## Characterisation of [RuH(CO)(PPh<sub>3</sub>){H<sub>2</sub>B(mt)<sub>2</sub>}] (3a)

### Commentary

The infrared spectrum reveals absorptions at v = 1936 cm<sup>-1</sup>, 2110 cm<sup>-1</sup>, 2201 cm<sup>-1</sup> and 2399 cm<sup>-1</sup> in CH<sub>2</sub>Cl<sub>2</sub> attributable to the carbonyl, Ru–H, B–H–Ru and terminal B–H groups, respectively. Consistent with  $C_1$  symmetry, the <sup>1</sup>H NMR spectrum shows two distinct NCH<sub>3</sub> singlets ( $\delta_H$  = 3.10, 3.57) and four doublets for the methimazolyl groups ( $\delta_H$  = 6.35, 6.45, 6.62, 6.64; <sup>3</sup>J<sub>HH</sub> = 2.0 Hz). The retention of a ruthenium hydride ligand in the resulting complex is indicated by the appearance in the <sup>1</sup>H NMR spectrum of a high frequency resonance ( $\delta_H$  = -12.22) the doublet multiplicity of which confirms a monophosphine complex with hydride and phosphine ligands mutually *cis*-coordinated (<sup>2</sup>J<sub>PH</sub> = 23.1 Hz).

The 3c-2e Ru–H–B interaction gives rise to a resonance at  $\delta_{\rm H} = -5.45$ , the broadness of which is due to the coupling to the quadrupolar boron nuclei (<sup>10</sup>B and <sup>11</sup>B). The terminal B–H resonance was not located, however there is no apparent site exchange between the terminal B–H and B–H–Ru groups over the temperature range 193 – 305 K, although at 305 K the <sup>11</sup>B quadrupole was sufficiently thermally decoupled that a <sup>1</sup>J<sub>BH</sub> coupling of *ca* 85 Hz could be discerned. The <sup>31</sup>P{<sup>1</sup>H} NMR spectrum shows a singlet at  $\delta$  56.7 ppm and the gross molecular composition was further confirmed by an APCI mass spectrum which included a molecular ion in addition to fragmentations attributable to loss of CO and PPh<sub>3</sub>.

#### <sup>1</sup>H NMR Spectrum of 3a







## <sup>11</sup>B{<sup>1</sup>H} NMR Spectrum of 3a

Parameter	Value		5.5				
Data File Name	/ Users/ kathym/ Documents/ APhD2015/ Abragam/ KMAb113_CDCl3/ 12/ fid						
Origin	Bruker BioSpin GmbH						
Owner	av400bb						
Solvent	CDCl3						
Temperature	300.0						
Pulse Sequence	zg						
Experiment	1D						
Probe	Z116098_0258 (PA BBO 400S1 BBF-H-D-05 Z SP)						
Number of Scans	200						
Receiver Gain	193						
Relaxation Delay	0.2000						
ulse Width	10.0000						
Acquisition Time	1.2845						
Acquisition Date	2016-09-21T17:05:09						
odification Date	2016-09-21T17:18:09		A				
Spectrometer Frequency	128.38		A				
Spectral Width	25510.2						
owest Frequency	-12754.9						
lucleus	11B						
Acquired Size	32768						
Spectral Size	65536						

