## **Supplementary Materials:**

## Palladium-Catalyzed Allylation/Benzylation of *H*-Phosphinate Esters With Alcohols

Anthony Fers-Lidou, Olivier Berger and Jean-Luc Montchamp

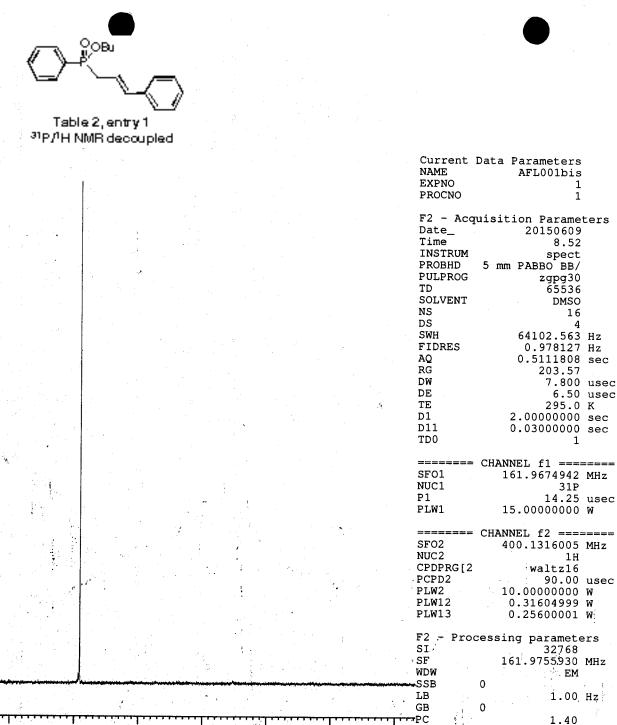
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## **General Chemistry:**

 $^{1}$ H NMR spectra were recorded on a 400-MHz Bruker Avance spectrometer (Bruker Daltonics Inc., Billerica, Massachusetts, USA). Chemical shifts for  $^{1}$ H NMR spectra (in parts per million) relative to internal tetramethylsilane (Me<sub>4</sub>Si,  $\delta$  = 0.00 ppm, s = singulet, d = doublet, m = multiplet, dd = doublet of doublets, dm = doublet of multiplets, quint. = quintuplet, t = triplet, ddd = doublet of doublet of doublets, dt = doublet of triplets, ddt = doublet of doublet of triplets, ) with deuterated chloroform.  $^{13}$ C NMR spectra were recorded at 75.5 or 101 MHz. Chemical shifts for  $^{13}$ C NMR spectra are reported (in parts per million) relative to CDCl<sub>3</sub> ( $\delta$  = 77.0 ppm).  $^{31}$ P NMR spectra were recorded at 121.5 or 162 MHz, and chemical shifts reported (in parts per million) relative to external 85% phosphoric acid ( $\delta$  = 0.0 ppm). Flash chromatography experiments were carried out on Silica Gel premium Rf grade (60 Å, 40–75 μm, Sorbent Technologies, Norcross, Georgia, USA). Ethyl acetate/hexane mixtures or dichloromethane/acetone were used as the eluent for chromatographic purifications. Whatman TLC plates (60 Å, thermo Fisher Scientific, Waltham, Massachusetts, USA) were visualized by UV or immersion in permanganate potassium (3 g KMnO<sub>4</sub>, 20 g K<sub>2</sub>CO<sub>3</sub>, 5 mL 5% aq. NaOH and 300 mL of water) followed by heating. High resolution mass spectra (HRMS) were obtained either by direct probe (EI/CI) and analyzed by magnetic sector, or by electrospray using a TOF analyzer.

## Reagent and solvents:

All starting materials were purchased from commercial sources and used as received. The solvents were distilled under  $N_2$  and dried according to standard procedures (THF from Na/ benzophenone ketyl; DMF from MgSO<sub>4</sub>; CH<sub>3</sub>CN, toluene and dichloromethane from CaH<sub>2</sub>).



100 90 80 70 60 50 40 30 20 10 0 pp

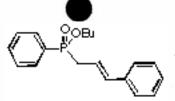
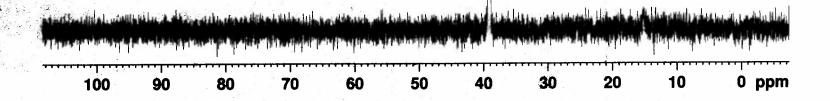


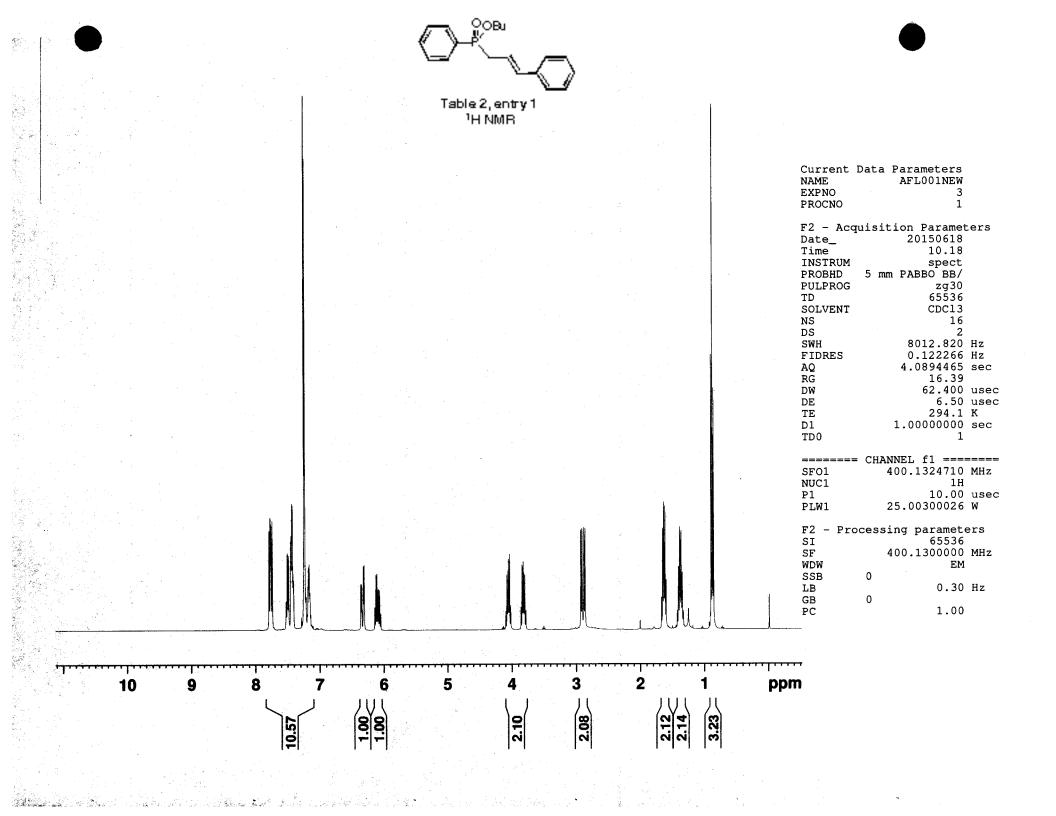
Table 2, entry 1 <sup>31</sup>P/<sup>1</sup>H NMR coupled

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NAME	,	AFL001bis
EXPNO		2
PROCNO		1

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SFO1	161.9674942	MHz
NUC1	31P	
P1	14.25	usec
PLW1	15.00000000	W

·2 –	Processing	paramete	ers
SI		32768	
SF	161	.9755930	MHz
WDW		EM	
SSB	0		
LB		1.00	Ηz
3B	0		
PC		1.40	

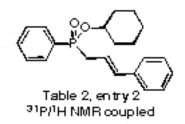




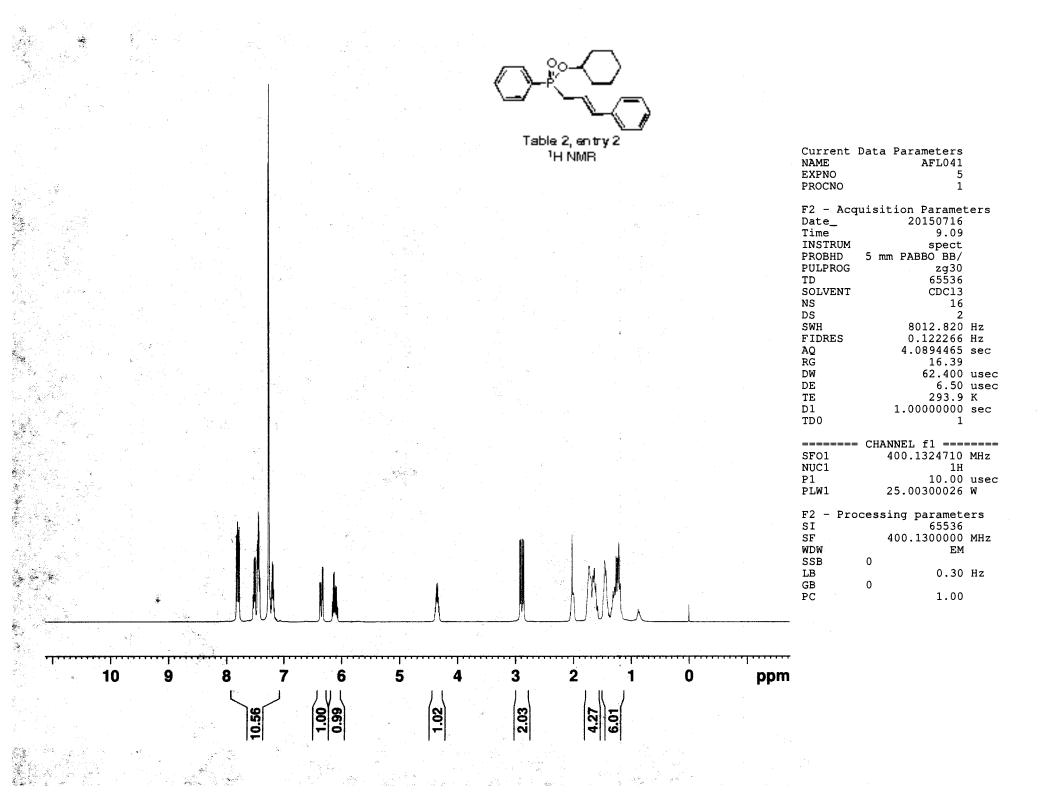
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Table 2, entry 2
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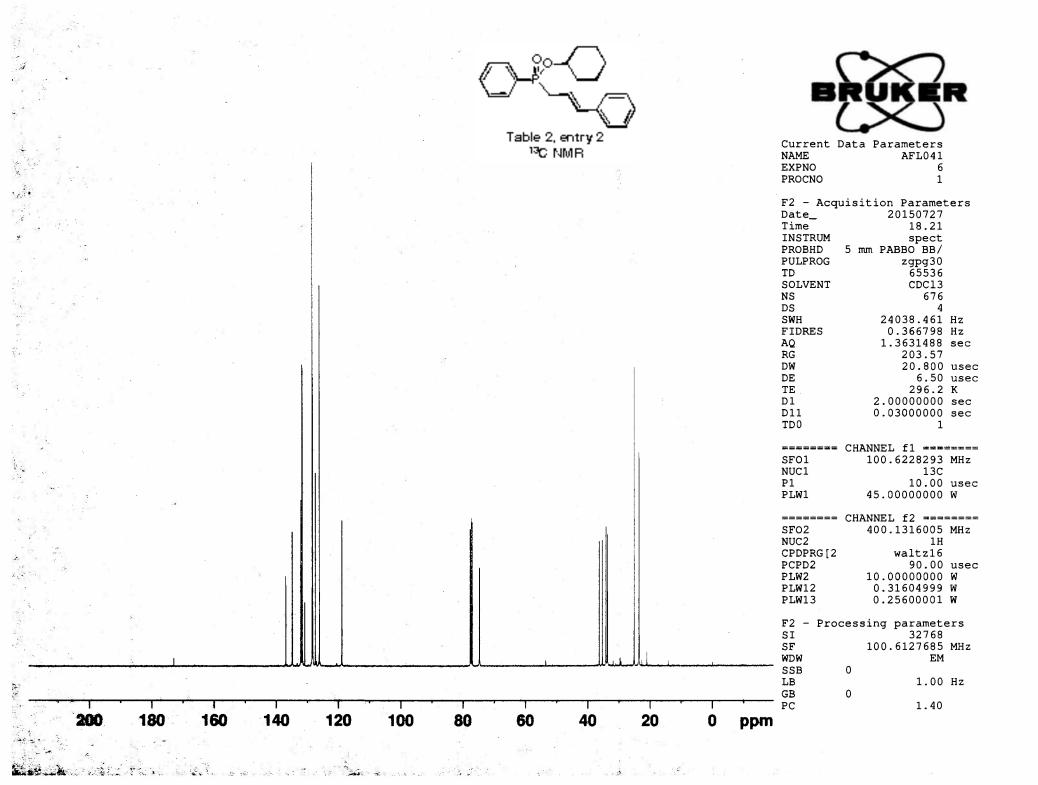
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Current Data Parameters
NAME
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EXPNO
                      3
                      1
PROCNO
F2 - Acquisition Parameters
Date_
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Time
                   9.02
INSTRUM
                  spect
PROBHD
         5 mm PABBO BB/
PULPROG
                 zgpg30
                  65536
SOLVENT
                Acetone
NS
                     16
DS
              64102.563 Hz
SWH
FIDRES
               0.978127 Hz
              0.5111808 sec
ΑQ
RG
                 203.57
                  7.800 usec
DW
DΕ
                   6.50 usec
ŤΕ
                   294.4 K
D1
             2.00000000 sec
             0.03000000 sec
D11
======= CHANNEL f1 ======
SF01
            161.9674942 MHz
                     31P
NUC1
P1
                  14.25 usec
PLW1
            15.00000000 W
====== CHANNEL f2 =======
SFO2
            400.1316005 MHz
                      1H
NUC2
CPDPRG[2
                waltz16
                   90.00 usec
PCPD2
            10.00000000 W
PLW2
             0.31604999 W
PLW12
PLW13
             0.25600001 W
F2 - Processing parameters
                   32768
SI
            161.9755930 MHz
SF
                      EΜ
WDW
SSB
         0
                    1.00 Hz
LB
GB
                    1.40
PC
```

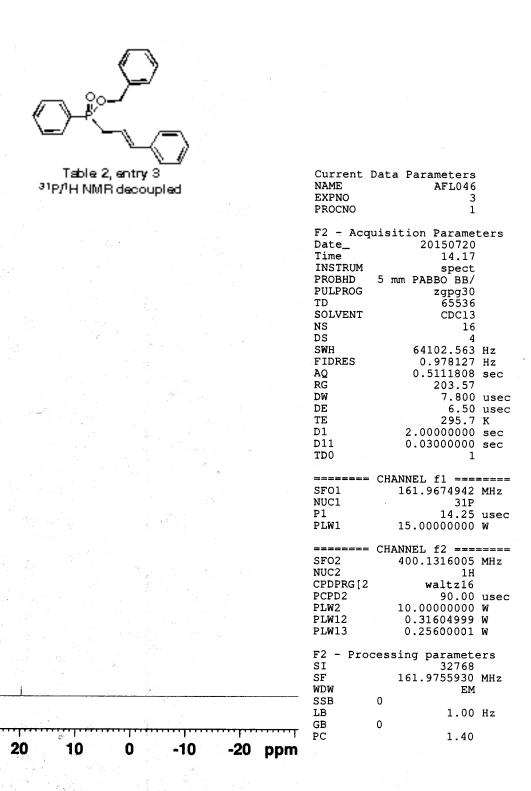
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```
Current Data Parameters
NAME
EXPNO
                      4
PROCNO
                      1
F2 - Acquisition Parameters
Date_
               20150716
Time
                   9.04
INSTRUM
                  spect
PROBHD
         5 mm PABBO BB/
PULPROG
TD
                  65536
SOLVENT
                Acetone
NS
                     19
DS
              64102.563 Hz
SWH
FIDRES
               0.978127 Hz
              0.5111808 sec
ΑQ
                 203.57
RG
                  7.800 usec
DW
DE
                   6.50 usec
ΤE
                  294.0 K
D1
             2.00000000 sec
TD0
====== CHANNEL f1 ======
SF01
            161.9674942 MHz
NUC1
                    31P
                  14.25 usec
Ρ1
            15.00000000 W
PLW1
F2 - Processing parameters
SI
                  32768
            161.9755930 MHz
SF
WDW
                     ΕM
SSB
LB
                   1.00 Hz
         0
GB
PC
                   1.40
```







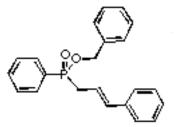
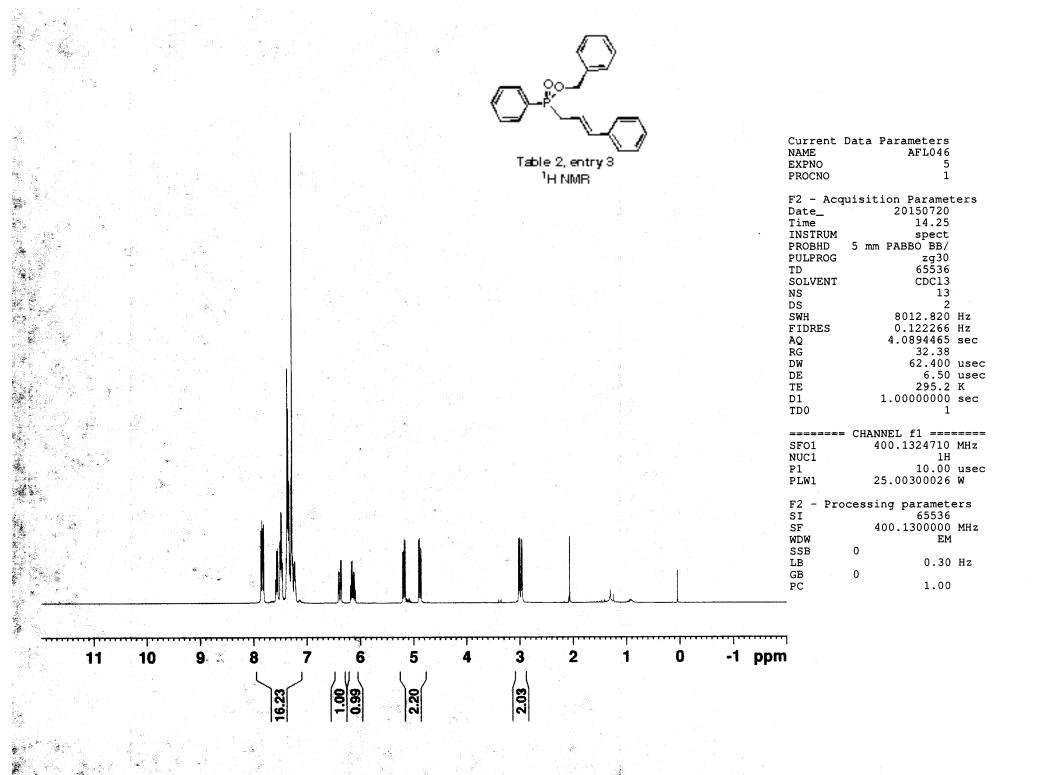


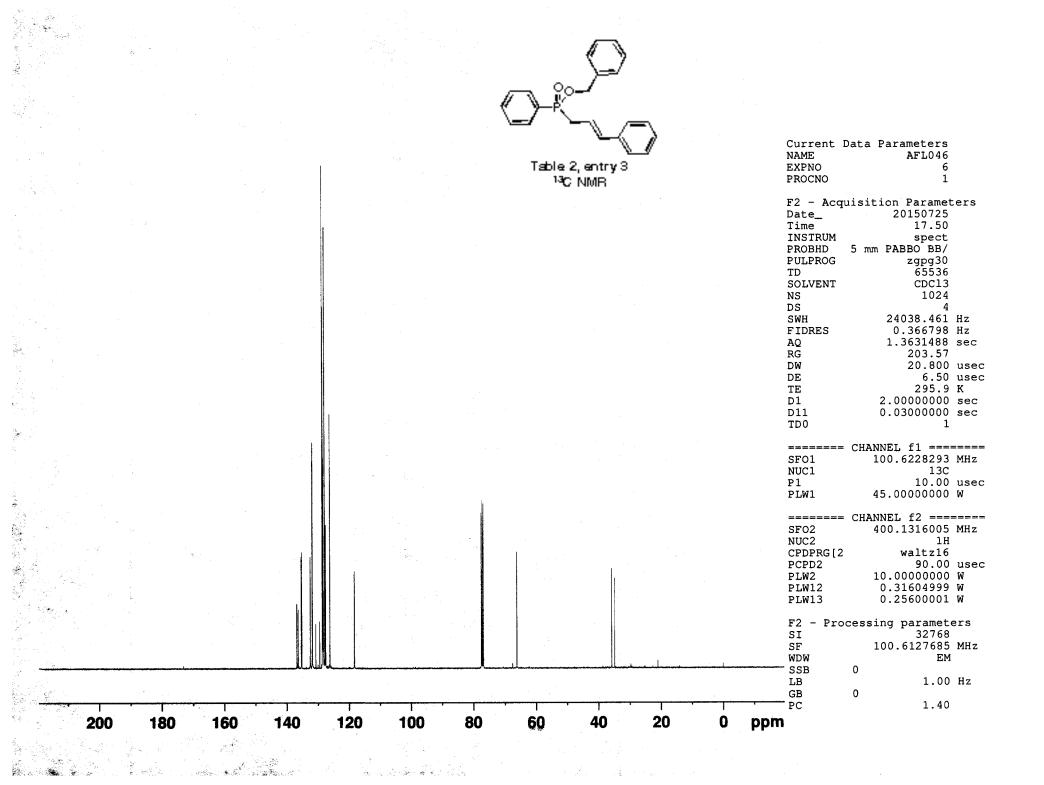
Table 2, entry 3 <sup>31</sup>P/<sup>1</sup>H NMR coupled Current Data Parameters NAME AFL046 EXPNO 4 PROCNO 1

