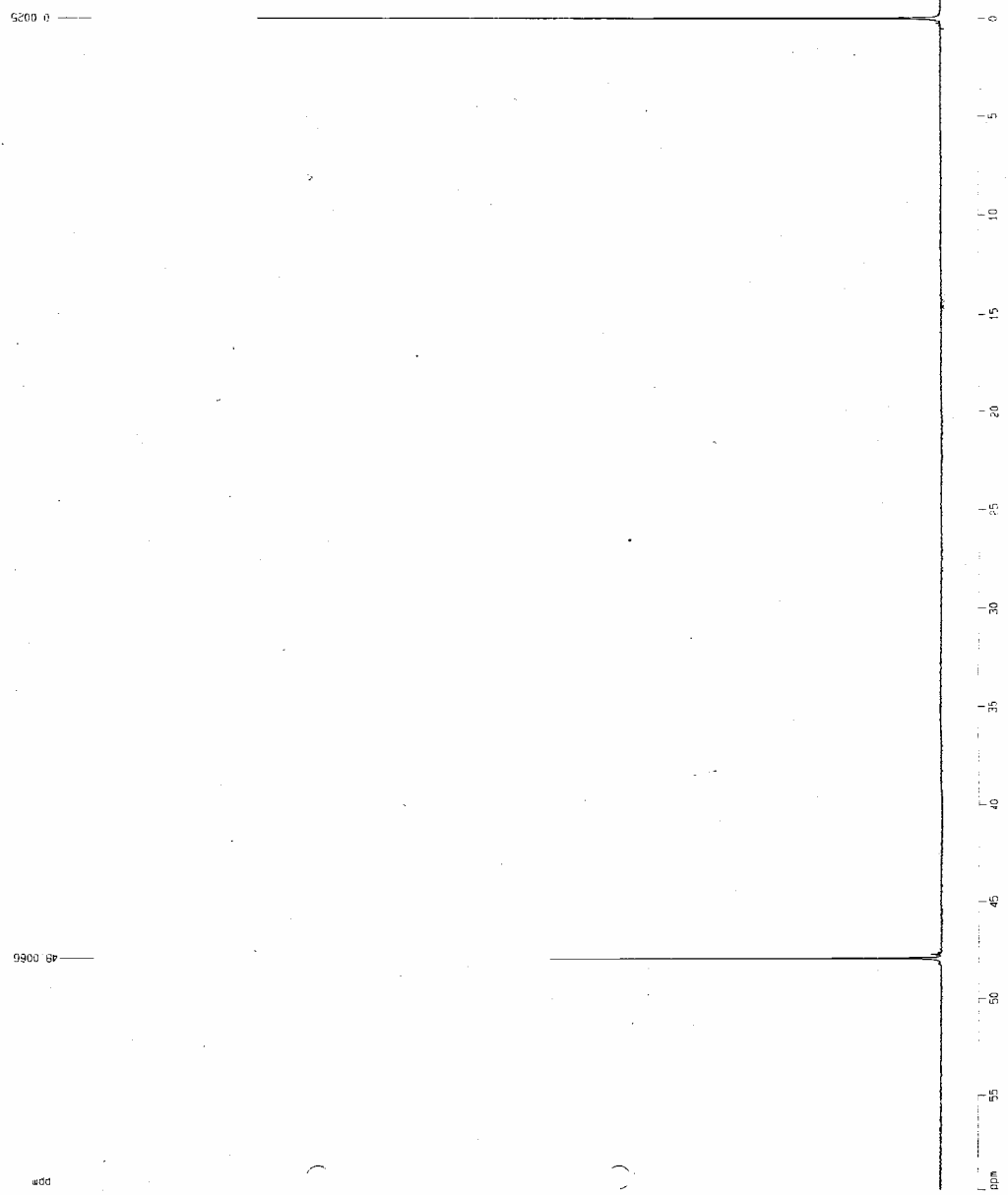
F2 - Acquisition Parameters
 Date_ 20071210
 Time_ 17:51
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TO 45030
 SOLVENT DMSO
 NS 64
 DS 2
 SWH 6435.066 Hz
 FIDRES 0.980530 Hz
 AQ 0.5046772 sec
 RG 14596.5
 DW 7.700 usec
 DE 6.00 usec
 TE 300.2 K
 D1 2.0000000 sec
 d11 0.0300000 sec
 DELTA 1.8959999 sec
 MCREST 0.0000000 sec
 MCPRK 0.0150000 sec