## <sup>31</sup>P{<sup>1</sup>H} NMR Spectrum of 3a

Data File Name   / Users/ kathym/   Documents/ APhD2015/     Abragam/   KMAb113a_CDC13/ 11/ fid     Origin   Bruker BioSpin ombH     Winer   av400bb     Documents/ APhD2015/   Abragam/     Johner   av400bb     Winer   av400bb     Solvent   CDC13     Solvent   200.0     Value Sequence   zgp30     syperiment   1D     Yobe   2116098_0258 (PA BBO     Momber of Scams   64     Value Sequence   193     Value Sequence   20000     Value Sequenci   10.0	Parameter	Value	1	2						
DriginBruker BioSpin GmbHDvinerav400bbSoldCDC3Pernperature30.0valos Sequence29303typeriment11Noshin BBF-H-D-05 Z SDvalos Sequence30.0valos Sequence30	Data File Name	/ Users/ kathym/ Documents/ APhD2015/ Abragam/ KMAb113a_CDCl3/ 11/ fid	ì	0 <b>C</b> –						
Numerav400bbSolventCDC3Femperature30.0Values Sequenezgg30Solvent10ProbenSilcoss_0258 (PA BB0) SolventValues Cequence64Values Cequence30.0Values Cequence40.0Values Cequence </td <td>Origin</td> <td>Bruker BioSpin GmbH</td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td>	Origin	Bruker BioSpin GmbH								
SolventCDCI3Femperature300.0Vulse Sequence3p30.0Styperiment10Probeno2116098.0258.(PA.BBO) SOIS BBF-H-D-05.Z S.P)Vulmber of Scano40.0Vulse Sequence30.0Vulse Sequence19.0Vulse Sequence10.0Vulse Sequence <td< td=""><td>Owner</td><td>av400bb</td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td></td<>	Owner	av400bb								
Temperature300.0Pulse Sequence2pg30.Experiment10Troban2116098_0258 (PA BBO doS1 BBF-H-D-05 Z SP)Number of Scan64Receiver Gain193Receiver Gain2000Relaxation Delay2000Number of Scan6112Nugbir of Scan1012Relaxation Delay2161-07T15:44:32Nugbir of Scan1012Nugbir of S	Solvent	CDCl3								
Pulse Sequencespg30Experiment10Probe2116098_0258 (PA BBO 40051 BBF-H-D-05 Z SP)Pulmer of Scan64Keceiver Gain193keceiver Gain2000keceiver Gain6.000Vage Width8.000kugistion Time05112kequistion Time01612-07115:44:32kequistion Time16.197kequistion Time64.02.6kequistion Time65.06	Temperature	300.0								
Experiment1DProbe2116098_0258 (PA BBO 0051 BBF-H-D-05 Z SP)Number of Sca644013Receiver Gain13090002000Vase Width80000511214Vase Width0161-2-07115:44:32Vase Width1016-2-07115:44:32Vase Width610-2-07115:44:32Vase Width610-2-07115:46:43Vase Width610-2-071110:46:43Vase Width	Pulse Sequence	zgpg30								
Probe2116098_0258 (PA BBO 40051 BBF-H-D-05 Z SP)Number of Scase6464193Receiver Gain1931932000Relaxation Delay20008.0001Valse Width8.0000.51121Koquisition Time0.51120.1612-07T15:44:324odification Data2016-12-07T15:44:32104Gification Data610-9spectro Width6102.6610.91010.1spectro Width4102.6Juceus31Juceus31spectra Width3268spectra Width536	Experiment	1D								
Number of Scans     64       Receiver Gain     193       Relaxation Delay     2.0000       Pulse Width     8.0000       Pulse Width     8.0000       Pulse Width     0.5112       Requisition Date     2016-12-07T15:44:32       Pulse Width     2016-12-07T15:46:43       Spectrometer     161.97       requency	Probe	Z116098_0258 (PA BBO 400S1 BBF-H-D-05 Z SP)								
Receiver Gain193193Relaxation Dela2.00001bulse Width8.00001valuisition Time0.5112valuisition Data2016-12-07T15:44:32Vadification Data2016-12-07T15:46:43bjectrometer requency161.97bjectral Width64102.66values40150.1values19values31Pvalues32768bjectral Size6536	Number of Scans	64		1						
Relaxation Delay2.0000Image: constraint of the sector of the secto	Receiver Gain	193		1						
Pulse Width8.0000Acquisition Time0.5112Acquisition Date2016-12-07T15:44:32Aodification Date2016-12-07T15:46:43Spectrometer161.97Spectral Width64102.6Auguess Frequency	Relaxation Delay	2.0000								
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Nucleus 31P   Acquired Size 32768   Sipectral Size 65536	Lowest Frequency	-40150.1								
Acquired Size 32768 Spectral Size 65536	Nucleus	31P								
Spectral Size 65536	Acquired Size	32768								
	Spectral Size	65536								
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## <sup>13</sup>C{<sup>1</sup>H} NMR Spectrum of 3a