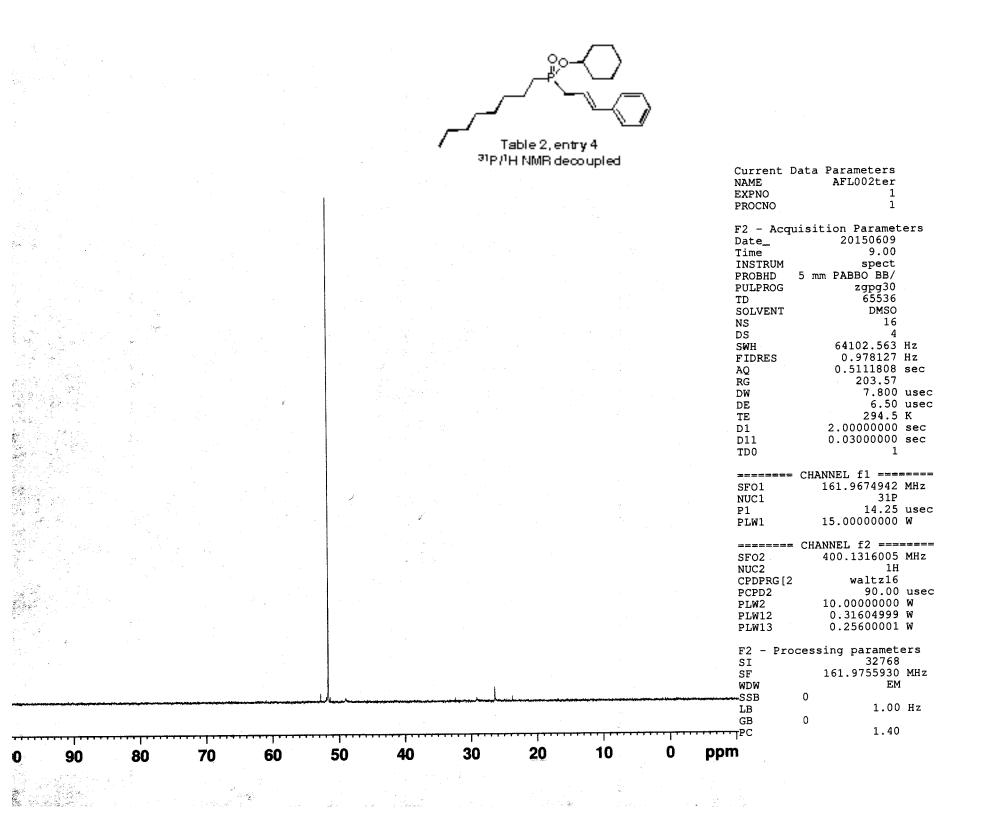
F2 - Acquisition Parameters Date\_ 20150720 14.19 Time INSTRUM spect PROBHD 5 mm PABBO BB/ PULPROG TD 65536 CDC13 SOLVENT NS 16 DS SWH 64102.563 Hz 0.978127 Hz FIDRES ΑQ 0.5111808 sec 203.57 DW 7.800 usec DΕ 6.50 usec 295.2 K ΤE 2.00000000 sec D1 TD0

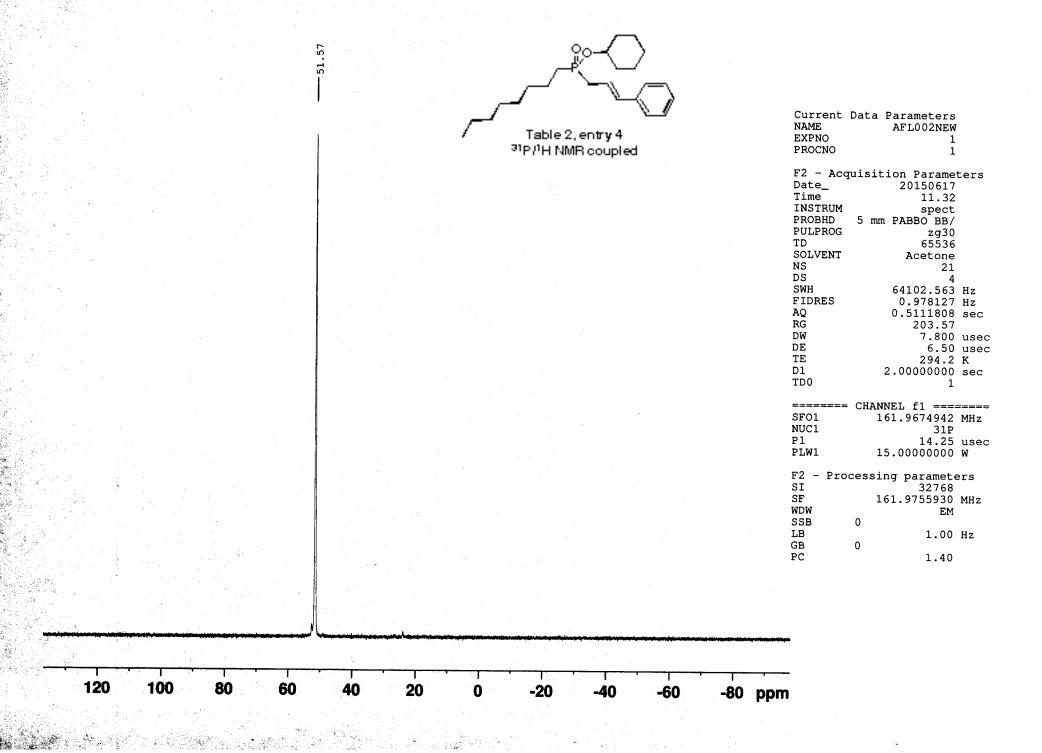
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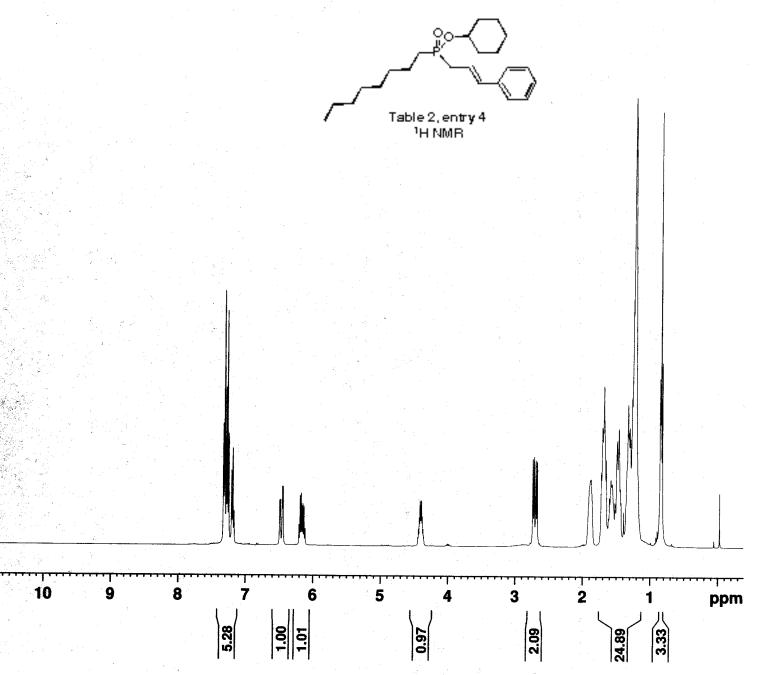
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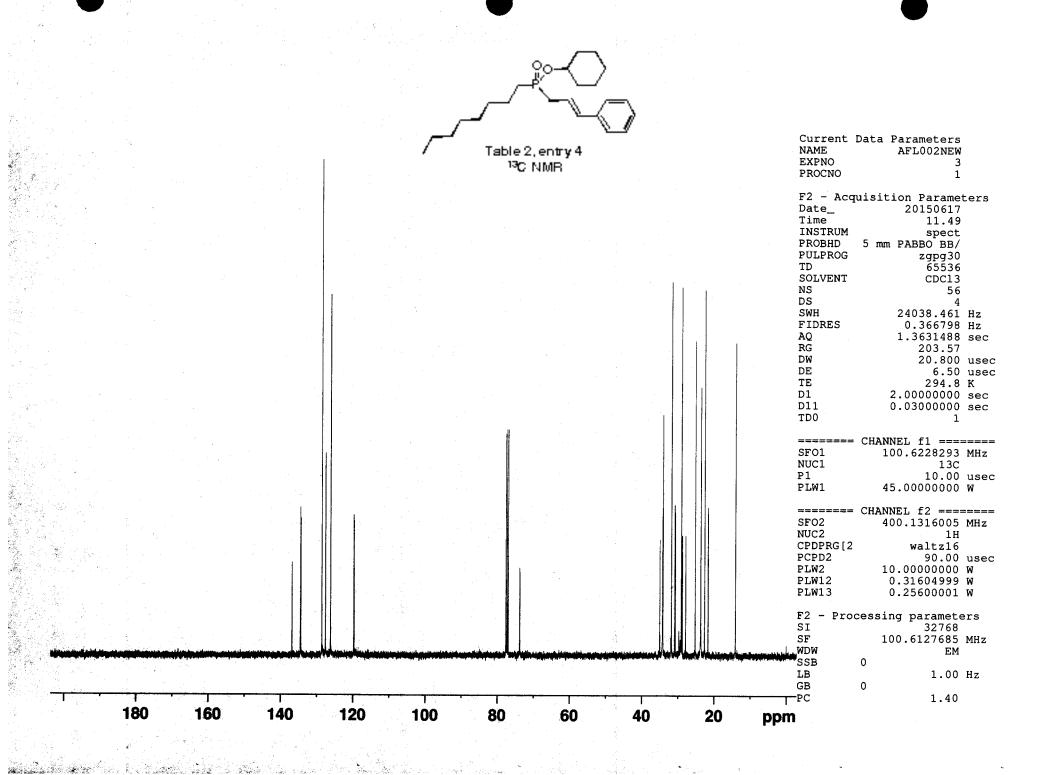


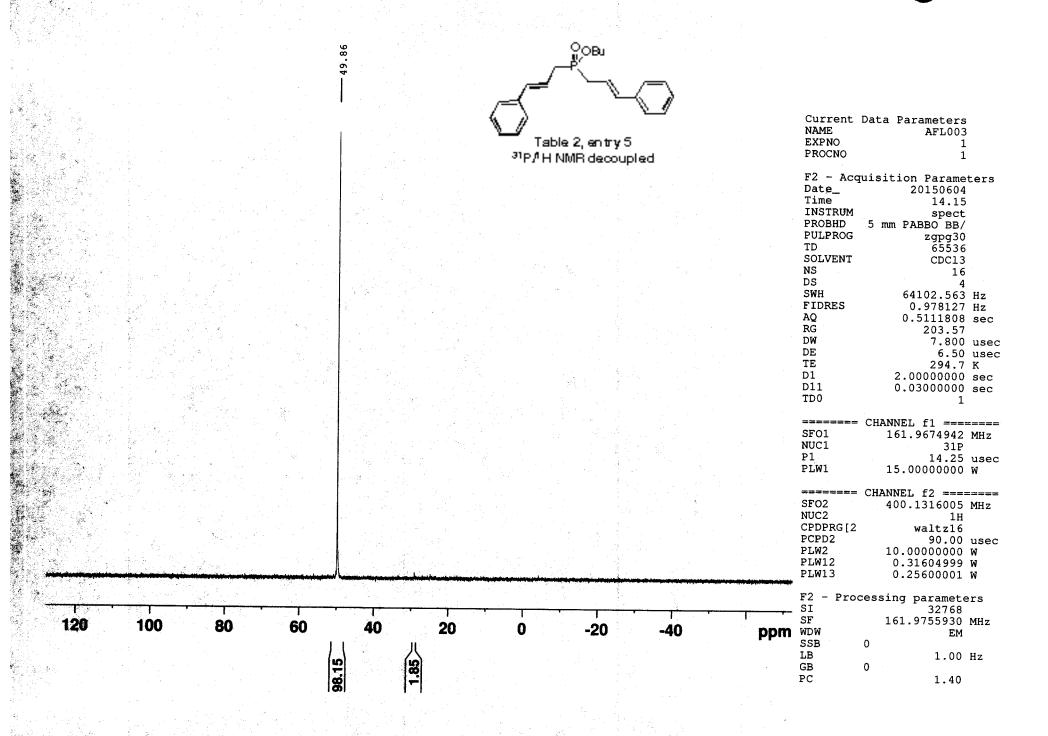






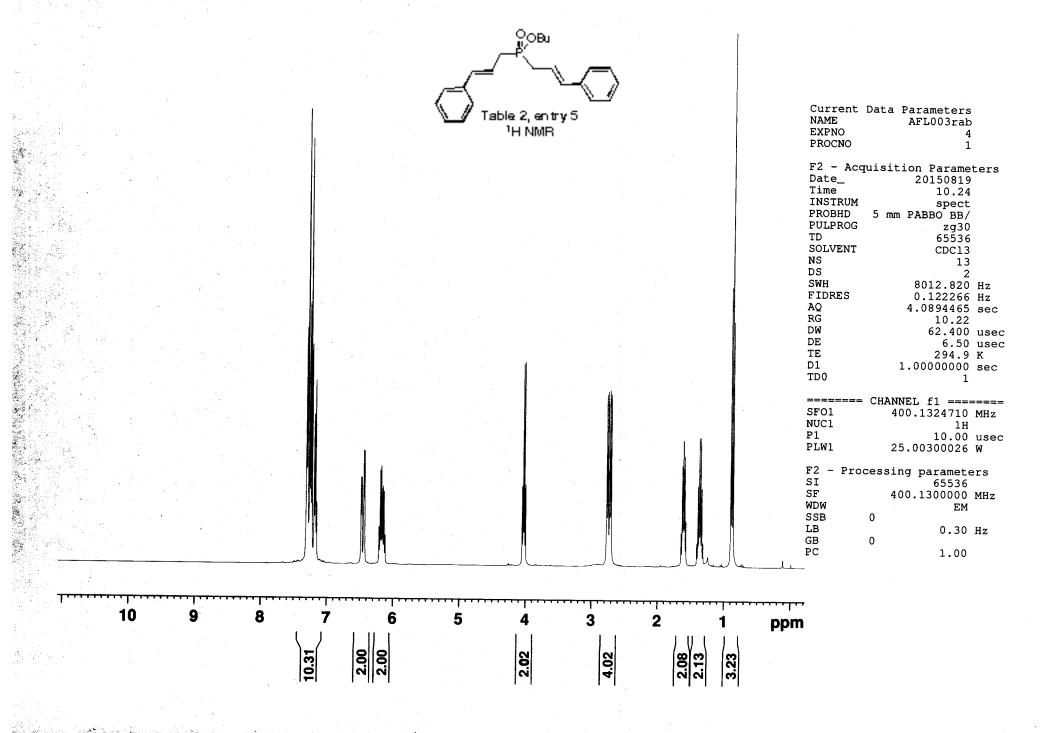
Current Data Parameters NAME AFL002NEW **EXPNO** PROCNO 1 F2 - Acquisition Parameters Date\_ 20150617 Time 11.38 INSTRUM spect 5 mm PABBO BB/ PROBHD PULPROG zg30 TD 65536 CDC13 SOLVENT NS 16 DS 2 SWH 8012.820 Hz FIDRES 0.122266 Hz ΑQ 4.0894465 sec RG 11.05 62.400 usec DW 6.50 usec ΤE 294.1 K D1 1.00000000 sec TD0 1 ====== CHANNEL f1 ====== SFO1 400.1324710 MHz NUC1 1H Ρ1 10.00 usec PLW1 25.00300026 W F2 - Processing parameters SI 65536 400.1300000 MHz SF WDW EM SSB LB 0.30 Hz GB PC 1.00

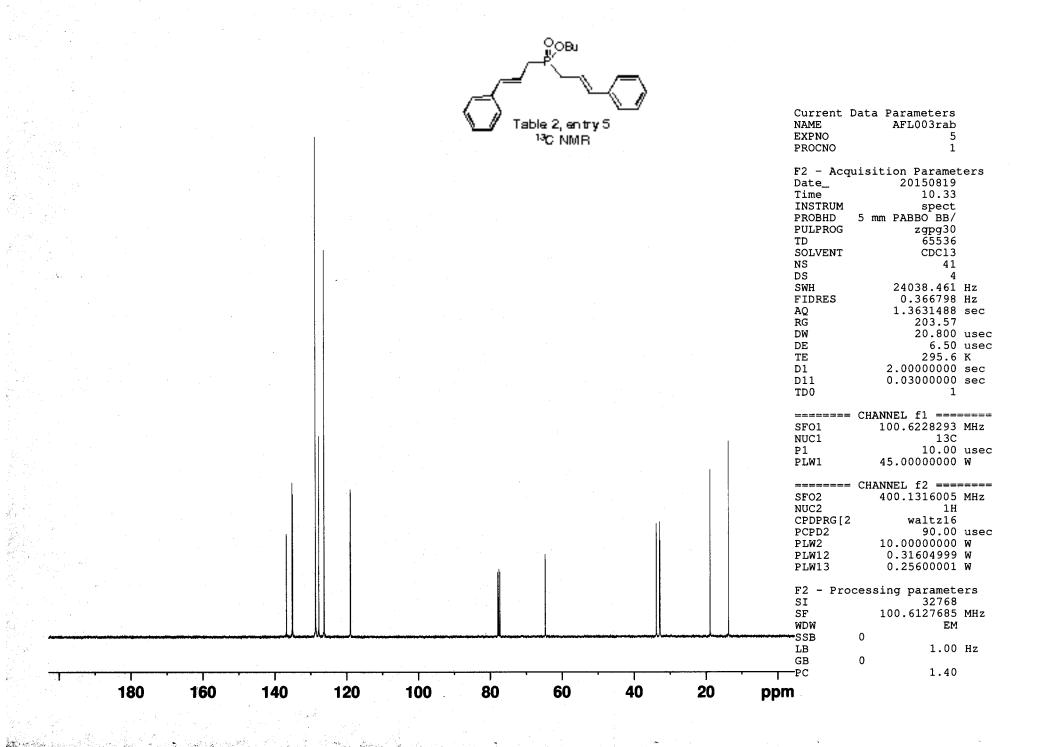


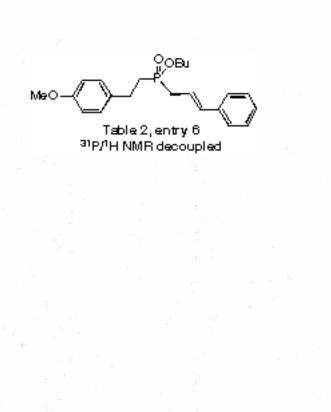


```
OBu
Table 2, entry 5
<sup>31</sup>P/<sup>1</sup>H NMR coupled
```

Current D NAME EXPNO PROCNO	oata Paramete AFLO	
Date_ Time INSTRUM PROBHD PULPROG TD SOLVENT NS DS		04 17 ct B/ 30 36 13 32 4
SWH FIDRES AQ RG	64102.5 0.9781 0.51118 203.	27 Hz 08 sec
DW DE TE D1 TD0		
SFO1 NUC1 P1		===== 42 MHz 1P 25 usec
PLW1	15.000000	
SI SF WDW		68
SSB LB GB PC	0	00 Hz 40