===== CHANNEL f1 =====
 NUC1 31P
 P1 6.90 usec
 PL1 -4.00 dB
 SF01 161.9350520 MHz

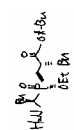
===== CHANNEL f2 =====
 CDPDPR2 waitz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 -4.00 dB
 PL12 17.00 dB
 PL13 17.00 dB
 SF02 400.0316001 MHz

F2 - Processing parameters
 SI 32768
 SF 161.9350548 MHz
 WDM EN
 SSF 0
 LB 1.00 Hz
 GB 0
 PC 1.40

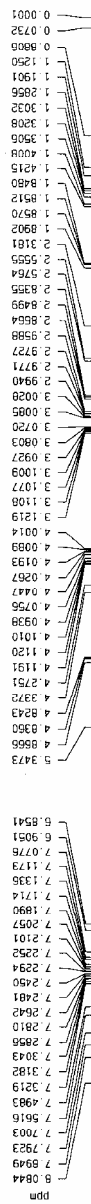
1D NMR plot parameters
 CX 32.00 cm
 CY 18.50 cm
 FIP 60.000.00m
 F1 9716.10 Hz
 F2 -1.000.00m
 F2 -161.93 Hz
 PPMCM 1.90625 ppm/cm
 HZCM 308.68860 Hz/cm



³¹P NMR of 30



1H15-3 proton



Current Data Parameters
 NAME 1H1
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20071215
 Time 12.35
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 D1 2.00
 DELTA 0.05000000
 SOLVENT CDCl3
 NS 32
 DS 2
 SWH 6278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 71.8
 DN 60.400 usec
 DE 19.00 usec
 TE 300.2 K
 O1 1.00000000 sec
 d11 0.03400000 sec
 DELTA 0.89599996 sec
 MCHRG 0.00000000 sec
 MCHRG 0.01500000 sec

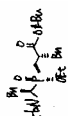
===== CHANNEL f1 =====
 NUC1 1H
 PL1 11.50 usec
 PL1 -4.00 dB
 SF01 400.0324703 MHz

===== CHANNEL f2 =====
 CPMRG2 gfg
 NUC2 13C
 PL2 100.00 usec
 PL2 120.00 dB
 PL2 120.00 dB
 PL2 120.00 dB
 SF02 400.0300000 MHz