34.3 25.7 18.5

Parameter	Value
ata File Name	/ Users/ kathym/ Desktop/ Fids/ KMAb113a_carbon/ 2/ fid
Origin	Bruker BioSpin GmbH
Dwner	hill
Solvent	CDCl3
Temperature	298.8
Pulse Sequence	1D_C.go
Experiment	1D
Probe	Z129133_0008 (PA BBO 700S3 BB-H&F-D-05 Z)
Number of Scans	18604
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	10.2500
Acquisition Time	0.5308
Acquisition Date	2017-02-01T08:54:12
Modification Date	2017-02-14T17:47:24
Spectrometer Frequency	176.09
Spectral Width	61728.4
Lowest Frequency	-13235.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536
	Service and a second state
260 2	40 220 200
200 2	10 220 200

#### <sup>13</sup>C{<sup>1</sup>H} NMR Spectrum of 3a (Phosphine Region)



Characterisation of [RuCl(CO)(PPh\_3){H\_B(mt)\_3] (3b)

#### <sup>1</sup>H NMR Spectrum of 3b



#### <sup>1</sup>H NMR Spectrum of 3b (Expansion)



## <sup>11</sup>B{<sup>1</sup>H} NMR Spectrum of 3b

Data File Name   E:/ APhD2015/ Abragam/ KMAb131_furtherEtOHwash_CDCl3 12/ fd     Title   KMAb131_furtherEtOHwash_CDCl3     Origin   Bruker BioSpin GmbH     Owner   av400bb     Solvent   CDCl3     Temperature   300.0     Pulse Sequence   zg     Experiment   ID     Number of Scans   200     Receiver Gain   193     Relaxation Delay   0.2000     Pulse Width   10.0000     Acquisition Time   1.2845     Acquisition Time   1.2845     Spectral Width   2510.2     Lowest   -12763.4     Frequency   11B     Acquired Size   32768     Spectral Size   65336	Parameter	Value	
Title     KMAb131_furtherEtOHwash_CDCl3       Origin     Bruker BioSpin GmbH       Owner     av400bb       Solvent     CDCl3       Temperature     300.0       Pulse Sequence     zg       Experiment     10       Number of Scans     200       Receiver Gain     193       Relaxation Delay     0.000       Acquisition Time     1.2845       Acquisition Tome     2166-11-30T12:14:01       Modificaction Date     2016-11-30T12:36:46       Spectrometer     28.38       Frequency	Data File Name	E:/ APhD2015/ Abragam/ KMAb131_furtherEtOHwash_CDCl3/ 12/ fid	
Origin     Bruker BioSpin GmbH       Owner     av400bb       Solvent     CDCI3       Temperature     300.0       Pulse Sequence     zg       Experiment     1D       Number of Scans     200       Receiver Gain     193       Relaxation Delay     0.2000       Pulse Vidth     10.0000       Acquisition Time     1.2845       Acquisition Time     1.2845       Acquisition Time     1.2845       Acquisition Action Delay     2016-11-30T12:136:16       Spectrometer     12763.4       Frequency     Variation Action Acti	Title	KMAb131 furtherEtOHwash CDCl3	
Owner     av400bb       Solvent     CDCI3       Temperature     300.0       Pulse Sequence     zg       Experiment     1D       Number of Scars     200       Receiver Gain     193       Relaxation Delay     0.2000       Pulse Width     10.0000       Acquisition Time     1.2845       Acquisition Table     2016-11-30T12:14:01       Modification Date     2016-11-30T12:36:46       Spectral Width     25510.2       Lowest     -12763.4       Frequency	Oriain	Bruker BioSpin GmbH	
Solvent     CDCl3       Temperature     300.0       Pulse Sequence     zg       Experiment     1D       Number of Scans     200       Receiver Gain     193       Relaxation Delay     0.2000       Pulse Width     10.0000       Acquisition Time     1.2845       Acquisition Time     2016-11-30T12:14:01       Modification Dats     2016-11-30T12:36:46       Spectrometer     128.38       Frequency     -       Vucleus     11B       Acquired Size     32768       Spectral Size     5536	Owner	av400bb	