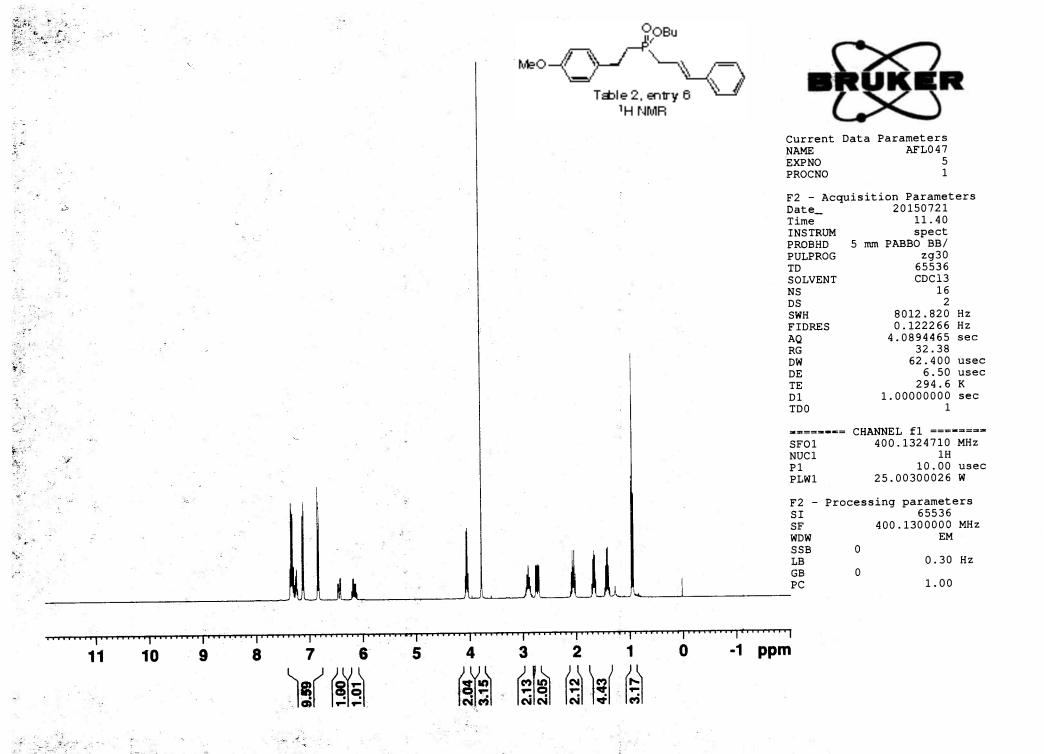
ppm

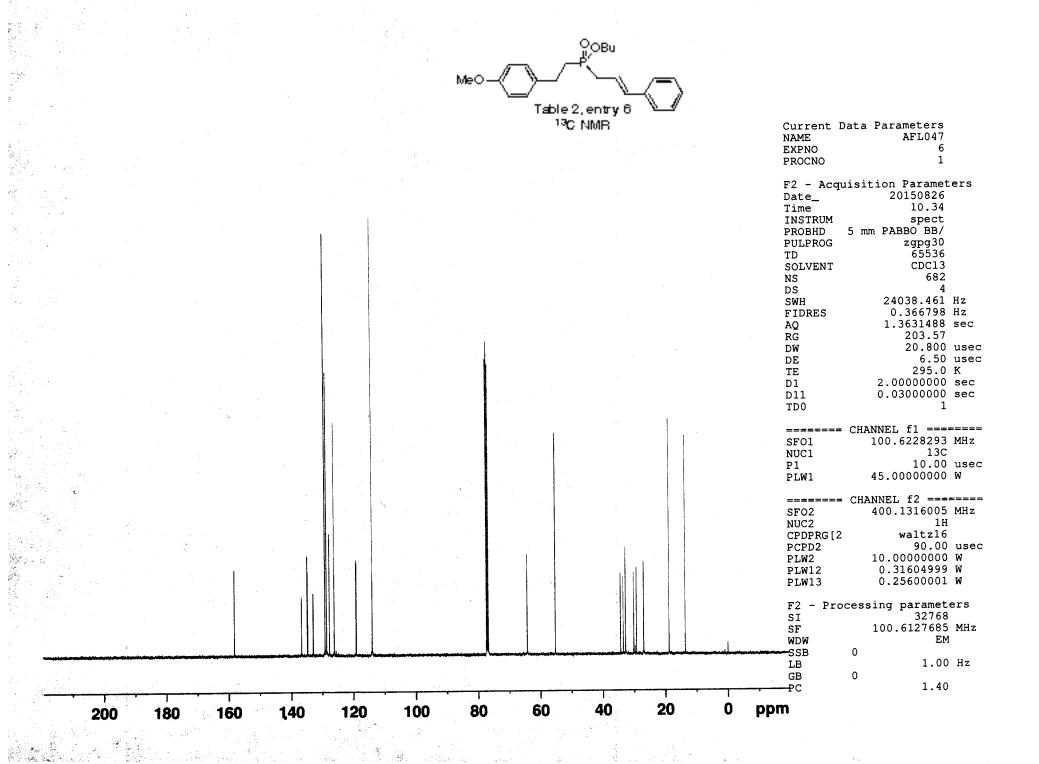


Current NAME EXPNO PROCNO	Data		AFL04	
F2 - Acc Date_ Time INSTRUM PROBHD PULPROG TD SOLVENT NS DS SWH FIDRES AQ RG DW		203 a PABE 2 6410 0.9 0.51	150722 11.33 spect 30 BB <sub>0</sub> 2gpg30 65536 CDC13 16 202.563 778127 11808	l l l l l l l l l l l l l l s sec
DE TE D1 D11 TD0			6.50 295.0 00000 00000 1	K sec sec
SF01 NUC1 P1 PLW1	1	NEL f 61.96 5.000	74942 31P 14.25	MHz usec
SFO2 NUC2 CPDPRG[2 PCPD2 PLW2 PLW12 PLW13	1		16005 1H 1tz16 90.00 00000 04999	MHz usec W
F2 - Prod SI SF WDW SSB LB GB	cessin 10 0		ramet 32768 55930 EM	MHz

1.40

Curre NAME EXPNO PROCN		ata	Par			ers 047 4 1		
F2 - Date_ Time INSTR PROBH PULPR TD SOLVE NS DS	.UM D 5	isit 5 mr	2	201	507 11 spe 0 1 20	amet 721 .33 ect 3B/ g30 536 C13 30	ers	:
SWH FIDRE AQ RG DW DE TE D1	s		0.	).9 .51 2	78: 11: 203 7.: 6	563 127 808 .57 800 .50 4.6	use use K	e c
TD0  SF01 NUC1 P1 PLW1	:=== (			96	74 14	1 942 31P .25	MHz use	:
F2 - SI SF WDW SSB	Proce		_	-	32	mete 768 930 EM		Z
LB GB PC	(	)				.00	Ηz	





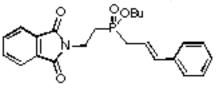


Table 2, entry 7 <sup>31</sup>P/<sup>1</sup>H NMR decoupled



```
Current Data Parameters
           OB 1977 pure
NAME
EXPNO
                      1
                      1
PROCNO
F2 - Acquisition Parameters
Date_
               20150409
                  17.05
Time 
INSTRUM
                  spect
PROBHD
         5 mm PABBO BB/
PULPROG
                 zgpg30
TD
                  65536
                  CDC13
SOLVENT
                     16
NS
DS
                      4
              64102.563 Hz
SWH
FIDRES
               0.978127 Hz
              0.5111808 sec
ΑQ
RG
                 203.57
DW
                  7.800 usec
DΕ
                   6.50 usec
                  294.4 K
ΤE
D1
             2.00000000 sec
D11
             0.03000000 sec
TDO
====== CHANNEL f1 ======
SF01
            161.9674942 MHz
                    31P
NUC1
P1
                  14.25 usec
PLW1
            15.00000000 W
====== CHANNEL f2 ======
SFO2
            400.1316005 MHz
                     1H
NUC2
CPDPRG[2
                waltz16
PCPD2
                  90.00 usec
PLW2
            10.00000000 W
PLW12
             0.31604999 W
             0.25600001 W
PLW13
F2 - Processing parameters
SI
                  32768
            161.9755930 MHz
SF
WDW
                     EΜ
-SSB
         0
                   1.00 Hz
LB
GB
         0
```

1.40

TPC.

100 90 80 70 60 50 40 30 20 10 0 ppm

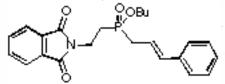


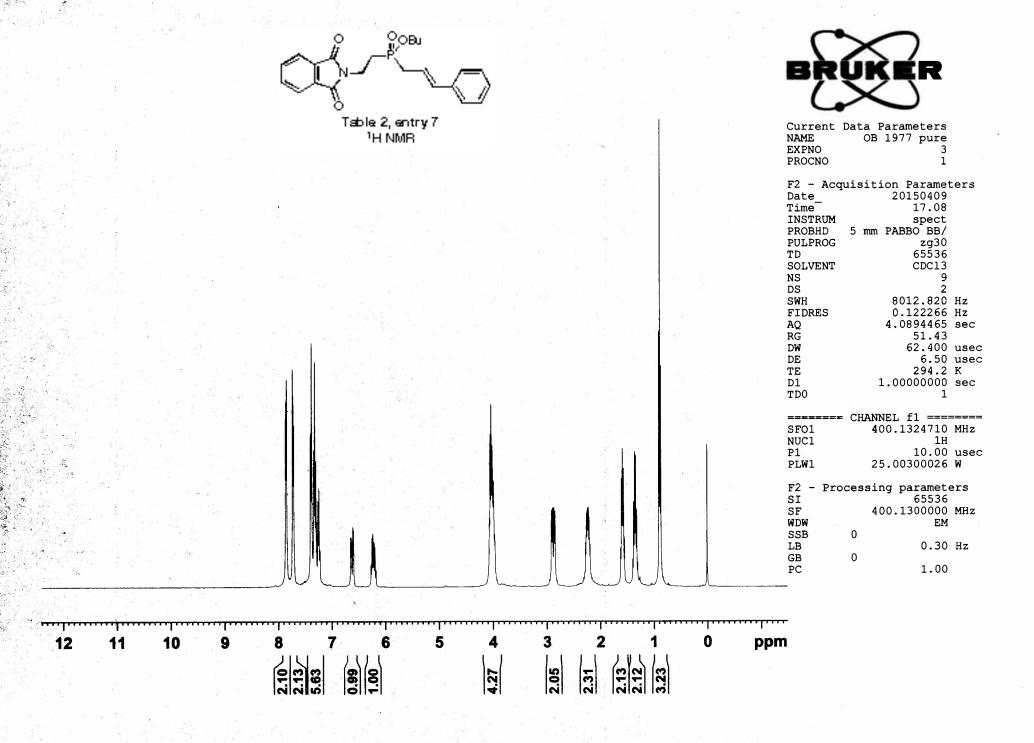
Table 2, entry 7 <sup>31</sup>P/<sup>1</sup>H NMR coupled

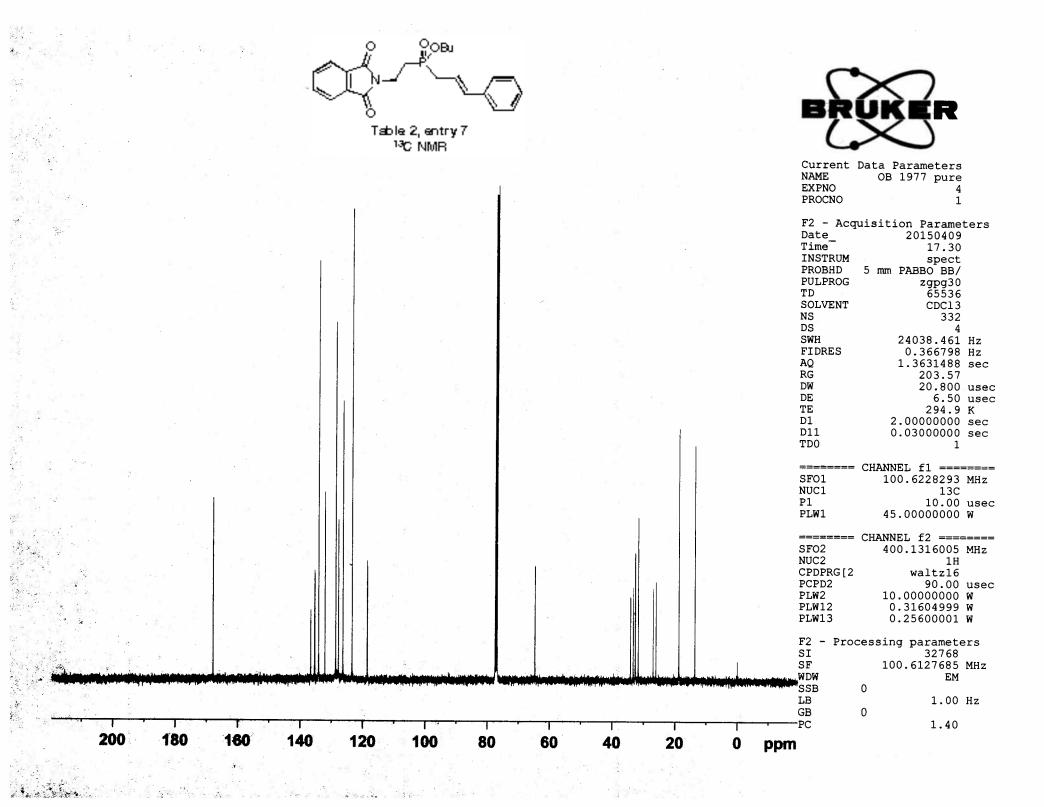


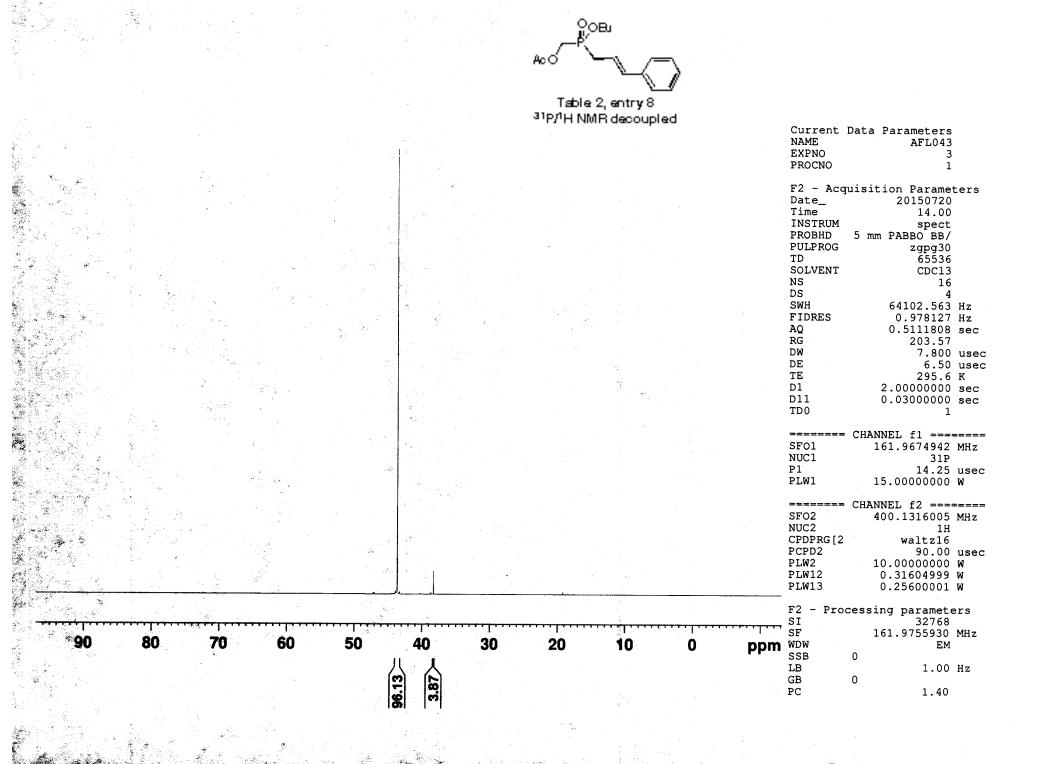
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EXPNO 2
PROCNO 1

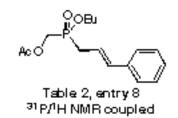
F2 - Acqu	ıis	siti	lon Pa	rame	ters
Date			2015		
Time Time			1	7.07	
INSTRUM			S	pect	
PROBHD	5	mm	PABBO	BB/	
PULPROG			:	zq30	
TD			6	5536	
SOLVENT			CI	DC13	
NS				32	
DS				4	
SWH			64102	.563	Ηz
FIDRES			0.978	3127	Hz
AQ			0.5113	1808	sec
RG			203	3.57	
D <b>W</b>			7.	800	usec
DE			(	5.50	usec
ΓE			29	94.1	K
D1		2	.00000	0000	sec
rd0				1	

=======	CHANNEL f1 ====	====
SFO1	161.9674942	MHz
NUC1	31P	
P1	14.25	usec
PLW1	15.00000000	W



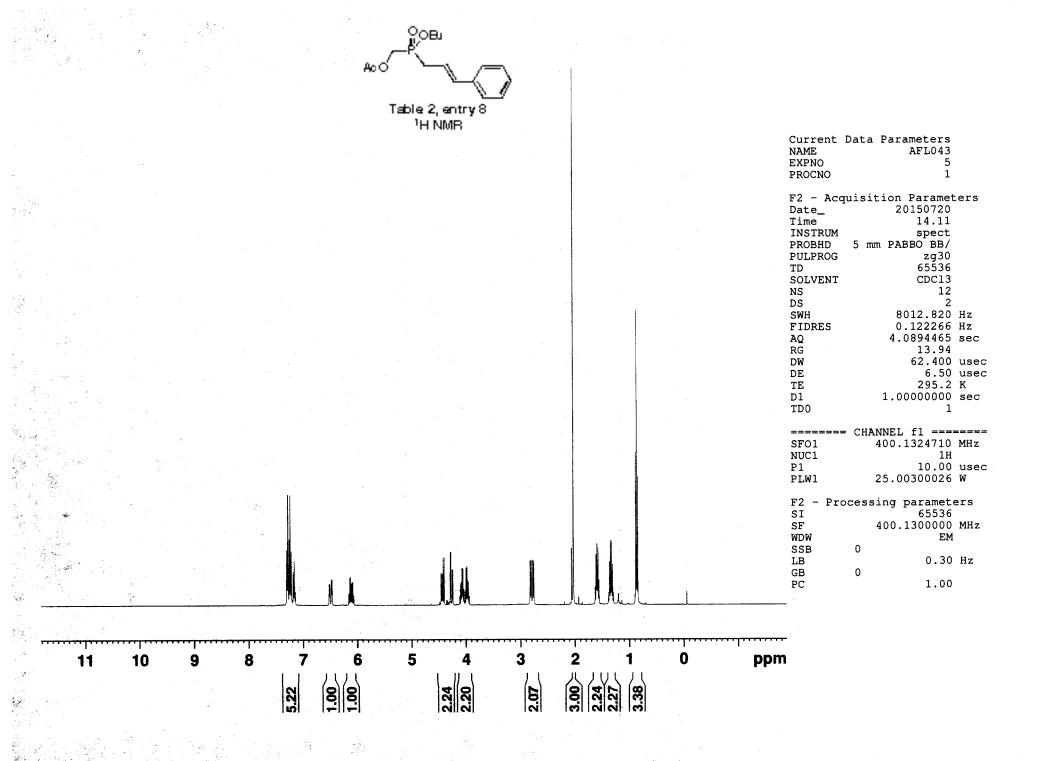


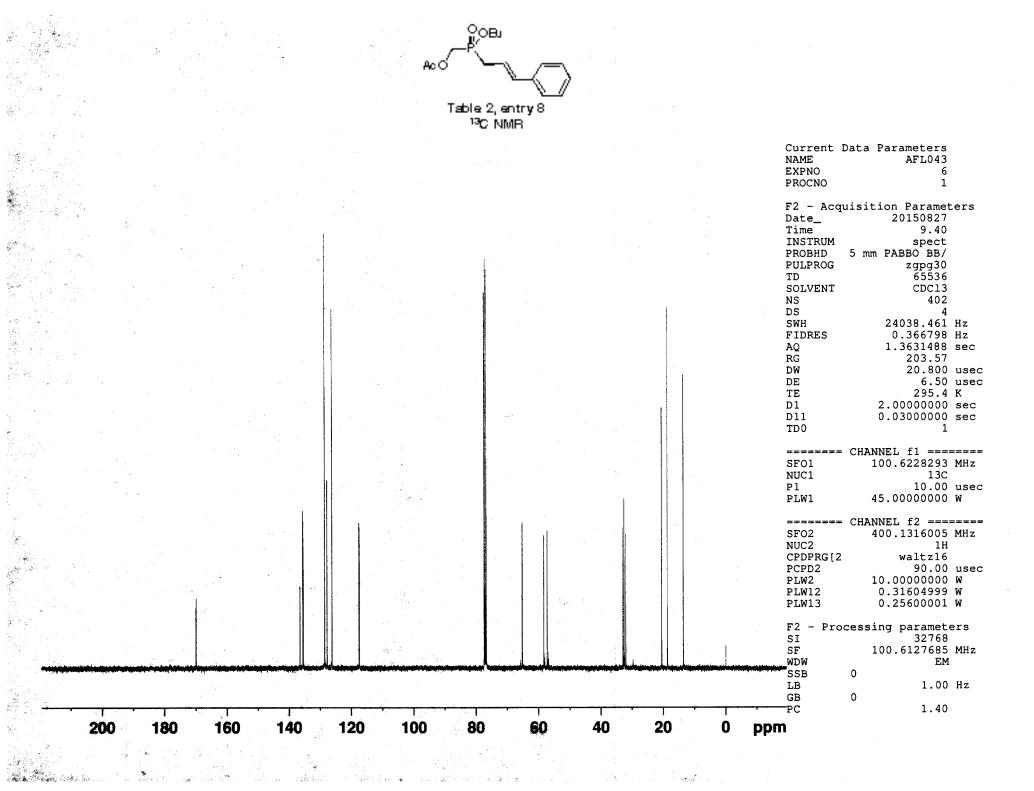




NAME		A	FL043	
EXPNO PROCNO			1	
PROCNO  F2 - Acqu Date_ Time INSTRUM PROBHD PULPROG TD SOLVENT NS DS SWH FIDRES AQ RG RG DW DE	isit: 5 mm	201 PABB 6410 0.9 0.51	arame 50720 14.04 spect O BB/ zg30 65536 CDC13 32 2.563 78127 11808	Hz Hz sec usec
TE			295.2	K
D1 TD0		2.000	00000	sec
SFO1 NUC1 P1 PLW1	1	61.96	1 === 74942 31P 14.25	MHz usec
F2 - Prod SI	cessi	ng pa	ramet 32768	
SF WDW	1	61.97	55930 EM	MHz
SSB LB	0		1.00	-
GB	0			
PC			1.40	1