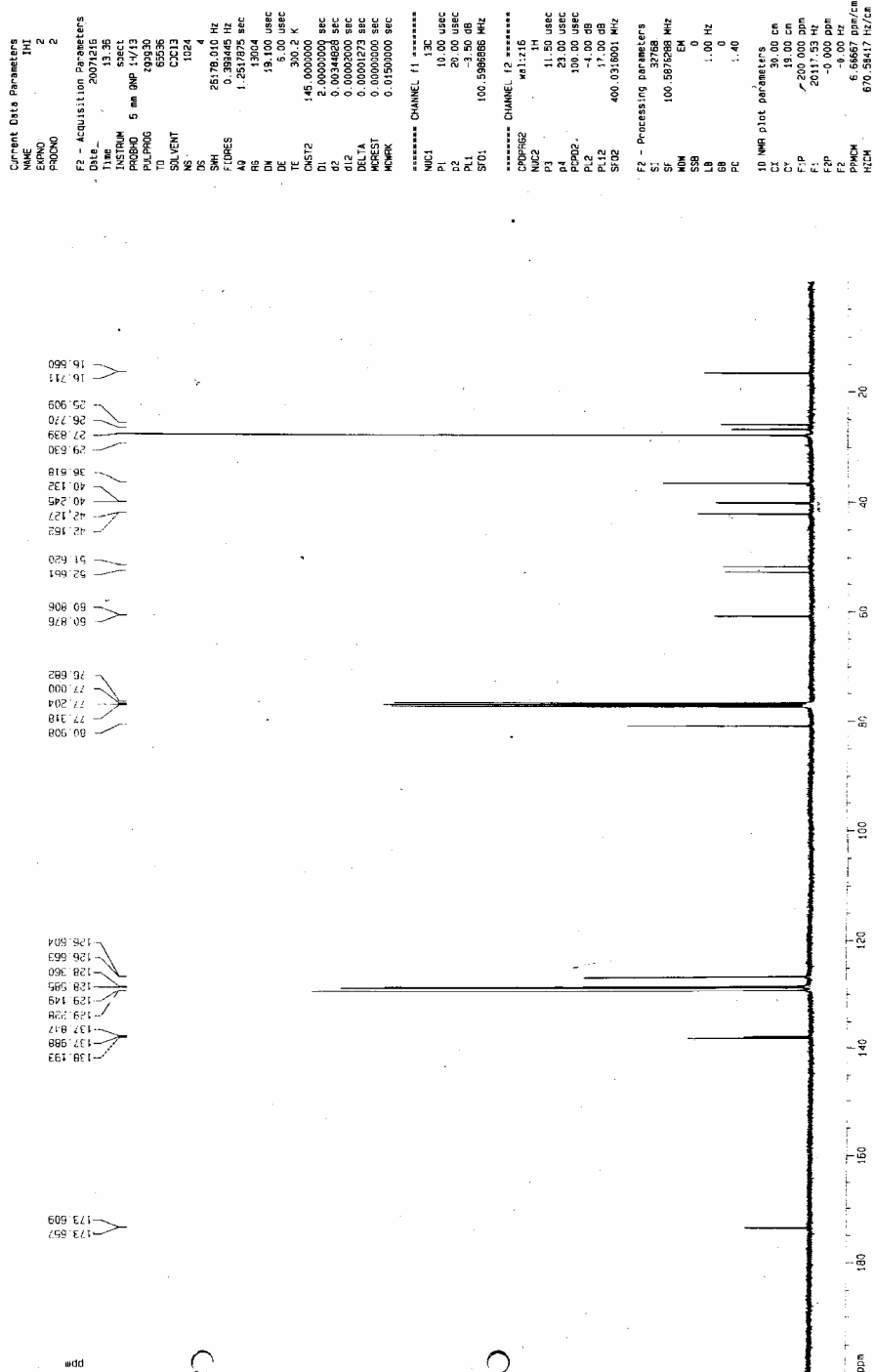
F2 - Processing parameters
 SI 32768
 SF 400.0300000 MHz
 NUCN 13C
 SFO 400.0300000 MHz
 EQ 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 32.00 cm
 FWHM 9.600000
 F1 3600.27 Hz
 F2 -0.100 ppm
 F2 28438.000 Hz
 FWHM 113.75853 Hz/cm