Temperature     300.0       Pulse Sequence     zg       Speriment     1D       Number of Scans     200       Receiver Gain     193       Relaxation Delay     0.2000       Pulse Width     10.0000       Acquisition Time     1.2845       Acquisition Tabe     2016-11-30T12:14:01       Modification Date     2016-11-30T12:36:46       Spectrometer     128.38       Frequency	Solvent	CDCl3	
Pulse Sequence     zg       Experiment     ID       Number of Scars     200       Receiver Gain     193       Relaxation Delay     0.2000       Pulse Width     10.0000       Acquisition Time     1.2845       Acquisition Date     2016-11.30T12:14:01       Modification Date     2016-11.30T12:36:46       Spectrometer     128.38       Frequency     -       Spectral Width     25510.2       Lowest     -12763.4       Frequency     -       Spectral Size     65536	Temperature	300.0	
Experiment     1D       Number of Scans     200       Receiver Gain     193       Relaxation Delay     0.2000       Pulse Width     10.0000       Acquisition Time     1.2845       Acquisition Time     2016-11-30T12:14:01       Modification Date     2016-11-30T12:36:46       Spectrometer     128.38       Frequency	Pulse Sequence	70	
Lay and the stand of the s	Experiment	1D	
Receiver Gain 193 Relaxation Delay 0.2000 Pulse Width 10.0000 Acquisition Time 1.2845 Acquisition Date 2016-11-30T12:14:01 Modification Date 2016-11-30T12:36:46 Spectrometer 128.38 Frequency Spectral Width 25510.2 Lowest -12763.4 Frequency Nucleus 11B Acquired Size 32768 Spectral Size 65536	Number of Scans	200	
Relaxation Delay 0.2000 Pulse Width 10.0000 Acquisition Time 1.2845 Acquisition Date 2016-11-30T12:14:01 Modification Date 2016-11-30T12:36:46 Spectrometer 128.38 Frequency Spectral Width 25510.2 Lowest -12763.4 Frequency Nucleus 11B Acquired Size 32768 Spectral Size 65536	Receiver Gain	103	
Nucleus   0.0000     Acquisition Time   1.2845     Acquisition Date   2016-11-30T12:14:01     Modification Date   2016-11-30T12:36:46     Spectrometer   128.38     Frequency   Spectral Width     Spectral Width   25510.2     Lowest   -12763.4     Frequency   Nucleus     Nucleus   11B     Acquired Size   32768     Spectral Size   65536	Relaxation Delay	0.2000	
Acquisition Time 1.2845 Acquisition Date 2016-11-30T12:14:01 Modification Date 2016-11-30T12:36:46 Spectrometer 128.38 Frequency Spectral Width 25510.2 Lowest -12763.4 Frequency Nucleus 11B Acquired Size 32768 Spectral Size 65536	Pulse Width	10,0000	
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Spectral Width 25510.2 Lowest -12763.4 Frequency Nucleus 11B Acquired Size 32768 Spectral Size 65536	Modification Date	2016-11-30T12:36:46	
Spectral Width 25510.2 Lowest -12763.4 Frequency Nucleus 11B Acquired Size 32768 Spectral Size 65536	Spectrometer Frequency	128.38	
Lowest -12763.4 Frequency Nucleus 11B Acquired Size 32768 Spectral Size 65536	Spectral Width	25510.2	
Nucleus 11B Acquired Size 32768 Spectral Size 65536	Lowest Frequency	-12763.4	A CONTRACTOR OF A CONTRACTOR A CONTRA
Acquired Size 32768 Spectral Size 65536	Nucleus	11B	
Spectral Size 65536	Acquired Size	32768	
	Spectral Size	65536	