Current Data Parameters





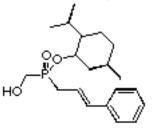


Table 2, entry 9 <sup>31</sup>P/H NMR decoupled

```
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EXPNO
PROCNO
F2 - Acquisition Parameters
Date_ 20150825
Time
INSTRUM
         spect
5 mm PABBO BB/
PROBHD
                  zgpg30
65536
CDC13
PULPROG
TD
SOLVENT
NS
                       16
DS
               64102.563 Hz
0.978127 Hz
SWH
FIDRES
AQ
RG
DW
               0.5111808 sec
                   203.57
                    7.800 usec
DE
TE
                     6.50 usec
                    294.9 K
D1
              2.00000000 sec
D11
              0.03000000 sec
====== CHANNEL f1 ======
             161.9674942 MHz
SF01
NUC1
                     31P
P1
                    14.25 usec
             15.00000000 W
PLW1
PCPD2
                   90.00 usec
PLW2
             10.00000000 W
0.31604999 W
PLW12
PLW13
              0.25600001 W
F2 - Processing parameters
             32768
161.9755930 MHz
SI
SF
WDW
                       EM
SSB
LB
                     1.00 Hz
GB
                     1.40
```

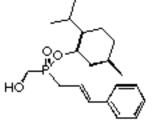


Table 2, entry 9 31P#HNMR coupled

Current Data Parameters NAME OB 2071 1st crystallization EXPNO PROCNO F2 - Acquisition Parameters Date\_ 20150825 Time 10.10 INSTRUM spect PROBHD PULPROG zg30 65536 TD SOLVENT NS DS 27 64102.563 Hz 0.978127 Hz 0.5111808 sec FIDRES ΑQ 203.57 ŔĠ DW DE 7.800 usec 6.50 usec TE 294.5 K D1 TD0 2.00000000 sec 1 ====== CHANNEL f1 ====== SF01 161.9674942 MHz NUC1 P1 14.25 usec 15.00000000 W PLW1 F2 - Processing parameters SI 32768 SF 161.9755930 MHz SF WDW SSB LB GB PC EM 1.00 Hz 0

1.40

60 **50** 30 ppm

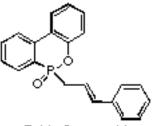


Table 2, entry 11
<sup>31</sup>P/IHNMR decoupled

Current Data Parameters AFL005 NAME **EXPNO** PROCNO F2 - Acquisition Parameters 20150605 Date\_ Time 16.35 INSTRUM spect PROBHD 5 mm PABBO BB/ PULPROG zgpg30 65536 TD CDC13 SOLVENT NS DS 64102.563 Hz SWH 0.978127 Hz FIDRES 0.5111808 sec ΑQ 203.57 RG 7.800 usec DW DE 6.50 usec 294.7 K ΤE 2.00000000 sec D1 0.03000000 sec D11 ====== CHANNEL f1 ====== SF01 161.9674942 MHz NUC1 31P 14.25 usec P1 PLW1 15.00000000 W ======= CHANNEL f2 ======= 400.1316005 MHz SF02 NUC2 1H waltz16 CPDPRG[2 90.00 usec PCPD2 10.00000000 W PLW2 0.31604999 W PLW12 0.25600001 W PLW13 F2 - Processing parameters SI 32768 161.9755930 MHz SF ΕM WDW SSB 1.00 Hz LBGB

1.40

PC

90 80 70 60 50 40 30 20 10 0 -10 -20 -30 ppm

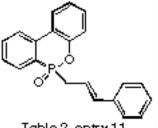
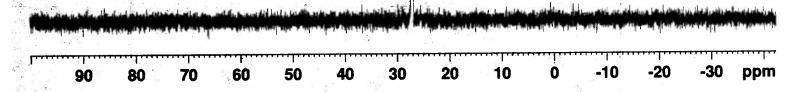


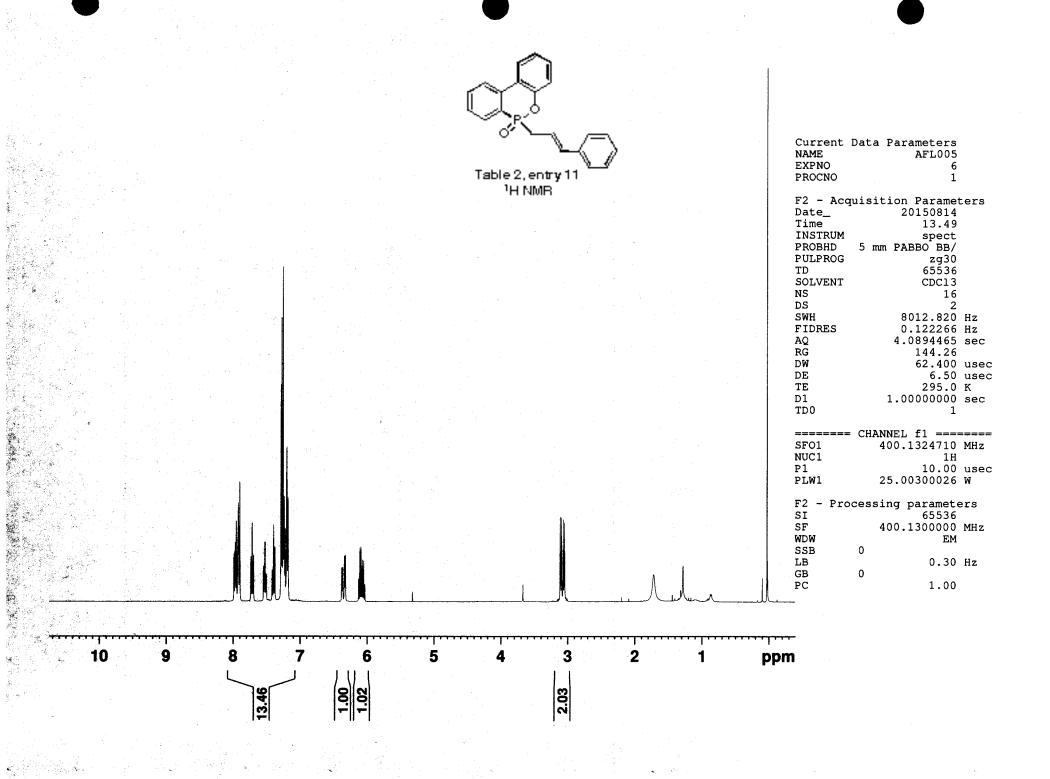
Table 2, entry 11
<sup>31</sup>P/<sup>1</sup>H NMR coupled

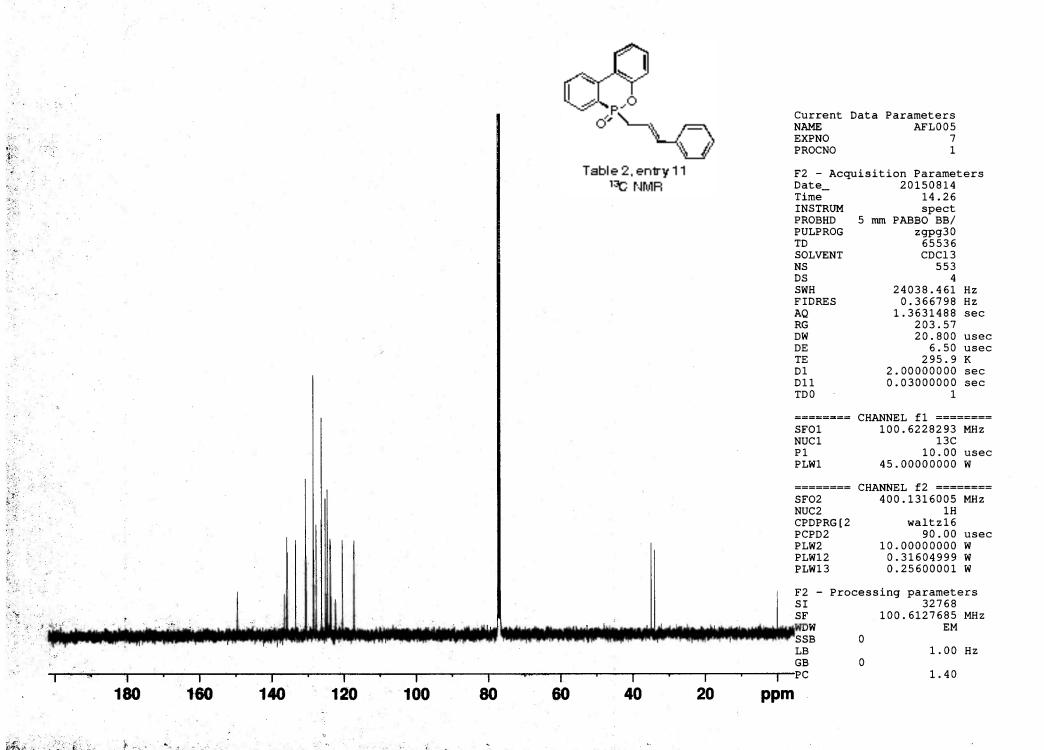
Current Data Parameters NAME AFL005 EXPNO 2 PROCNO 1

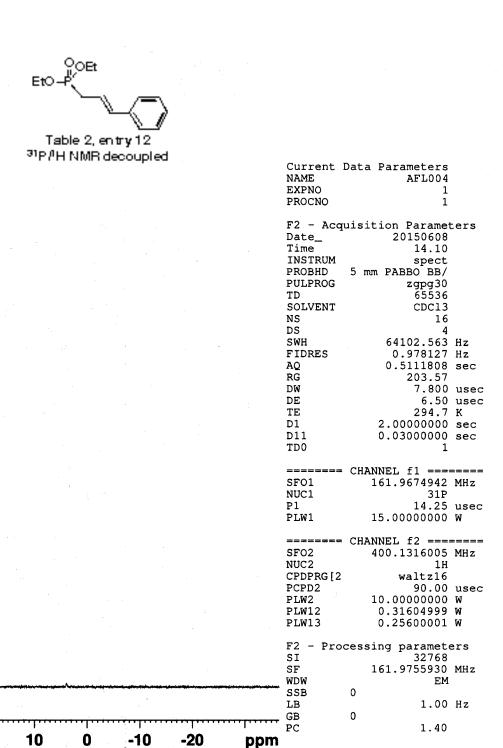
r	<u> </u>	Acq	uıs	51 C I	LOn 1	arai	uec	ers
D	ate_	_			20	1506	05	
Т	ime					16.	37	
Ι	NST	RUM				spe	ct	
Ρ	ROBI	HD	5	mm	PABI	30 B	B/	
Ρ	ULPI	ROG				zg	30	
Т	Ď					65 <u>5</u>	36	
S	OLV	ENT				CDC	13	
N	S						24	
D	S						4	
S	WH				641	02.5	63	Ηz
F	IDR!	ES			0.	9781	27	Ηz
Α	.O				0.5	1118	80	sec
	Ğ					203.	57	
D	W					7.8	00	usec
D	E					6.	50	usec
1	Έ					294	. 4	K
D	1				2.00	0000	00	sec
_	.D0						1	

##====##	CHANNEL f1 ====	====
SFO1	161.9674942	MHz
NUC1	31P	
P1	14.25	use
PT.W1	15.00000000	W





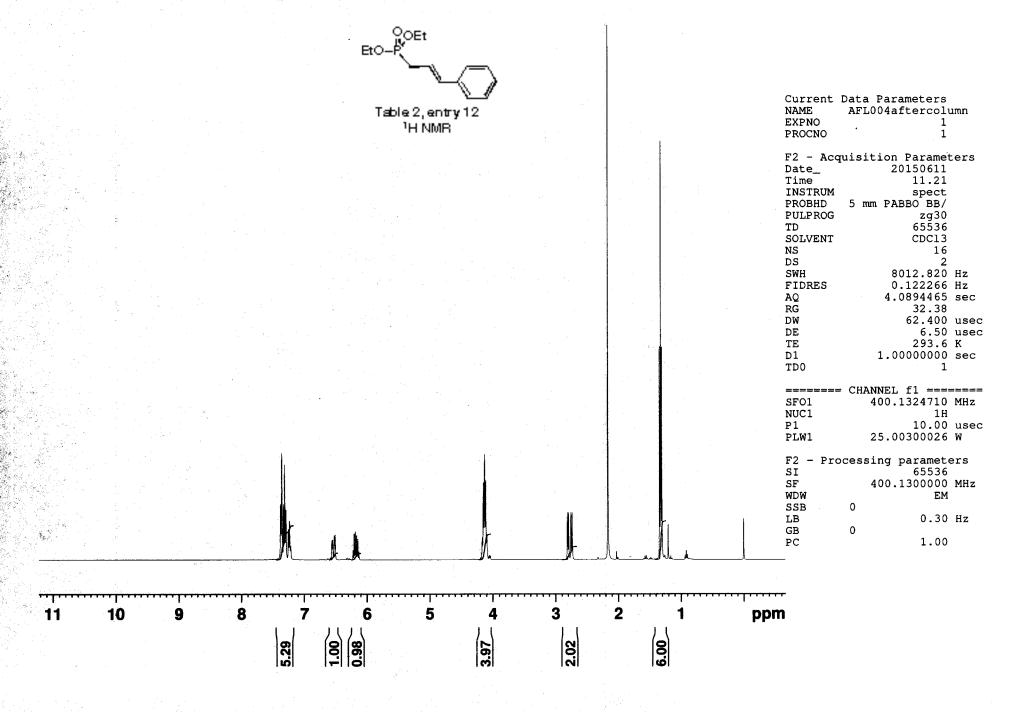


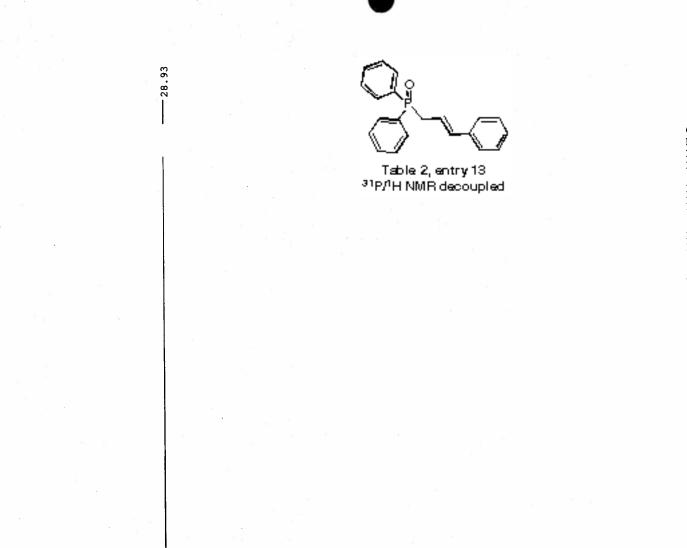


```
Table 2, entry 12
```

NAME EXPNO PROCNO	AFL004 2 1	
F2 - Acqu Date_ Time INSTRUM PROBHD PULPROG TD SOLVENT NS DS	uisition Parame 20150608 14.12 spect 5 mm PABBO BB/ zg30 65536 CDC13 32	ters
SWH FIDRES AQ RG DW DE TE D1 TD0	64102.563 0.978127 0.5111808 203.57 7.800	Hz sec usec usec K
SF01 NUC1 P1 PLW1	CHANNEL f1 === 161.9674942 31P 14.25 15.000000000	MHz usec
F2 - Proc SI SF WDW SSB	cessing paramet 32768 161.9755930 EM	MHz
LB GB PC	1.00	Hz

Current Data Parameters





-50

-100

-150

-200

ppm

50

100

F2 - Acquisition Parameters Date_ 20150615 Time 15.32 INSTRUM spect PROBHD 5 mm PABBO BB/ PULPROG 2930 TD 65536 SOLVENT Acetone NS 32 DS 4 SWH 64102.563 Hz FIDRES 0.978127 Hz AQ 0.5111808 sec RG 203.57 DW 7.800 usec DE 6.50 usec TE 294.3 K D1 2.00000000 sec TTE 294.3 K D1 2.00000000 sec TTO 1  ======= CHANNEL f1 ======= SF01 161.9674942 MHz NUC1 31P P1 14.25 usec PLW1 15.00000000 W  F2 - Processing parameters SI 32768 SF 161.9755930 MHz WDW EM SSB 0 LB 1.00 Hz GB 0 PC 1.40	Current NAME EXPNO PROCNO	Data		ters L010 1 1	
SFO1 161.9674942 MHz NUC1 31P P1 14.25 usec PLW1 15.00000000 W  F2 - Processing parameters SI 32768 SF 161.9755930 MHz WDW EM SSB 0 LB 1.00 Hz GB 0	Date_ Time INSTRUM PROBHD PULPROG TD SOLVENT NS DS SWH FIDRES AQ RG DW DE TE D1	-	2015 1 sn PABBC Ace 64102 0.97 0.511	30615 5.32 Spect BB/ zg30 55536 Stone 32 4 2.563 78127 1808 3.57 7.800 6.50 6.50 294.3	Hz Hz sec usec usec K
SI 32768 SF 161.9755930 MHz WDW EM SSB 0 LB 1.00 Hz GB 0	SFO1 NUC1 P1	:	161.967 1	74942 31P 14.25	MHz usec
	SI SF WDW SSB LB GB	0		32768 55930 EM 1.00	MHz

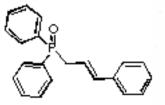
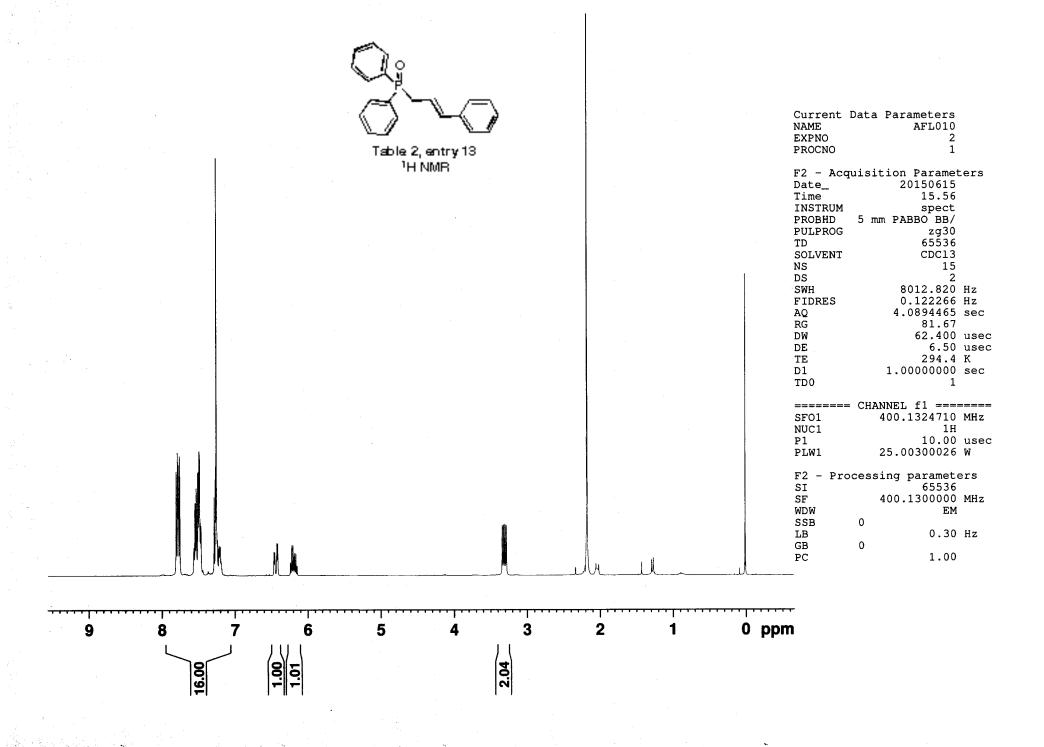


Table 2, entry 13 <sup>31</sup>P/<sup>1</sup>H NMR coupled

Current NAME EXPNO PROCNO	Data	Par		ters L010 1 1	
F2 - Acc Date_ Time INSTRUM PROBHD PULPROG TD SOLVENT NS DS	quisit	2 n PA	015 1 s BBO	rame 0615 5.32 pect BB/ zg30 5536 tone 32	:
SWH FIDRES AQ RG DW DE		0	.97 511 20 7	6.50	Hz sec usec
TE D1 TD0		2.0	_	94.3	sec
SF01 NUC1			967	4942 31P	
P1 PLW1		15.0		4.25 0000	usec W
F2 - Pr SI		•	• 3	2768	1
SF WDW		161.	975	5930 EN	MHz
SSB LB GB	. 0			1.00	Hz
PC.	J			1.40	1



## C COBU

Table 3, entry 1 <sup>31</sup>P/<sup>1</sup>H NMR decoupled

```
Current Data Parameters
NAME
                 AFL013
EXPNO
                     10
PROCNO
                      1
F2 - Acquisition Parameters
Date_
               20150825
Time
                  10.53
INSTRUM
                  spect
PROBHD
         5 mm PABBO BB/
PULPROG
                 zgpg30
TD
                  65536
SOLVENT
                Acetone
NS
DS
SWH
              64102.563 Hz
FIDRES
               0.978127 Hz
ΑQ
              0.5111808 sec
                 203.57
RG
                  7.800 usec
DW
DΕ
                   6.50 usec
                  294.8 K
             2.00000000 sec
D1
D11
             0.03000000 sec
TD0
                      1
====== CHANNEL f1 ======
SF01
            161.9674942 MHz
NUC1
                    31P
Ρ1
                  14.25 usec
            15.00000000 W
PLW1
====== CHANNEL f2 =======
SFO2
            400.1316005 MHz
                     1H
NUC2
                waltz16
CPDPRG[2
PCPD2
                  90.00 usec
            10.00000000 W
PLW2
             0.31604999 W
PLW12
PLW13
             0.25600001 W
F2 - Processing parameters
SI
                  32768
SF
            161.9755930 MHz
WDW
                     ΕM
SSB
LB
                   1.00 Hz
GB
         0
PC
                   1.40
```