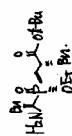
¹H NMR of 31



DPX400 carbon
IHI-53 carbon



¹³C NMR of 31



Current Data Parameters
NAME IH1
EXPNO 3
PROCNO 2

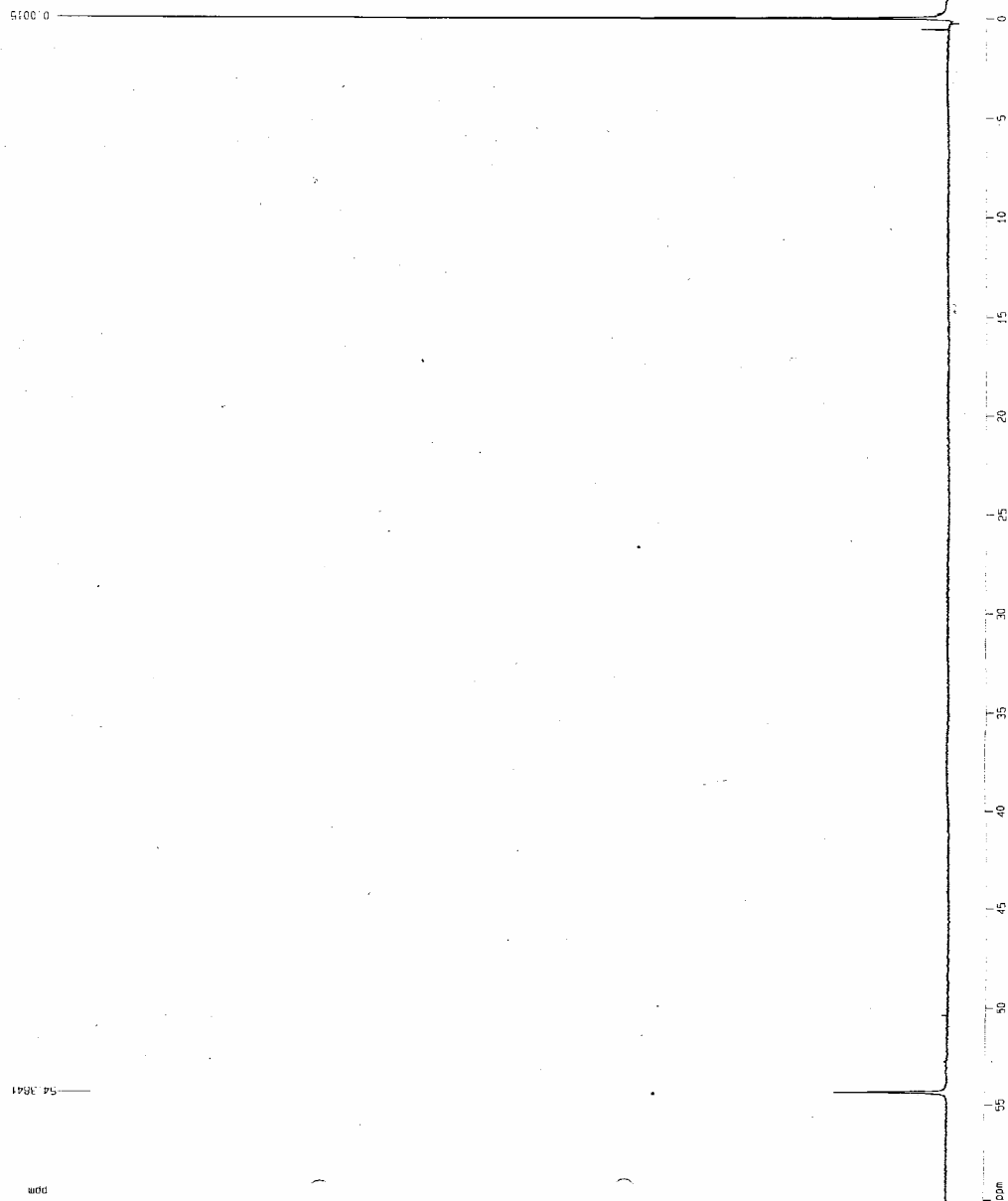
F2 - Acquisition Parameters
Date_ 20071220
Time 22.07
INSTRUM spect
PROBHD 5 mm DNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 6435.065 Hz
FIDRES 0.350330 Hz
AQ 0.304672 sec
RG 512
RW 7.700 usec
DE 5.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.8995998 sec
MCREST 0.00000000 sec
MCMRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 31P
P1 6.90 usec
PL1 -4.00 dB
SFO1 161.9350920 MHz

===== CHANNEL f2 =====
CPOB22 waltz16
NUC2 1H
P2 100.00 usec
PL2 4.00 dB
PL3 17.00 dB
PL4 17.00 dB
SFO2 400.0316001 MHz

F2 - Processing parameters
SI 32768
SF 161.9350928 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 32.00 cm
CY 50.00 cm
FIP 60.000 ppm
F1 9716.10 Hz
F2 1.000 ppm
F3 161.93 Hz
PITCH 1.00000000 mm/cm
WZCM 308.66860 Hz/cm



³¹P NMR of 31