### <sup>31</sup>P{<sup>1</sup>H} NMR Spectrum of 3b

Data File Name Fitle Drigin Dwner Golvent Temperature Yulse Sequence	E:/ APhD2015/ Abragam/ KMAb131_DCMEtOHworkup_CDCl3/ 11/ fid KMAb131_DCMEtOHworkup_CDCl3 Bruker BioSpin GmbH av400bb CDCl3 200.0			(n 									
Fitle Drigin Dwner Solvent Temperature Yulse Sequence	KMAb131_DCMEtOHworkup_CDCl3 Bruker BioSpin GmbH av400bb CDCl3 200.0												
Drigin Dwner Solvent Temperature Yulse Sequence	Bruker BioSpin GmbH av400bb CDCl3												
Dwner Solvent Temperature Yulse Sequence	av400bb CDCl3												
Solvent Femperature Pulse Sequence	CDCl3			1									
Temperature Pulse Sequence	200.0			i i									
ulse Sequence	300.0												
	zapa30												
-xperiment	1D												
Jumber of Scans	64												
Receiver Gain	193												
Relaxation Delay	2.0000												
ulse Width	8.0000												
Acquisition Time	0.5112												
Acquisition Date	2016-11-30T10:13:57												
1odification Date	2016-11-30T10:14:08												
pectrometer requency	161.98												
Spectral Width	64102.6												
owest requency	-39986.0												
lucleus	31P												
cquired Size	32768												
pectral Size	65536												
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and any other and a second star from the second star of the	ni ne mining in the state state for internal party gaining as a free birth party internal to by federal amount of y basis party	and here the second second	and an a second state of some	and and a second standard and a second			and a sub-	and a second second second					1
130 12	0 110 100 90 80	70	60 50	) 40	30	20	10	0	-10	-20	-30	-40	

#### <sup>13</sup>C{<sup>1</sup>H} NMR Spectrum of 3b





34.67

-18.60

#### <sup>13</sup>C{<sup>1</sup>H} NMR Spectrum of 3b (Expansion)



## Characterisation of [Ru(SePh)(CO)(PPh<sub>3</sub>){H<sub>2</sub>B(mt)<sub>2</sub>}] (3c)

## Commentary

Spectroscopic data for **3b** call for little comment other than to note the conspicuous loss of the hydride doublet resonance in the <sup>1</sup>H NMR spectrum, whilst the broad B–H–Ru resonance shifts to  $\delta_{\rm H}$  = –18.11.

#### <sup>1</sup>H NMR Spectrum of 3c



#### <sup>1</sup>H NMR Spectrum of 3c (Expansion)



## <sup>11</sup>B{<sup>1</sup>H} NMR Spectrum of 3c

		-5.50
Parameter	Value	
Data File Name	/ Users/ kathym/ Desktop/ Fids/ KMAb144_char_CDCl3/ 13/ fid	
Origin	Bruker BioSpin GmbH	
Owner	av400bb	
Solvent	CDCl3	
Temperature	300.0	
Pulse Sequence	zgpg	
Experiment	1D	
Probe	Z116098_0258 (PA BBO 400S1 BBF-H-D-05 Z SP)	
Number of Scans	200	
Receiver Gain	193	
Relaxation Delay	0.2000	
Pulse Width	10.0000	
Acquisition Time	1.2845	
Acquisition Date	2017-04-03T11:45:02	
Modification Date	2017-04-10T18:32:44	
Spectrometer Frequency	128.38	
Spectral Width	25510.2	
Lowest Frequency	-12754.9	
Nucleus	11B	
Acquired Size	32768	
Spectral Size	65536	

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## <sup>31</sup>P{<sup>1</sup>H} NMR Spectrum of 3c

Parameter	Value		6								
Data File Name	/ Users/ kathym/ Desktop/ Fids/ KMAb144_char_CDCl3/ 11/ fid		(n 								
Origin	Bruker BioSpin GmbH										
Owner	av400bb										
Solvent	CDCl3										
Temperature	300.0										
Pulse Sequence	zapa30										
Experiment	1D										
Probe	Z116098_0258 (PA BBO 400S1 BBF-H-D-05 Z SP)										
Number of Scans	128										
Receiver Gain	193										
Relaxation Delay	2.0000										
Pulse Width	8.0000										
Acquisition Time	0.5112										
Acquisition Date	2017-04-03T11:32:18										
Modification Date	2017-04-10T18:32:44										
Spectrometer Frequency	161.97										
Spectral Width	64102.6										
Lowest Frequency	-40150.1										
Nucleus	31P										
Acquired Size	32768										
Spectral Size	65536										
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0 120	100 80	60		20	0	10	20		70	00	110