Current D NAME EXPNO PROCNO	Data Parameter AFL01	
F2 - Acqu Date Time INSTRUM PROBHD PULPROG TD SOLVENT NS DS SWH FIDRES AO	11sition Param 2015061 9.5 spec 5 mm PABBO BB 2g3 6553 CDC1 3 64102.56 0.97812 0.511180	6 8 7 0 6 3 2 4 3 Hz
RG DW DE TE D1 TD0	203.5 7.80	7 00 usec 50 usec 1 K
SFO1 NUC1 P1 PLW1	161.967494 31	.P 25 usec
F2 - Pros SI SF WDW SSB LB GB	0	58
PC	1.4	10

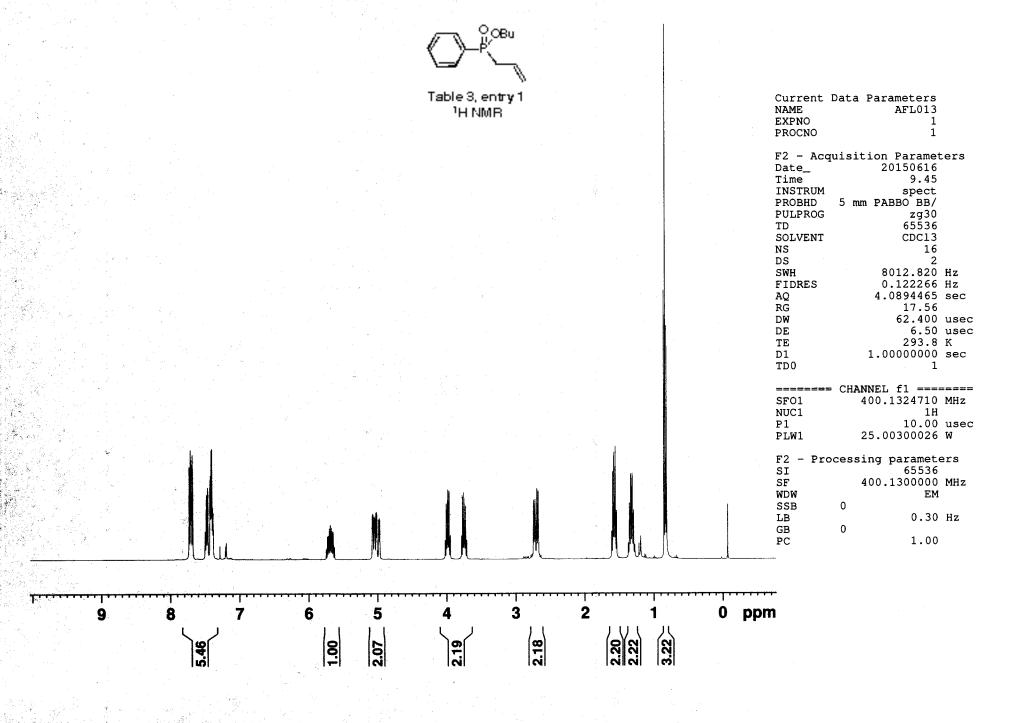
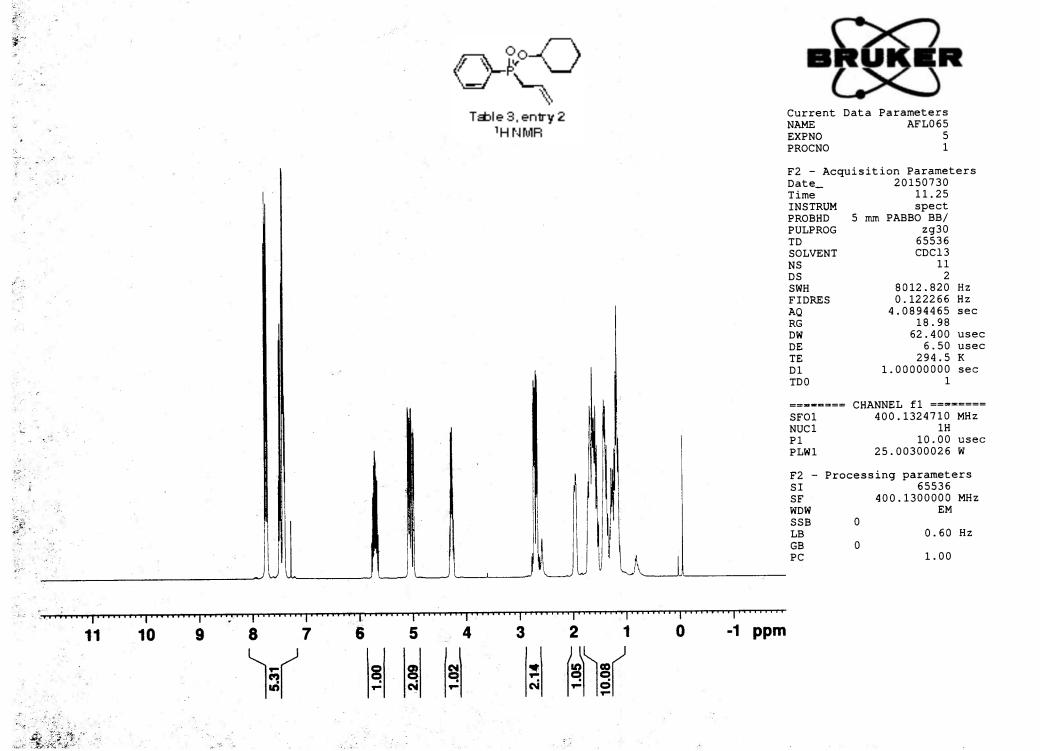


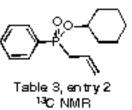
Table 3, entry 2 <sup>31</sup>P/H NMR decoupled

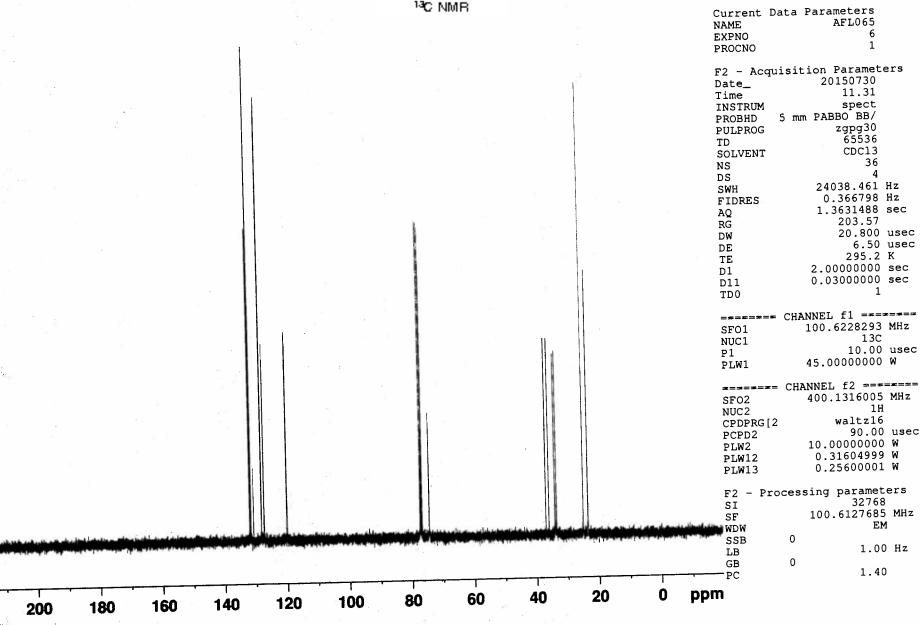
	Current NAME EXPNO PROCNO	Data	Para		te: L0:		
	F2 - Accordance Time INSTRUM PROBHD PULPROG TD SOLVENT NS DS		2 m PA	015 1 8BO 2g 6 Ace	07 1. pe Bl pg 55 to	30 17 ct 30 36 ne 16 4	
	SWH FIDRES AQ RG DW DE TE D1 D11 TD0		0.	7 2 000	81 3. 6. 94	27 08 57 00 50 .9	sec
	SFO1 NUC1 P1 PLW1	≔ CHA	NNEL 161. 15.0	967 1	49 3 14.	42 1P 25	usec
	SFO2 NUC2 CPDPRG[ PCPD2 PLW2 PLW12 PLW13		10.0	131 wal	ltz 90. 000	05 1H 16 00 00 99	MHz usec W W
	F2 - Pr SI SF WDW SSB LB GB PC	0 0	sing 161.	3	327 559 1.	68 30 EM	
* 1							

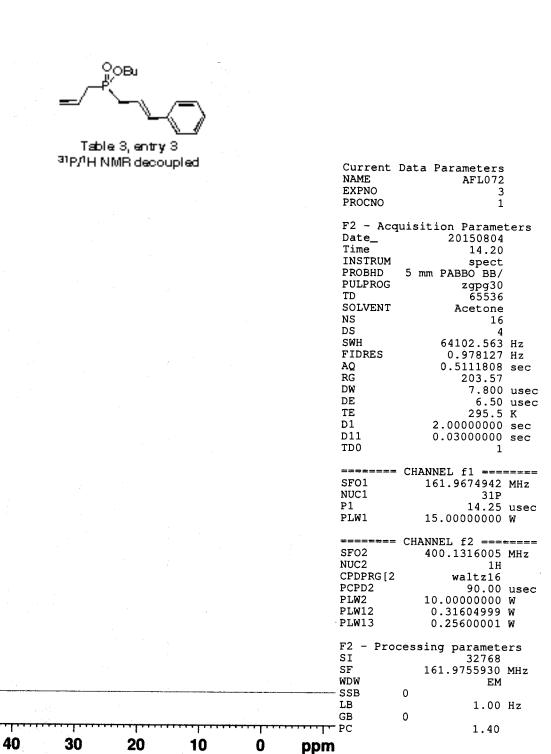
## Table 3, entry 2

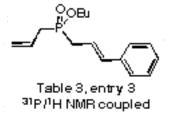
Current NAME EXPNO PROCNO	Data Parameter AFL06	
F2 - Acq Date_ Time INSTRUM PROBHD PULPROG TD SOLVENT NS DS	uisition Param 2015073 11.1 spec 5 mm PABBO BB zg3 6553 Aceton	0 9 t / 0 6
SWH FIDRES AQ RG DW DE TE D1 TD0		3 Hz 7 Hz 8 sec 7 00 usec 50 usec 5 K
SFO1 NUC1 P1 PLW1		12 MHz .P 25 usec
F2 - Pro SI SF WDW SSB LB GB PC	0	58 30 MHz EM





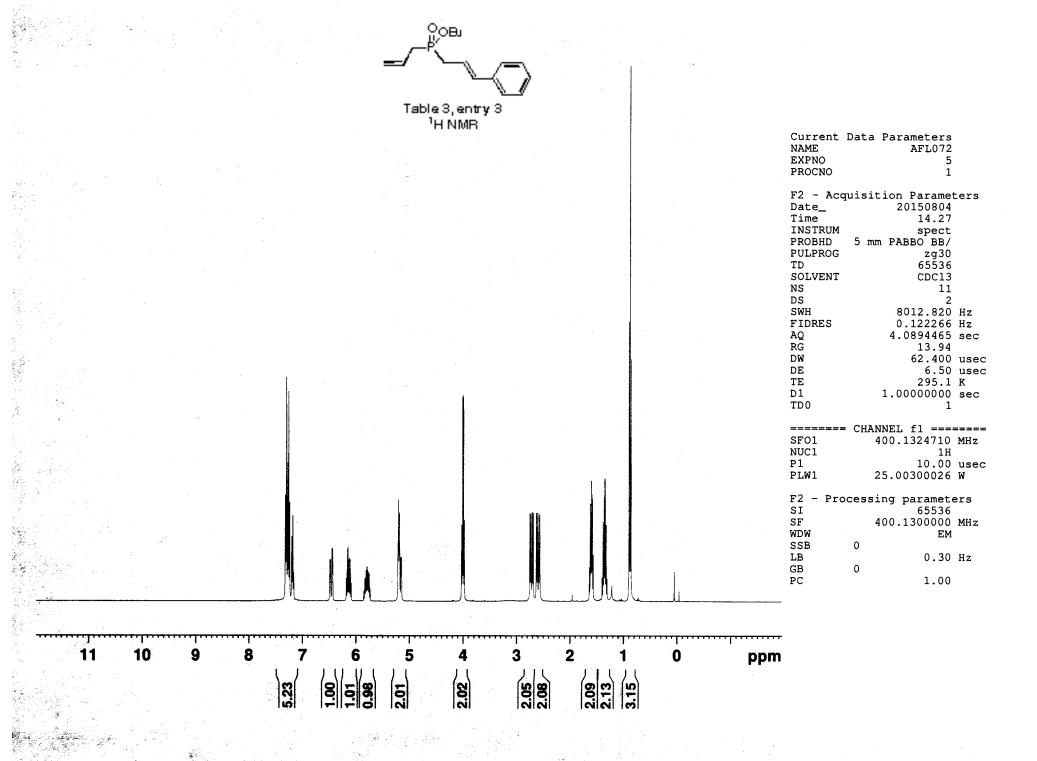


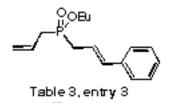


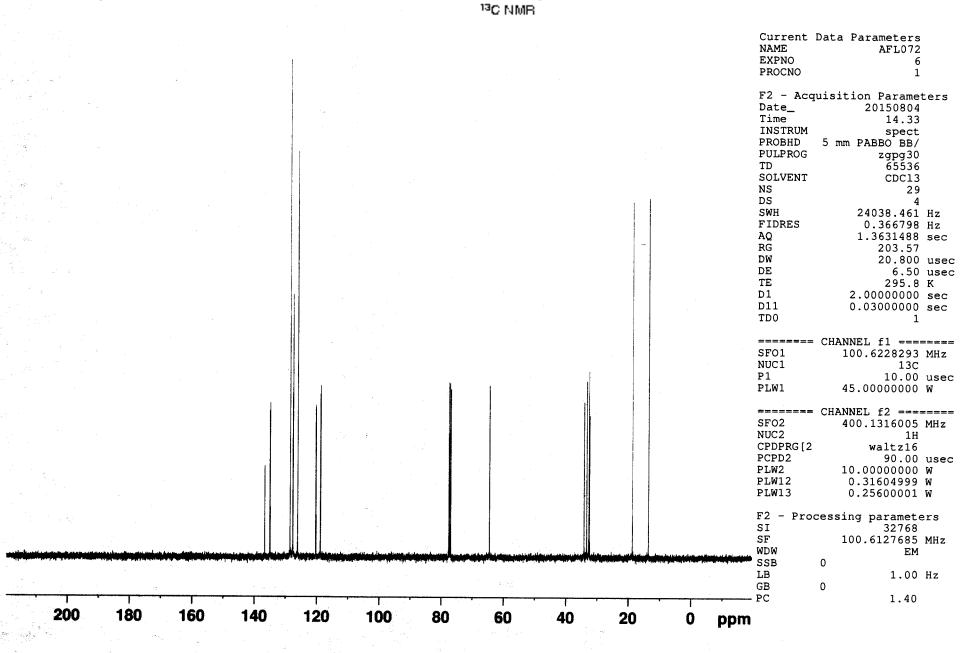


NAME	AFL072	
EXPNO	4	
PROCNO	1	
F2 - Acq	uisition Parame	ters
Date	20150804	
Time	14.21	
INSTRUM	spect	
PROBHD	5 mm PABBO BB/	
PULPROG	zq30	
TD	65536	
SOLVENT	Acetone	•
NS	4	
DS	4	(
SWH	64102.563	Hz
FIDRES	0.978127	Hz
AQ	0.5111808	sec
RG	203.57	!
DW	7.800	usec
DE	6.50	usec
TE	295.1	
D1	2.00000000	
TD0	1	
	CHANNEL f1 ===	
SFO1	161.9674942	
NUC1	31F	
P1		usec
PLW1	15.00000000	
- 2	20.0000000	••
	cessing paramet	
SI	32768	
SF	161.9755930	
WDW	EM	I
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	I

Current Data Parameters







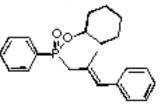


Table 3, entry 4
<sup>31</sup>P/HNMR decoupled

Current I NAME EXPNO PROCNO	Data Param P	neters AFL066 3 1	
F2 - Acqu Date_ Time INSTRUM PROBHD PULPROG TD SOLVENT NS DS	5 mm PABE	Paramet 150730 11.35 spect 80 BB/ gpg30 65536 CDC13 16	ters
SWH FIDRES AQ RG DW DE TE D1	0.9 0.51 2	02.563 078127 11808 003.57 7.800 6.50 295.1	Hz Hz sec usec usec K sec
D11 TD0 SF01		74942	sec  MHz
NUC1 P1 PLW1	15.000	31P 14.25 00000	usec W
SFO2 NUC2 CPDPRG[2 PCPD2 PLW2 PLW12 PLW13	10.000	316005 1H 11tz16 90.00	MHz
	0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0	ramete 32768	ers

90 80 70 60 50 40 30 20 10 0 -10 -20 ppm

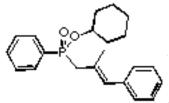
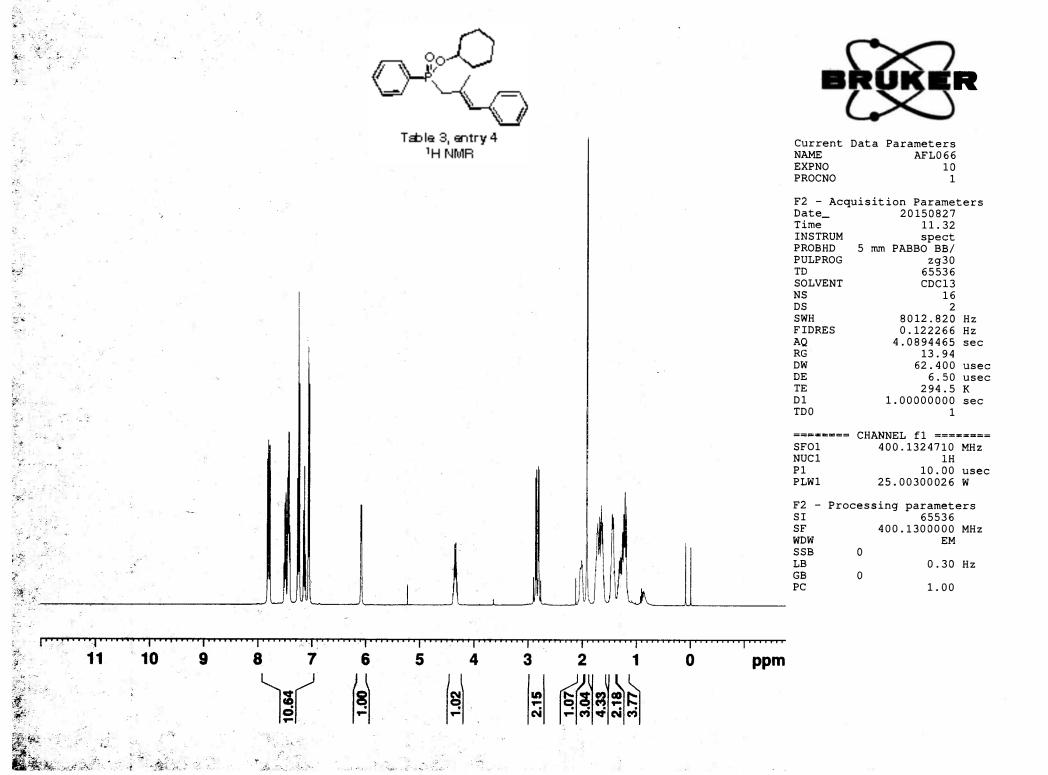


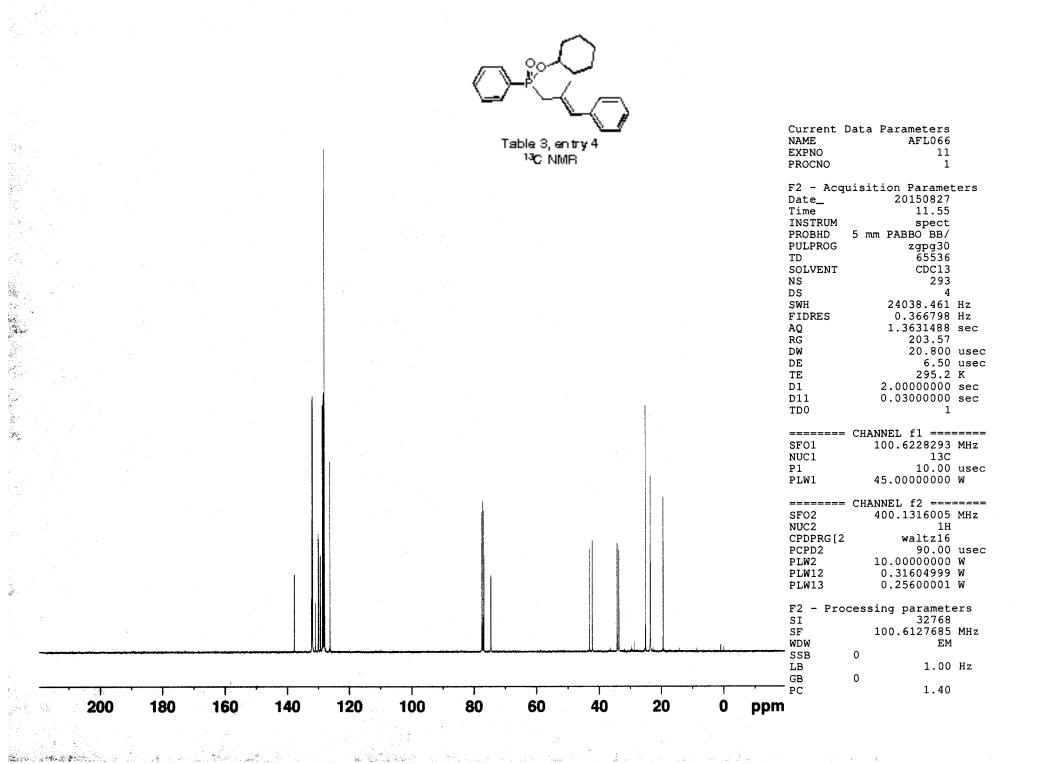
Table 3, entry 4

31P/1H NMR coupled

Current NAME EXPNO PROCNO	Data	Par		ters L066 4 1	
F2 - Acc Date_ Time INSTRUM PROBHD PULPROG TD SOLVENT NS DS SWH FIDRES AQ RG DW DE		2 m PA 64 0	015 1 8BO 6 C 102 .97 511 20	0730 1.37 pect BB/ zg30 5536 DC13 18 4.563 8127 1808 3.57 .800	Hz Hz sec usec
TE D1 TD0		2.0	2	94.7 0000 1	K
SFO1				4942	MHz
NUC1 P1 PLW1		15.0		31P 4.25 0000	usec
F2 - Pr SI SF WDW		-	<sup>-</sup> 3	amet 2768 5930 EM	MHz
SSB LB	0			1.00	
GB PC	0			1.40	