#### <sup>13</sup>C{<sup>1</sup>H} NMR Spectrum of 3c



#### <sup>13</sup>C{<sup>1</sup>H} NMR Spectrum of 3c (Expansions)









## Characterisation of [Ru(BCat)(CO)(PPh<sub>3</sub>){H<sub>2</sub>B(mt)<sub>2</sub>}] (3e)

#### Commentary

The formulation of **3e** follows from spectroscopic data akin to those for **3a-c** in addition to the appearance of two resonances in the <sup>11</sup>B NMR spectrum. The resonance at  $\delta_{11B} = -5.44$  corresponds to the BH<sub>2</sub> group (*cf*.  $\delta_{11B} = -5.5$  for **3a**) whilst that at  $\delta_{11B} = 52.3$  corresponds to the trigonal boron of the BCat ligand. The <sup>31</sup>P{<sup>1</sup>H} NMR spectrum comprises a single *sharp* resonance ( $\delta_P = 47.2$ ) confirming that the boryl and phosphine ligands are mutually *cis*.

#### <sup>1</sup>H NMR Spectrum of 3e



#### <sup>11</sup>B{<sup>1</sup>H} NMR Spectrum of 3e



## <sup>31</sup>P{<sup>1</sup>H} NMR Spectrum of 3e

Data File Name / Users/ kathym/ Documents/ APD2015/ Abragam/ KMAb135 DCMEtOHwor kup_CDC13/ 11 /f id Title KMAb135 DCMEtOHwor kup_CDC13 Origin Bruker BioSpin GmbH Owner av400bb Solvent CDC13 Temperature 300.0 Pulse Sequence zgpg30 Experiment 1D Number of Scans 64 Receiver Gain 193 Relaxation Delay 2.0000 Pulse Width 8.0000 Pulse Width 8.0000 Pulse Vidth 8.0000 Pulse Vidth 6.0102 Spectrometer 161.97 Frequency 40150.1 Nucleus 31P Acquired Size 32768 Spectral Size 65536	Data File Name / Users/ kathym/ Documents/ APh2015/ Abragam/ KMAb135_DCMEEOHwor kup_CDCL3 / 11/ fid KWAb135_DCMEEOHwor kup_CDCL3 Title KMAb135_DCMEEOHwor kup_CDCL3 Origin Bruker BioSpin GmbH Owner av400bb Solvent CDC3 Temperature 300.0 Pulse Sequence zgpg30 Experiment 1D Number of Scans 64 Receiver Gain 193 Relaxation Delay 2.0000 Pulse Width 8.0000 Acquisition Tube 2016-12-08T20:58:06 Spectrometer 161.97 Frequency Spectral Width 64102.6 Lowest Frequency - 40150.1 Nucleus 31P Acquired Size 32768 Spectral Size 65536	Parameter	Value		7.1								
KMADL35_LOMEKUHWORkup_CDCL31/11/idTitleKMAb135_DCMEt0Hworkup_CDCL3briginBruker BioSpin GmbHDomerav400bbSolventCDCI3Emperature300.0Vulse Sequencezgpg30Experiment10Vumber of Scans64Seceiver Gain193Relaxation Delay2.0000Vulse Width8.0000Acquiristion Time0.5112Lequisition Table2016-12-08T20:58:06Spectral Uddt64102.6Jowest Frequency40150.1Vucleus31PAcquired Size32768Spectral Size65336	KMAD 13-5_UCMEUDHWOR     KUP_COCL3/ 11/ fd     Tritle   KMAD 135_DCMEXDHWOR     kup_COCL3/   KMAD 135_DCMEXDHWOR     kup_COCL3/   KMAD 135_DCMEXDHWOR     brigin   Bruker BioSpin GmbH     Domer   av400bb     Solvent   CDCI3     Emperature   30.0     Pulse Sequence   zpg30     Experiment   1D     Vumber of Scans   64     Seceiver Gain   193     Relaxation Delay   2.0000     Vulse Width   8.0000     Acquisition Time   0.5112     Acquisition Tab   2016-12-08T20:58:06     Spectral Midth   64102.6     Cowest Frequency   40150.1     Vulceus   31P     Acquired Size   32768     Spectral Size   6536	Data File Name	/ Users/ kathym/ Documents/ APhD2015/ Abragam/		-47								
Kap_GOC_JAT/RdTitleKup_COCL3OriginBruker BioSpin GmbHOwnerav400bbSolventCDCl3Temperature300.0Pules Sequencezpg30Experiment1DNumber of Scans64Receiver Gain193Relaxation Delay2.0000Pules Width8.0000Acquisition Time0.5112Acquisition Tota2016-12-08720:58:06Spectral Width64102.6Lowest Frequency-40150.1Nucleus31PAcquisition Size65536	Kup_Cocl3OriginBruker BioSpin GmbHOwnerav400bbSolventCDCl3Temperature300.0Pulse Sequencezgpg30Experiment1DNumber of Scans64Receiver Gain193Relaxation Delay2.0000Pulse Width8.0000Acquisition Time0.511.2Acquisition Time0.511.2Acquisition Time0.516.12-08T20:58:06Spectrometer161.97Frequency-Spectral Width31PAcquired Size32768Spectral Size65536		KMAb135_DCMEtOHwor										