100 90 80 70 60 50 40 30 20 10 0 -10 ppm

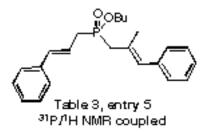




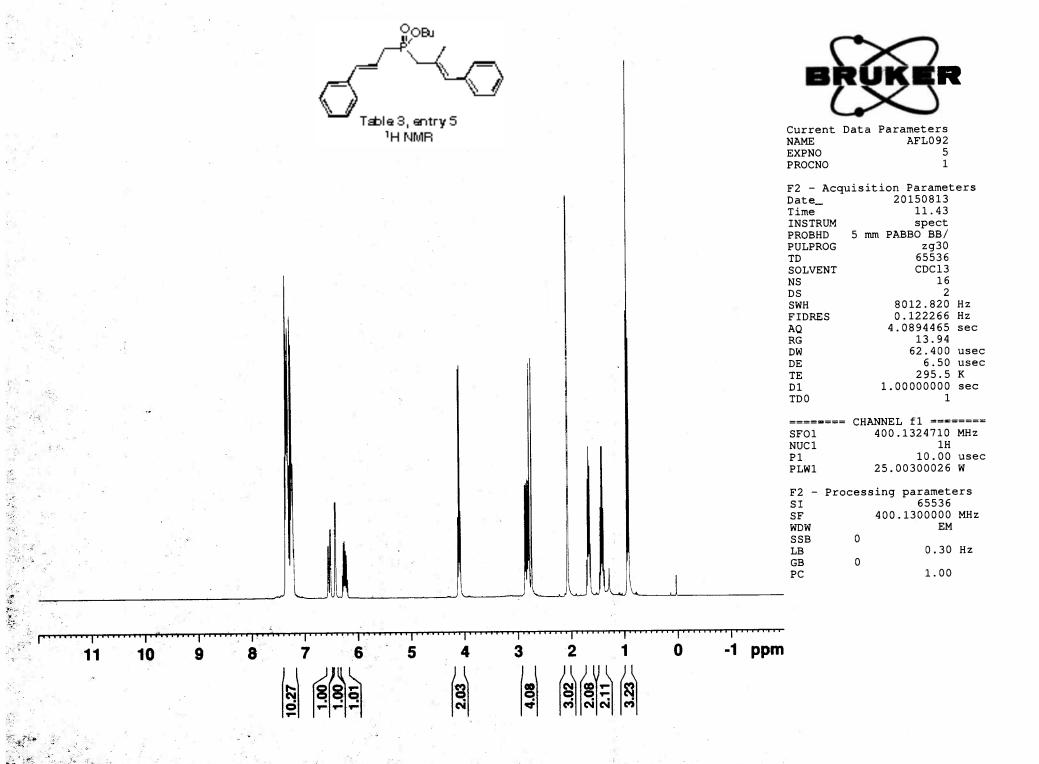
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Table 3, entry 5
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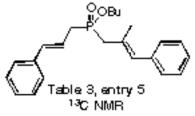
```
Current Data Parameters
    NAME
    EXPNO
                           3
                           1
    PROCNO
    F2 - Acquisition Parameters
                    20150813
    Date_
                       11.37
    Time
                       spect
    INSTRUM
              5 mm PABBO BB/
    PROBHD
                      zgpg30
    PULPROG
                       65536
    TD
                     Acetone
    SOLVENT
                           9
    NS
    DS
                   64102.563 Hz
    SWH
                    0.978127 Hz
    FIDRES
                   0.5111808 sec
    AQ
                      203.57
    RG
                       7.800 usec
                        6.50 usec
    DΕ
                       295.8 K
    ΤE
                  2.00000000 sec
    D1
                  0.03000000 sec
    D11
    TD0
     ======= CHANNEL f1 =======
                 161.9674942 MHz
     SF01
    NUC1
                         31P
                       14.25 usec
    Ρ1
                 15.00000000 W
     PLW1
     ====== CHANNEL f2 ======
                 400.1316005 MHz
     SFO2
     NUC2
                          1H
                     waltz16
     CPDPRG[2
                       90.00 usec
     PCPD2
                 10.00000000 W
     PLW2
                  0.31604999 W
     PLW12
                  0.25600001 W
     PLW13
     F2 - Processing parameters
                        32768
     SI
                 161.9755930 MHz
     SF
                           EM
     WDW
     SSB
              0
     LB
                         1.00 Hz
     GB
                         1.40
PC Trrr
```

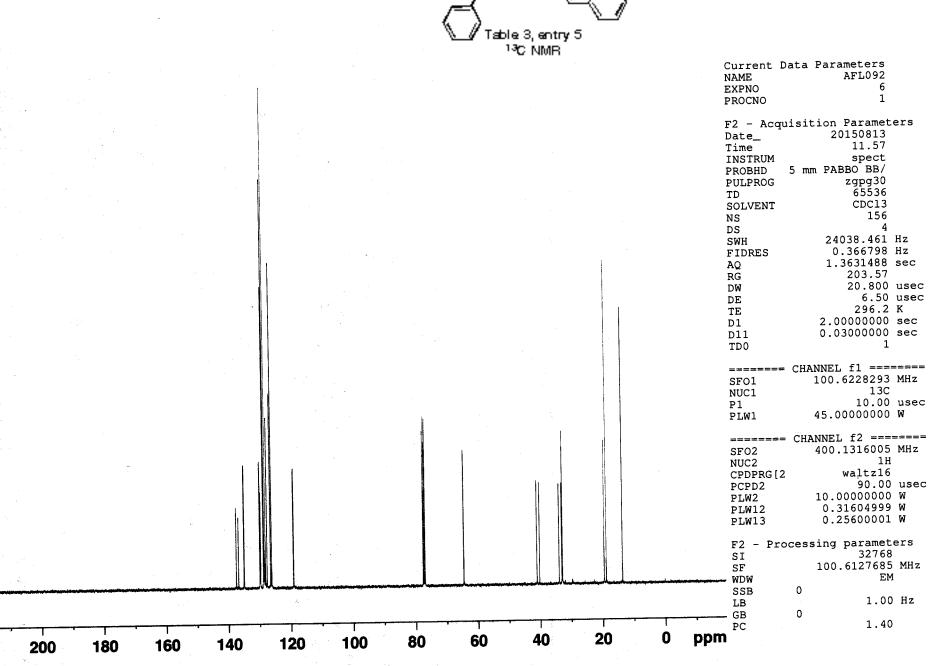
100 90 80 70 60 50 40 30 20 10 0 -10 ppm

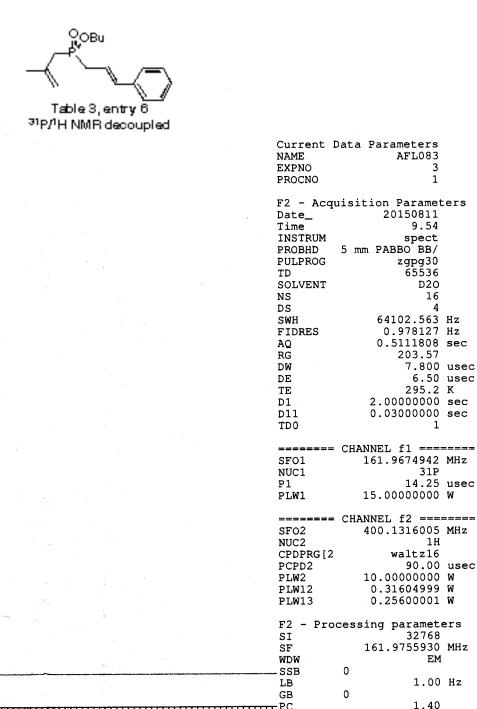


Current Data Parameters NAME AFL092 **EXPNO** PROCNO 1 F2 - Acquisition Parameters Date\_ 20150813 Time 11.39 INSTRUM spect PROBHD 5 mm PABBO BB/ **PULPROG** TD 65536 SOLVENT Acetone NS 29 DS 64102.563 Hz 0.978127 Hz SWH FIDRES ΑQ 0.5111808 sec 203.57 7.800 usec 6.50 usec RG DW DE TE 295.5 K 2.00000000 sec TDO ====== CHANNEL f1 ====== SF01 161.9674942 MHz NUC1 31P P1 14.25 usec PLW1 15.00000000 W F2 - Processing parameters 32768 161.9755930 MHz SI SF WDW EMSSB LB 1.00 Hz GB РC 1.40

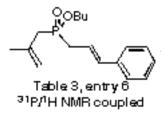




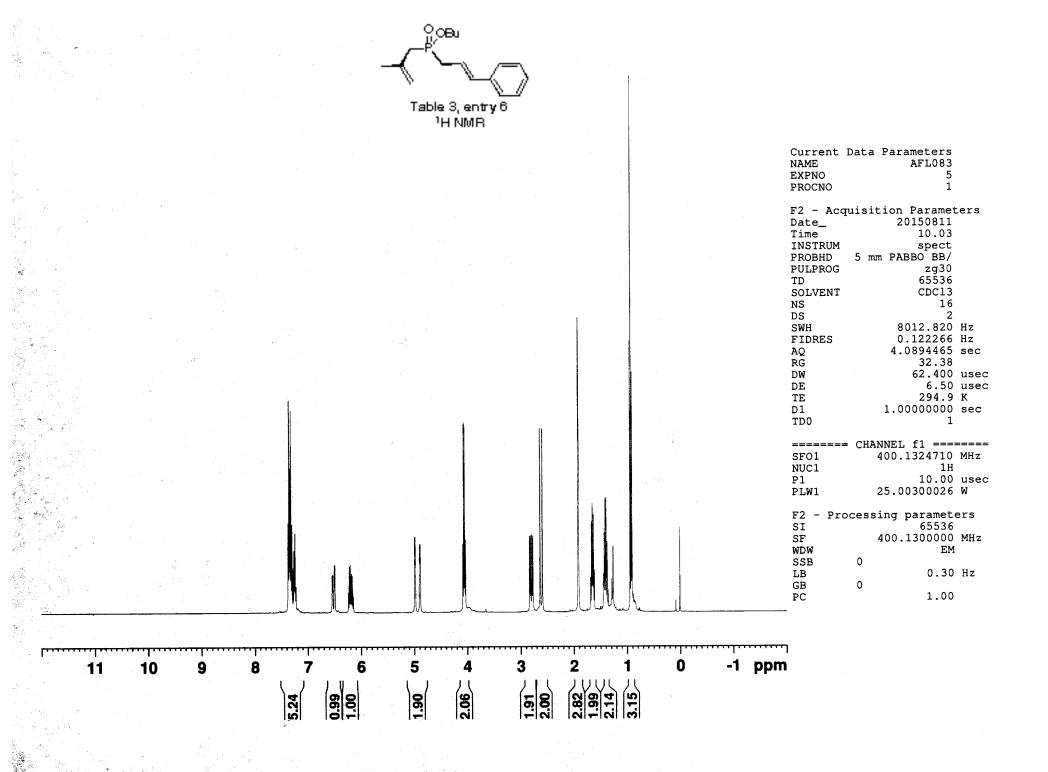


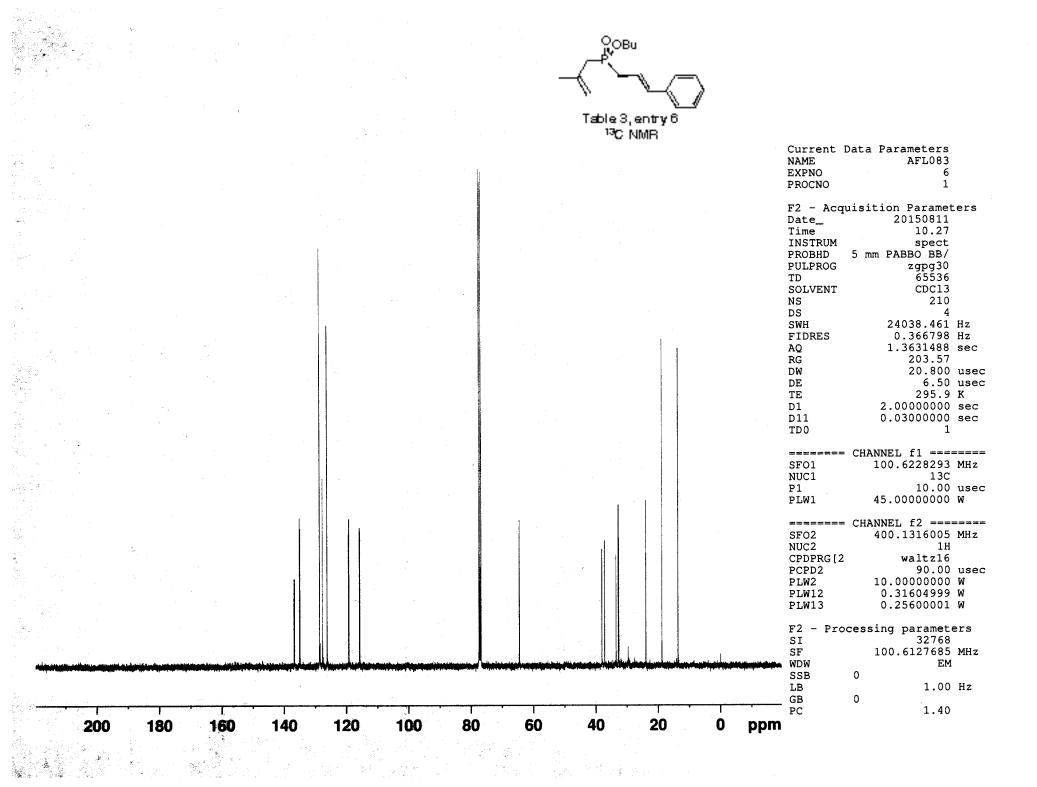


100 90 80 70 60 50 40 30 20 10 0 ppm



Current Da	ta Parameters
NAME	AFL083
EXPNO	4
PROCNO	1
Date Time INSTRUM PROBHD S PULPROG TD SOLVENT NS DS	sition Parameters 20150811 9.56 spect mm PABBO BB/ 2g30 65536 D20 18 4
SWH	64102.563 Hz
FIDRES	0.978127 Hz
AQ	0.5111808 sec
RG	203.57
DW	7.800 usec
DE	6.50 usec
TE	294.8 K
D1	2.000000000 sec
TD0	1
SFO1 NUC1	CHANNEL f1 ===================================
P1	14.25 usec
PLW1	15.00000000 W
SI	essing parameters 32768 161.9755930 MHz
	EM
LB	1.00 Hz
GB	)
PC	1.40





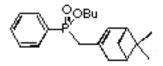
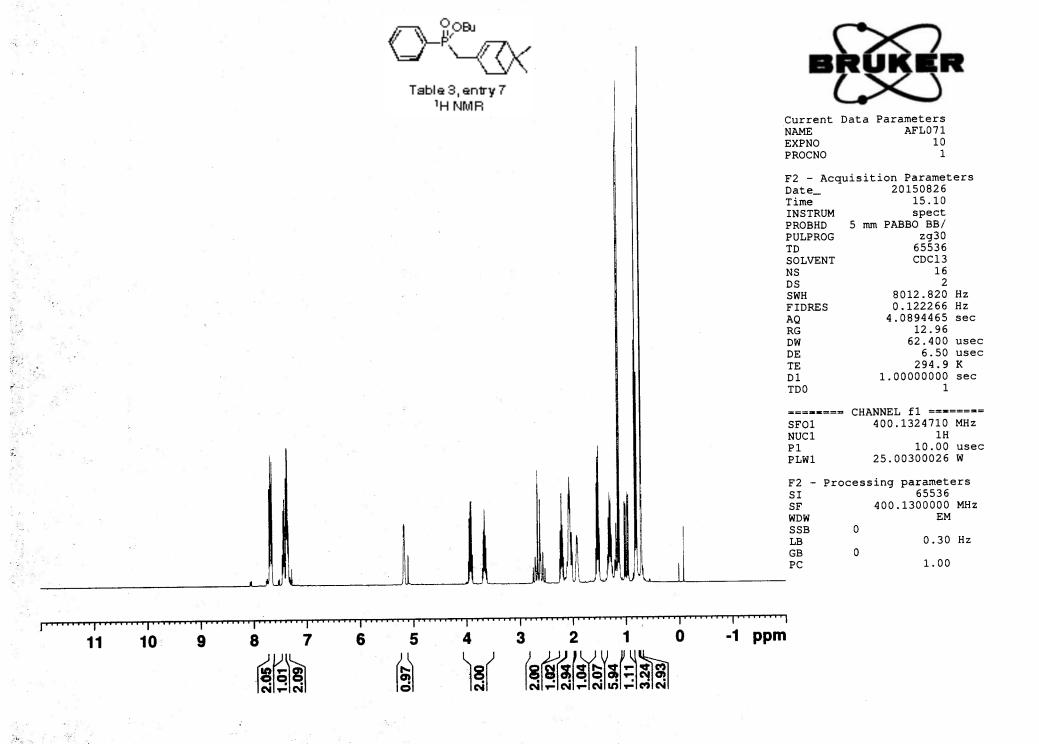


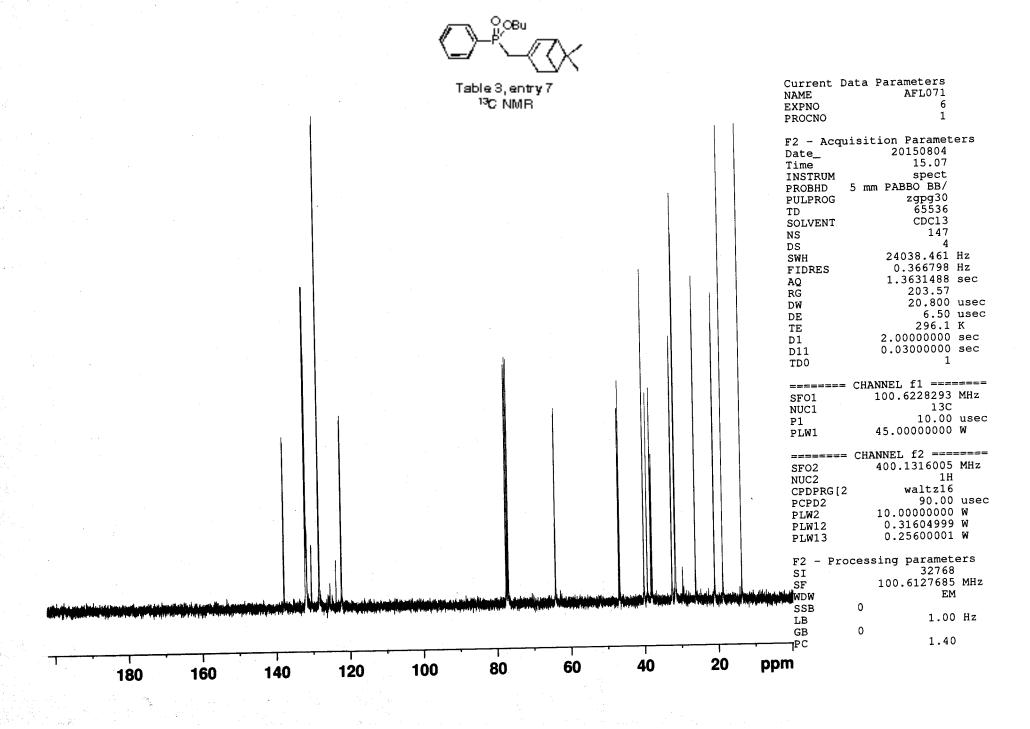
Table 3, entry 7 <sup>31</sup>P/<sup>1</sup>H NMR decoupled

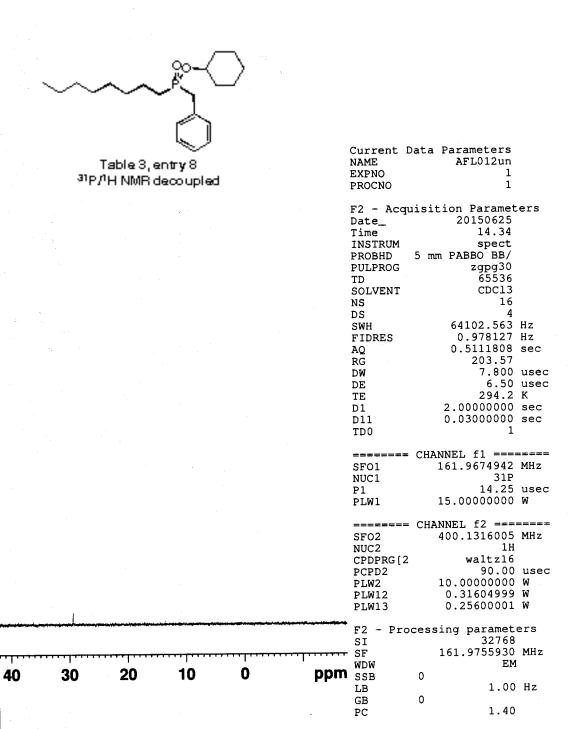
Current NAME EXPNO PROCNO	Data Parameters AFL071 3	3
F2 - Acq Date_ Time INSTRUM PROBHD PULPROG TD SOLVENT NS DS	quisition Parame 20150804 14.42 spect 5 mm PABBO BB/ zgpg30 65536 CDC13	1
SWH FIDRES AQ RG DW DE TE D1 D11 TD0	64102.563 0.97812 0.5111808 203.5 7.800 6.55 295. 2.0000000	Hz Hz Hz Sec usec usec K Sec
SF01 NUC1 P1 PLW1	= CHANNEL f1 === 161.967494 31 14.2 15.0000000	2 MHz P 5 usec
SF02 NUC2 CPDPRG[2 PCPD2 PLW2 PLW12 PLW13		5 MHz H 6 0 used 0 W 9 W
SI SF WDW	ocessing parame 3276 161.975593 E	8
LB GB TTTPC	-	0 Hz 0

Table 3, entry 7 <sup>31</sup>P/<sup>1</sup>H NMR coupled

	ata Paramete		
NAME	AFL0		
EXPNO		4	
PROCNO		1	
F2 - Acquate_Time INSTRUM PROBHD PULPROG TD SOLVENT NS DS SWH FIDRES AQ RG	201508 14. spe 5 mm PABBO B 2g 655 CDC 64102.5 0.9781 0.51118 203.	meters 04 44 62 8 8 30 36 13 12 4 63 Hz 27 Hz 08 sec 57	
DW	7.8	00 usec	
DE	6.	50 usec .3 K	
TE	295	.3 K	
D1 TD0	2.000000	1 sec	
100		1	
====== CHANNEL f1 ======			
SFO1	161.96749		
NUC1	-	1P	
P1		25 usec	
PLW1	15.000000	00 W	
F2 - Pro	cessing param 327	68	
SF	161.97559		
WDW	•	EM	
SSB	0	00 11-	
LB GB	0	00 Hz	
PC	•	40	
10	<b></b>		







3.46

96.54

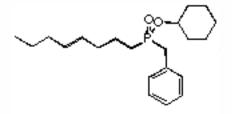
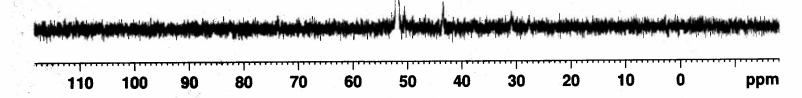
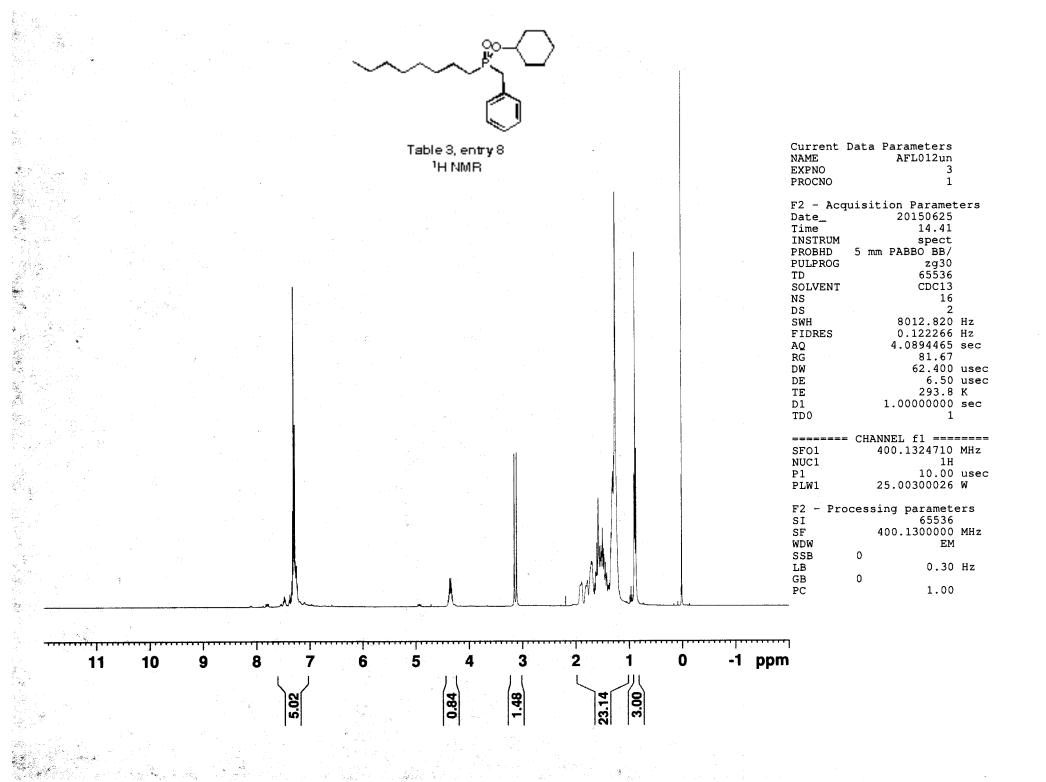
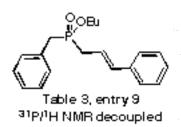


Table 3, entry 8 <sup>31</sup>P/H NMR coupled

Current I NAME EXPNO PROCNO		FL012ui	
F2 - Acqu Date_ Time INSTRUM PROBHD PULPROG TD SOLVENT NS DS SWH	5 mm PA	20150625 14.36 spect ABBO BB, zg30 65536 CDC1	5 5 7 0 6 3 2
FIDRES AQ RG	(	0.97812 511180 203.5	7 Hz 8 sec 7
DW DE TE D1 TD0	2.0	6.5 293. 000000	
SF01 NUC1		.967494 31	2 MHz P
P1 PLW1	15.0	14.2	5 usec 0 <b>W</b>
F2 - Pro	-	3276	8
SF WDW SSB	161	.975593 E	
LB GB	0	1.0	0 Hz
PC		1.4	0





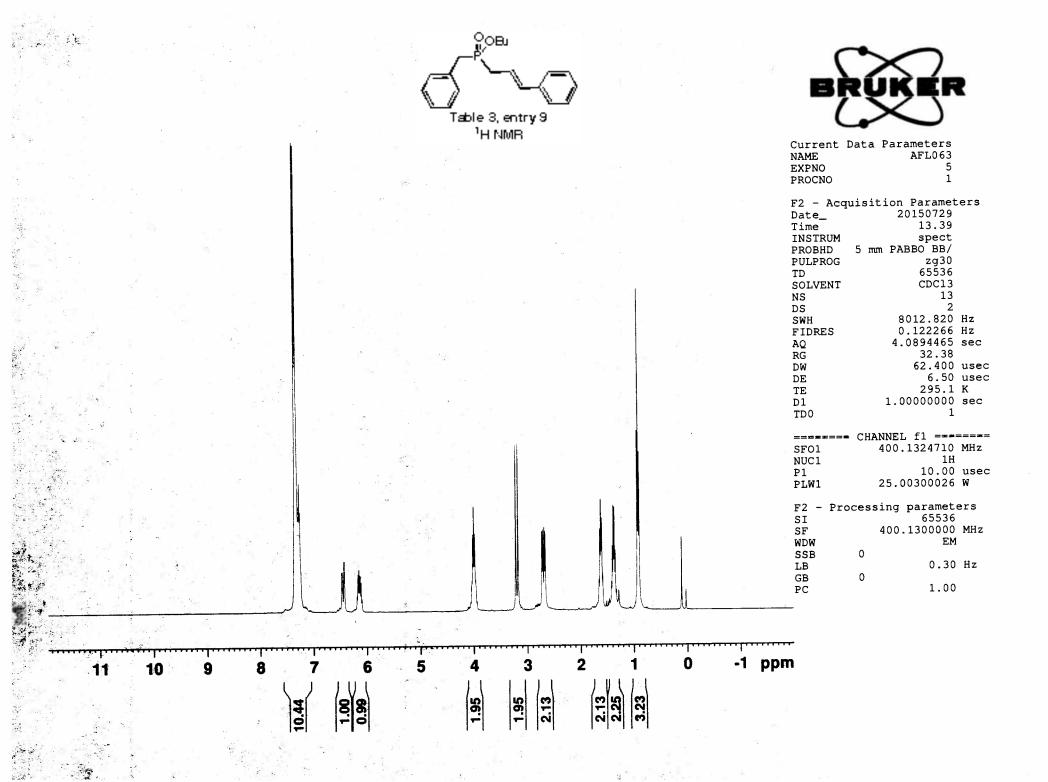


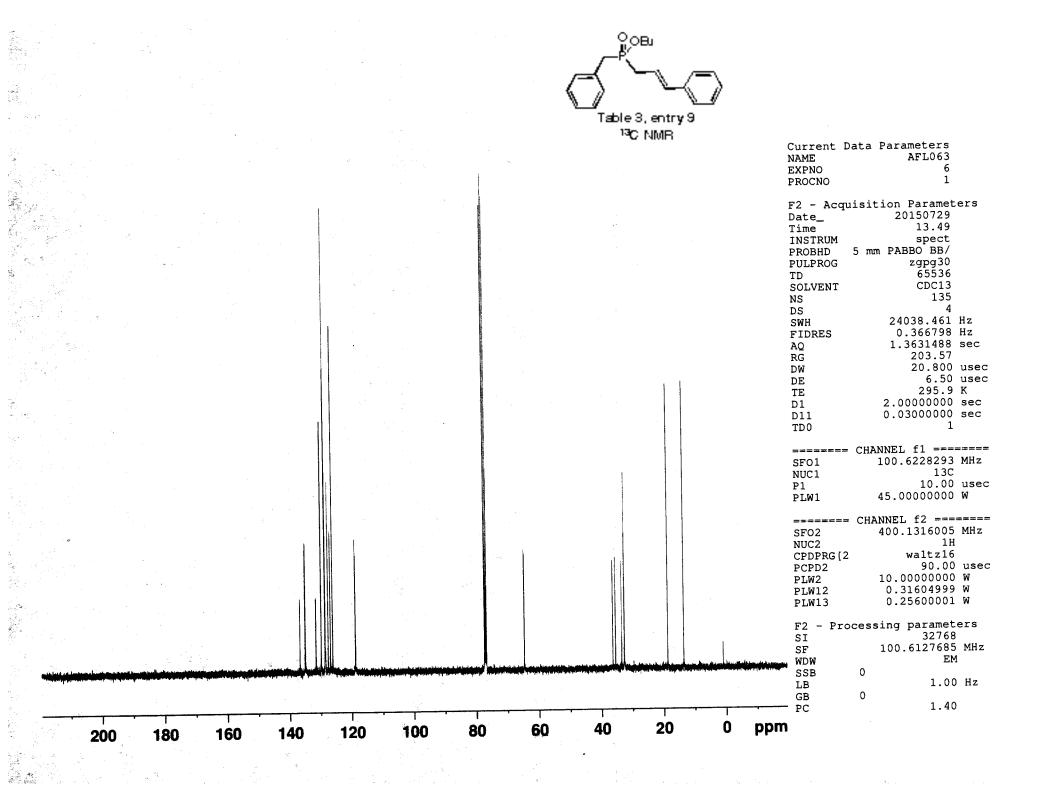
	Current D	ata	Par	ame	te	cs	
	NAME			AF	L06		
	EXPNO					3	
	PROCNO					1	
	F2 - Acqu Date Time INSTRUM PROBHD PULPROG TD SOLVENT NS DS SWH FIDRES		2 m PA	015 1 s BBO zg	072 3.3 peo BI Pg: 555: DC:	29 30 37 30 36 13 4 63	ers Hz Hz
	AQ		0.	511	18	80	sec
	RG DW DE TE			2	6. 295	00 50 .5	usec usec K
	D1		2.0	000	000	00	sec
	D11		0.0	300	00		sec
	TD0					1	
	SFO1 NUC1 P1 PLW1	СНА	NNEI 161.	. 967 1	749 3 14.	42 1P 25	
	=======	СНА	NNE	L fi	2 =	==:	=====
	SFO2		400	. 13.	160	1H	MHz
	NUC2 CPDPRG[2			wa.	ltz		
	PCPD2						usec
	PLW2		10.0				
	PLW12		0.3	3160	049	99	W
	PLW13		0.2	2560	000	01	W
	F2 - Pro SI SF	cess	_		327	68	ers MHz
	WDW					ΕM	
	SSB	0					
	LB				1.	00	Ηz
	GB	0				4.0	
1	PC				Ι.	40	
ppm							

## Table 3 entry 9

Table 3, entry 9 <sup>31</sup>P/<sup>1</sup>H NMR coupled

Current NAME EXPNO PROCNO	Data		eters FL063 4 1	
F2 - Accordance Time INSTRUM PROBHD PULPROCE TD SOLVENT NS DS	լ 5 mm	201 : n PABBo	50729 13.34 spect	ers
SWH FIDRES AQ RG DW DE TE D1 TD0		0.9 0.51 2	2.563 78127 11808 03.57 7.800	Hz sec usec usec K
SFO1 NUC1 P1 PLW1		NNEL f 161.96 15.000	74942 31P 14.25	MHz usec
F2 - PSISFWDWSSBLBGBPC		ing pa	32768	MHz Hz





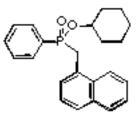
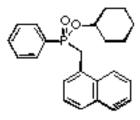


Table 3, entry 10 <sup>31</sup>P#H NMR decoupled

	Curre NAME EXPNO PROCN	,	ata	P	ar			70	rs 69 14 1		
	F2 - Date_ Time INSTR PROBH PULPR TD SOLVE NS DS	RUM ID ROG	iisi 5 m		2 PA	01 BB z	50 15 51 0 91 65	08 5. e B og	06 14 ct	ers	
	SWH FIDRE AQ RG DW DE	:s			0	.9 51	78 1: 0: 7	31 18 3.	63 27 08 57	use	: (
	TE D1 D11 TD0						2	96 00	. 1	K sec	;
	SFO1 NUC1 P1 PLW1		CHA	16	1.	96	7 ·	49 3	42 1P	MHz	
	SFO2 NUC2 CPDPF		СНА	NN 40	0.	13	1	60	==: 05 1H 16	HHz	
	PCPD2 PLW2 PLW12 PLW13	2		0	. 3	16	0	00 49	00 00 99 01		:(
	F2 - SI SF WDW SSB	Prod	cess 0		-	-	3.	27	68	ers MHz	:
	LB GB PC		0						00 40	Ηz	
1								- •			

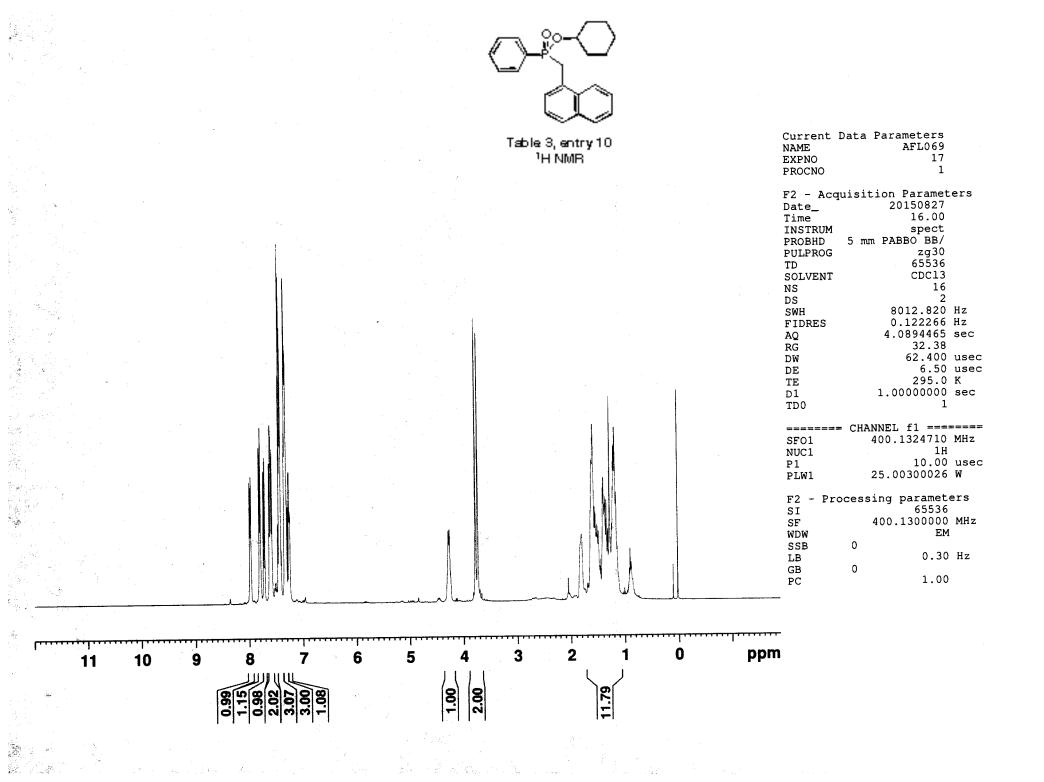
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 pp

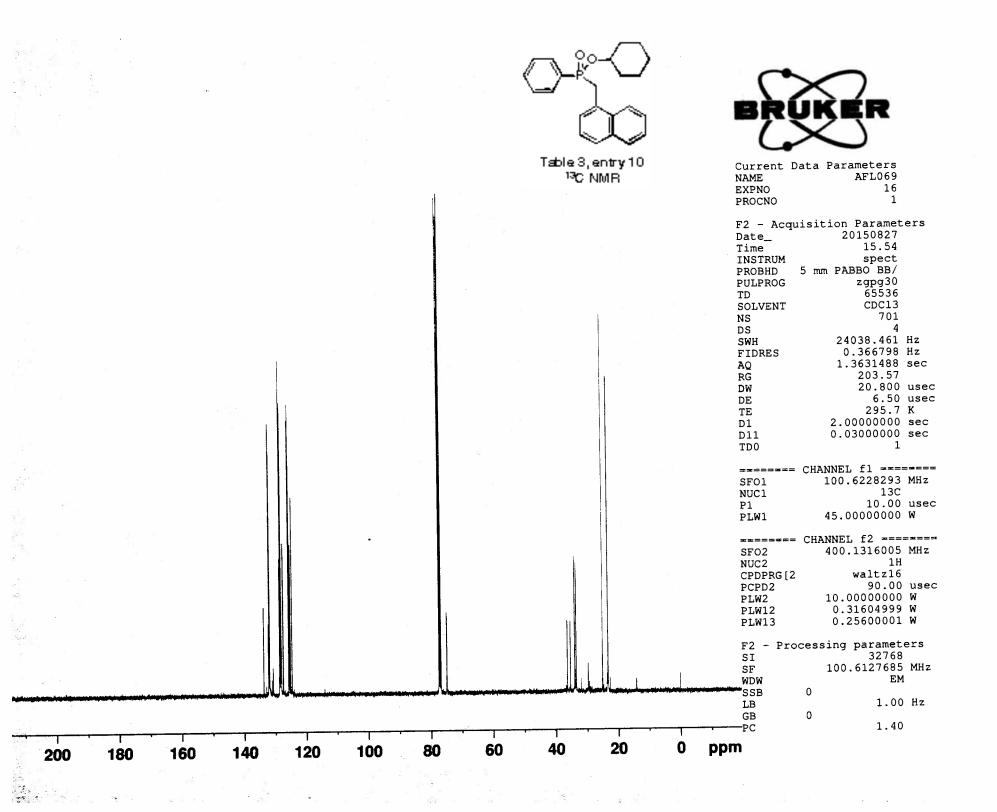


```
Table 3, entry 10
<sup>31</sup>P/<sup>1</sup>H NMR coupled
```

]	Curren NAME EXPNO PROCNO		ata	Par	ame AI	ete FLO	rs 69 15 1		
]	F2 - F Date_ Time INSTRU PROBHI PULPRO TD SOLVEN NS DS SWH	JM ) )G		2 n P.F	ABBO Zo	508 16. spe 3pg 655 etc	06 06 ct BB/ 30		
	FIDRES AQ RG DW DE TE D1 D11 TD0			2	0.9 .51 2 000 030	781 118 03. 7.8 290 000	127 308 .57 300 .50 6.2 000 000	Hz sec usec usec K sec sec	2
	SFO1 NUC1 P1 PLW1	=#=	CHA		.96	74: 14	942 31P .25	MHz used	
	SFO2 NUC2 CPDPR PCPD2 PLW2 PLW12 PLW13	.G [ 2	CHA	10.	.13 wa 000 316	11t 90 000	005 1H 216	use W W	
	F2 - SI SF WDW SSB LB GB	Prod	0 0			32 755	768 930 EM	MHz	
_	PC		U			1	.40		

90 80 70 60 50 40 30 20 10 0 -10 -20 ppm





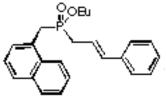


Table 3, entry 11 <sup>31</sup>P/<sup>1</sup>H NMR decoupled

ppm

I	Curren NAME EXPNO PROCNO		ata	Par		te L0			
	F2 - A Date_ Time INSTRU PROBHI PULPRO TD SOLVEN NS DS SWH FIDRES AQ	JM OG NT	isit	2 n PA 64	BBC Z G (ACC)	508 9. spe D B 9P9 655 eto 2.5 781	12 22 ct 30 36 ne 16 4 63 27	ers Hz Hz sec	
	RG DW DE TE D1 D11 TD0				000	6. 295 000	50 50 5.1	used used K sec sec	: :
	SFO1 NUC1 P1 PLW1		CHA	NNE: 161 15.	.96	749 3	942 31P . 25	MHz	
	SFO2 NUC2 CPDPR PCPD2 PLW2 PLW12 PLW13	G[2	СНА	10.	.13 wa	160 1t2 90 000 049	005 1H 216 .00 000	MHz use	
_	F2 - SI SF WDW	Prod				32	768	MHz	
	SSB LB GB		0				.00	Ηz	

1.40

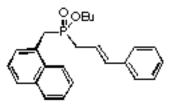
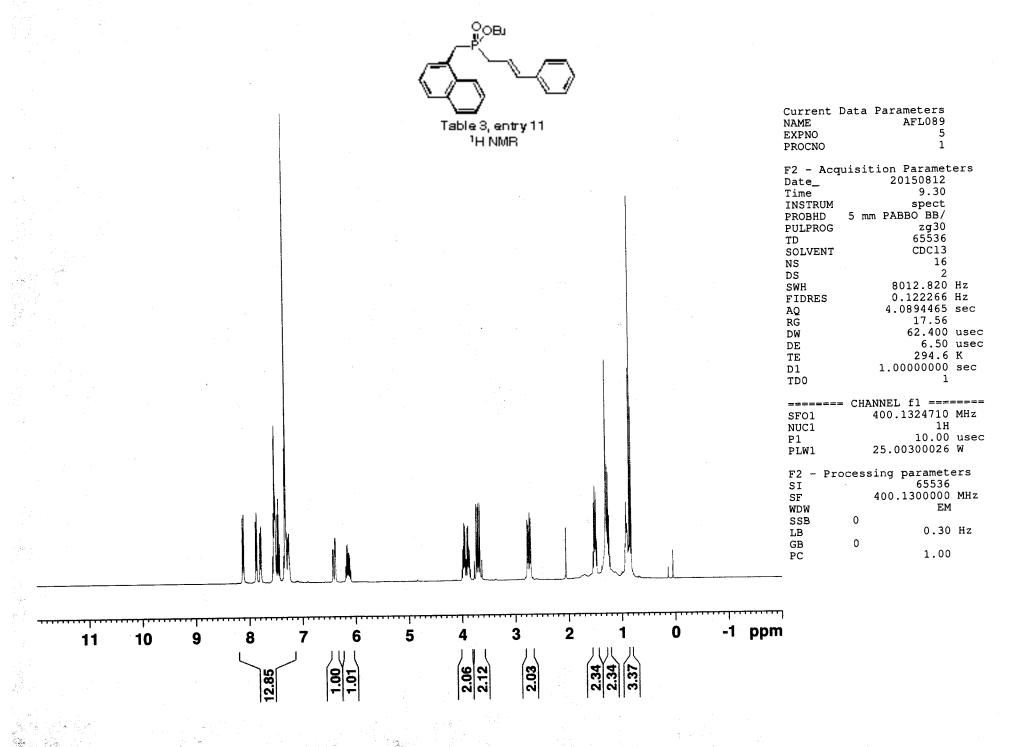
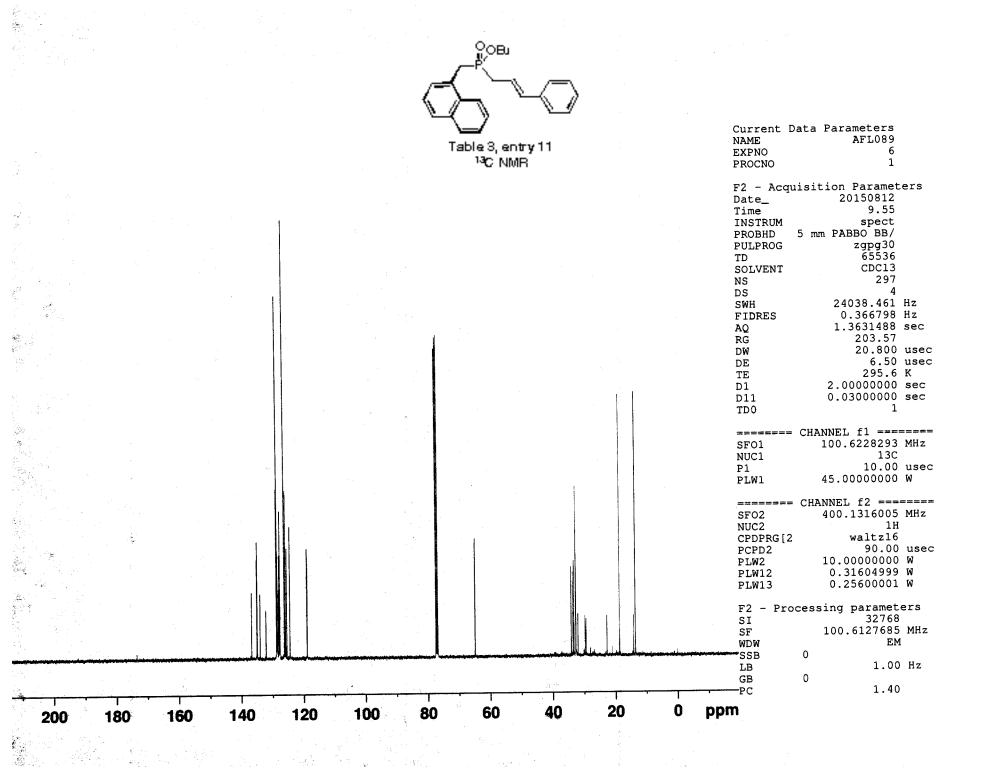
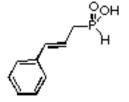


Table 3, entry 11 <sup>31</sup>P/<sup>1</sup>H NMR coupled

Current NAME EXPNO PROCNO	Data Paran I	meters AFL089 4 1	
Date_ Time INSTRUM PROBHD PULPROG TD SOLVENT NS	5 mm PAB	9.23 spect	ers
DS SWH FIDRES AQ RG DW DE TE D1 TD0	0.5	02.563 978127 111808 203.57 7.800	Hz sec usec usec K
SFO1 NUC1 P1 PLW1		f1 === 674942 31P 14.25	MHz
F2 - Pro SI SF WDW SSB LB GB PC	161.9 0	2768 32768 3755930 EM 1.00	MHz Hz







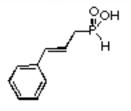
Scheme 3c <sup>31</sup>P/H NMR decoupled



Current Data Parameters OB 1971 after column **EXPNO** 1 PROCNO F2 - Acquisition Parameters 20150325 Date\_ 17.06 Time Time INSTRUM spect 5 mm PABBO BB/ PROBHD PULPROG zgpg30 TD 65536 CDC13 SOLVENT 16 NS DS 64102.563 Hz SWH FIDRES 0.978127 Hz 0.5111808 sec ΑQ 203.57 RG 7.800 usec DW 6.50 usec DΕ 294.4 K 2.00000000 sec D1 0.03000000 sec D11 TD0 ====== CHANNEL f1 ======= 161.9674942 MHz SFO1 31P NUC1 14.25 usec P1 15.00000000 W PLW1 ======= CHANNEL f2 ======== SFO2 400.1316005 MHz 1H NUC2 waltz16 CPDPRG[2 90.00 usec PCPD2 10.00000000 W PLW2 0.31604999 W PLW12 0.25600001 W PLW13 F2 - Processing parameters 32768 SI 161.9755930 MHz SF EM WDW SSB 1.00 Hz LB GB 0 PC 1.40

90 80 70 60 50 40 30 20 10 0 -10 -20 ppm

38.13 37.90 37.90 37.91 37.91 37.87 37.87 34.61 34.61 34.61 34.61



Scheme 3c <sup>31</sup>P/<sup>1</sup>H NMR coupled

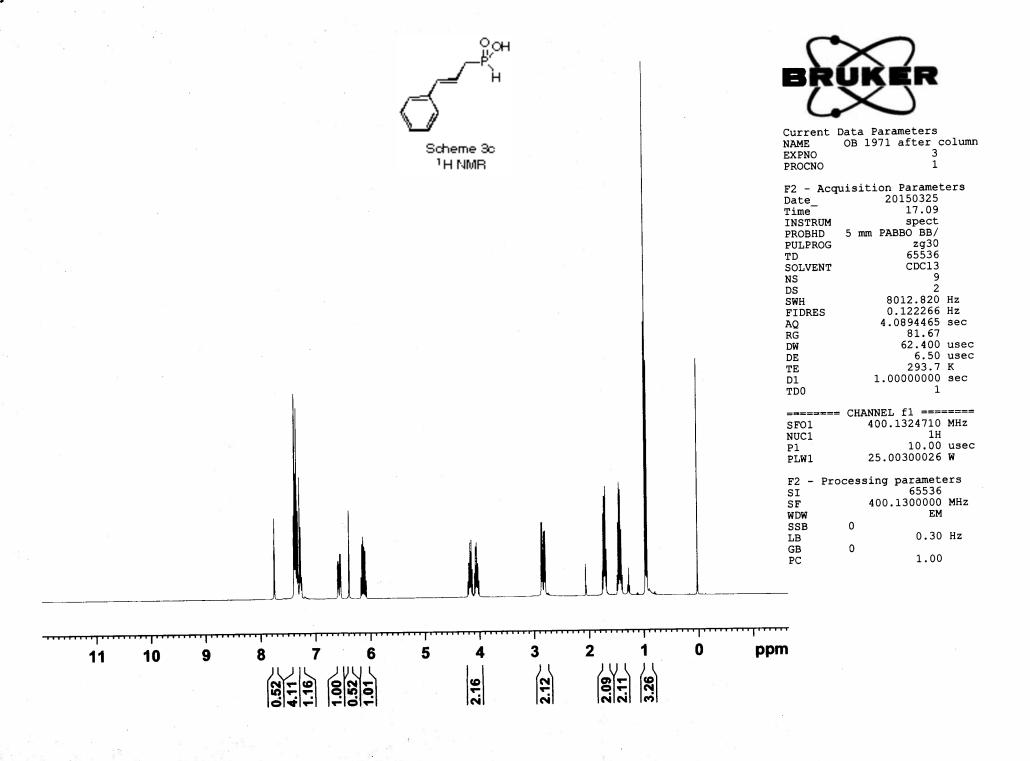


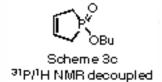
Current	Data Parameters	
NAME	OB 1971 after column	
EXPNO	2	
PROCNO	1	

F2 - Acquis:	ition Paramet	ers
Date	20150325	
Time	17.08	
INSTRUM	spect	
PROBHD 5 i	nm PABBO BB/	
PULPROG	zg30	
TD	65536	
SOLVENT	CDC13	
NS	32	
DS	4	
SWH	0	Ηz
FIDRES	0.978127	Ηz
AQ	0.5111808	sec
RG	203.57	
DW	7.800	usec
DE	6.50	
TE	293.8	
D1	2.00000000	sec
TD0	1	

	CHANNEL II	
SFO1	161.9674942	MHz
NUC1	31P	
P1	14.25	usec
PLW1	15.00000000	W

90 80 70 60 50 40 30 20 10 0 -10 -20 ppn



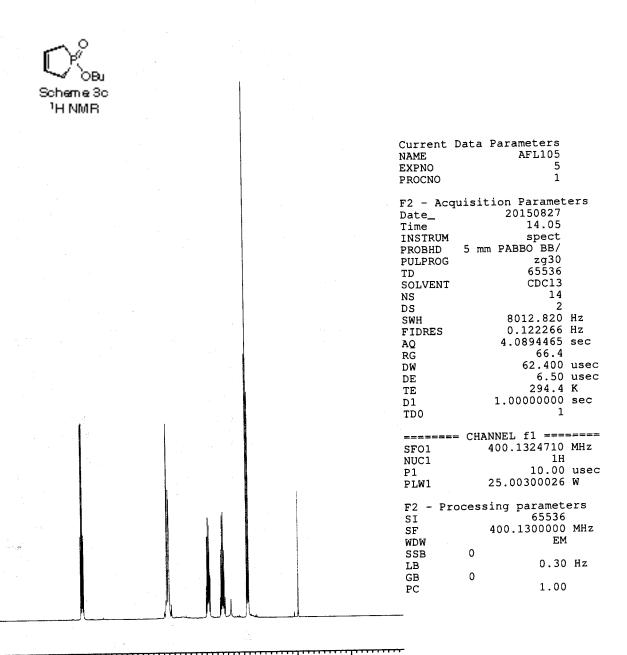


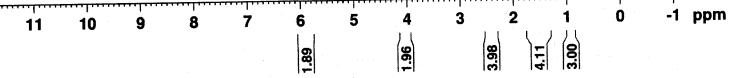


```
Current Data Parameters
                    AFL105
   NAME
                         3
   EXPNO
                         1
   PROCNO
   F2 - Acquisition Parameters
                  20150827
   Date_
                     13.59
   Time
                     spect
   INSTRUM
            5 mm PABBO BB/
   PROBHD
                     zgpg30
   PULPROG
                      65536
   TD
                      CDC13
   SOLVENT
   NS
                          4
   DS
                  64102.563 Hz
   SWH
   FIDRES
                  0.978127 Hz
                  0.5111808 sec
   ΑQ
                     203.57
   RG
                      7.800 usec
   DW
                      6.50 usec
                      294.7 K
   TE
                 2.00000000 sec
   D1
                 0.03000000 sec
   D11
   ======= CHANNEL f1 =======
                161.9674942 MHz
   SF01
                        31P
   NUC1
                      14.25 usec
   P1
                15.00000000 W
   PLW1
   ======= CHANNEL f2 =======
                400.1316005 MHz
    SFO2
                        1 H
   NUC2
                    waltz16
   CPDPRG[2
                      90.00 usec
   PCPD2
                10.00000000 W
   PLW2
                 0.31604999 W
    PLW12
                 0.25600001 W
    PLW13
   F2 - Processing parameters
                      32768
    SI
                161.9755930 MHz
    SF
                         EM
    WDW
             0
    SSB
                       1.00 Hz
    LB
             0
    GB
                       1.40
PC TTT
```

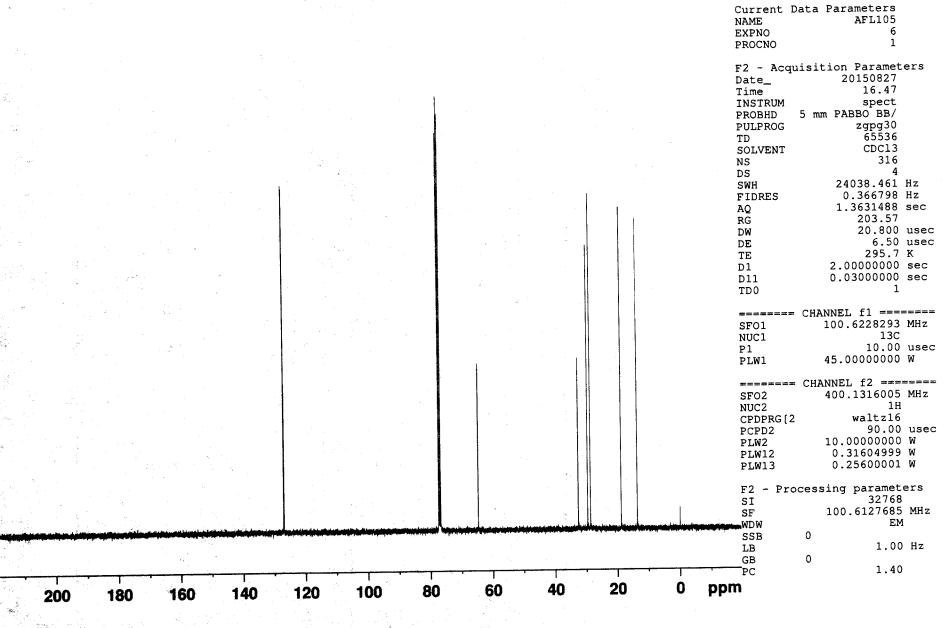
## OBu Scheme 3c 31P/1H NMR coupled

```
Current Data Parameters
                 AFL105
NAME
EXPNO
                      1
PROCNO
F2 - Acquisition Parameters
               20150827
Date_
                  14.00
Time
INSTRUM
                  spect
         5 mm PABBO BB/
PROBHD
                   zg30
PULPROG
                   65536
TD
                  CDC13
SOLVENT
                     13
NS
                      4
DS
              64102.563 Hz
SWH
               0.978127 Hz
FIDRES
              0.5111808 sec
ΑQ
                  203.57
RG
                  7.800 usec
DW
                   6.50 usec
DΕ
                   294.4 K
TE
             2.00000000 sec
D1
                       1
TDO
====== CHANNEL f1 ======
             161.9674942 MHz
SF01
                    31P
NUC1
                   14.25 usec
P1
            15.00000000 W
PLW1
F2 - Processing parameters
                   32768
SI
             161.9755930 MHz
SF
                      EΜ
WDW
 SSB
          0
                    1.00 Hz
 LB
 GB
                    1.40
PC
```









H1 0 sc vs th

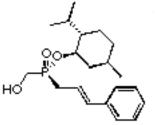
at cdc ph

35 6700

12

tpwr pw

dof dm dmm



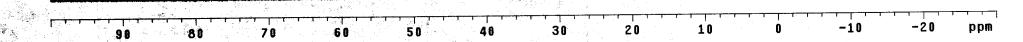
Scheme 4 31P/IH NMR decoupled

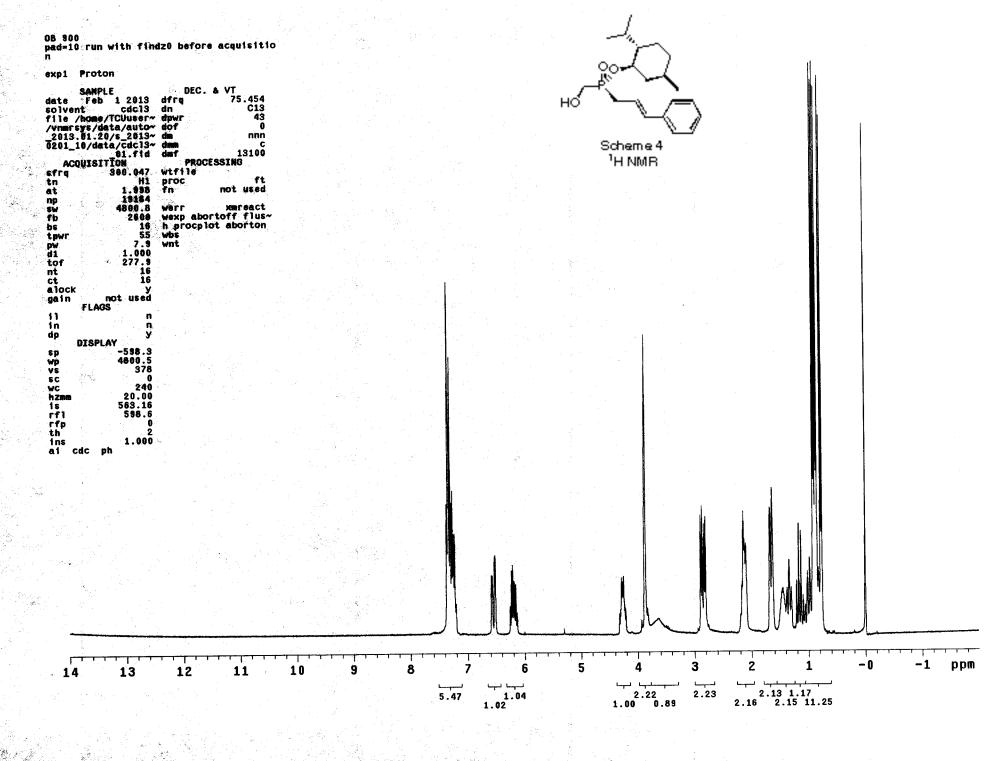
70 60 30 20 10 -20 ppm

ex		OFU	

SA	MPLE		SPECIAL
date F	eb 1 2013	temp	not used
solvent	cdc13	gain	20
file /ho	me/TCUuser~	spin	20
/vnmrsys	/data/auto~	hst	0.008
2013.01	.20/8_2013~	pw90	18.300
0201 12/	data/cdc13~	alfa	10.000
–	_04.fid		FLAGS
ACQUI	SITION	. <b>1</b> 1	n
SW	15797.8	1n	n
at	1.600	dp	У
np	59552	hs .	ny
fb	8800		PROCESSING
bs	64	1b	1.00
d1	1.000	fn	not used
nt	16		DISPLAY
ct 🕝	16	5 p	-3647.3
	MITTER	Wp	15797.3
tn	P81	rfl	3647.8
sfrq	121.465	rfp	4- 0
tof	7421.1	rp	15.6
tpwr	55	1 p	-113.7
pw	8.150	1.1	PLOT
	OUPLER	WC .	250
dn_	H1	SC	
dof	0	VS	35 3
din	yan	th	
dan	¥	. at	cdc ph
dpwr	35		10 Sept. 14 Sept. 14 Sept. 15
def	6700		

Scheme 4 <sup>31</sup>P/HNMR coupled





20

60

80

100

120

180

160

40