Origin     Bruker BioSpin GmbH       Owner     av400bb       Solvent     CDCI3       Temperature     300.0       Pulse Sequence     zgpg30       Experiment     1D       Number of Scans     64       Receiver Gain     193       Relaxation Delay     2.0000       Pulse Width     8.0000       Acquisition Time     0.5112       Acquisition Time     0.5112       Acquisition Date     2016-12-08T20:58:06       Spectrometer     161.97       Frequency     40150.1       Nucleus     31P       Acquired Size     32768       Spectral Size     65536	Origin     Bruker BioSpin GmbH       Owner     av400bb       Solvent     CDCl3       Temperature     300.0       Pulse Sequence     zgpg30       Experiment     1D       Number of Scans     64       Receiver Gain     193       Relaxation Delay     2.0000       Pulse Width     8.0000       Acquisition Time     0.5112       Acquisition Time     0.5112       Acquisition Time     0.5112       Acquisition Time     0410.2.6       Lowest Frequency     40150.1       Nucleus     31P       Acquired Size     32768       Spectral Size     65536	Title	KMAb135_DCMEtOHwor kup_CDCL3		1								
Ownerav400bbSolventCDCl3Temperature300.0Pulse Sequencezgp30Experiment1DNumber of Scans64Receiver Gain193Relaxation Delay2.0000Pulse Width8.0000Acquisition Time0.5112Acquisition Time0.5112Spectrometer161.97Frequency	Owner     av400bb       Solvent     CDCI3       Temperature     300.0       Pulse Sequence     zgpg30       Experiment     1D       Number of Scans     64       Receiver Gain     193       Relaxation Delay     2.0000       Pulse Writh     8.0000       Acquisition Tem     0.5112       Acquisition Date     2016-12-08T20:58:06       Spectral Width     64102.6       Lowest Frequency     -40150.1       Nucleus     31P       Acquired Size     32768       Spectral Size     65536	Origin	Bruker BioSpin GmbH		1								
Solvent     CDCl3       Temperature     300.0       Pulse Sequence     zgp30       Experiment     1D       Number of Scans     64       Receiver Gain     193       Relaxation Delay     2.0000       Pulse Width     8.0000       Acquisition Time     0.5112       Acquisition Scans     64102.6       Lowest Frequency	Solvent     CDCl3       Temperature     300.0       Pulse Sequence     zgpg30       Experiment     1D       Number of Scans     64       Receiver Gain     193       Relaxation Delay     2.0000       Acquisition Time     0.5112       Acquisition Time     0.5112       Acquisition Delay     2016-12-08T20:58:06       Spectrometre     161.97       Frequency     -       Spectrometre     101.1       Nucleus     31P       AcquisitSize     32768       Spectral Size     65536	Owner	av400bb										
Temperature   300.0     Pulse Sequence   zgp30     Experiment   1D     Number of Scans   64     Receiver Gain   193     Relaxation Delay   2.0000     Pulse Width   8.0000     Acquisition Time   0.5112     Acquisition Date   2016-12-08T20:58:06     Spectrometer   161.97     Frequency   -     Spectral Width   64102.6     Lowest Frequency   -     Spectral Size   32768     Spectral Size   65536	Temperature   300.0     Pulse Sequence   zgpg30     Experiment   10     Number of Scans   64     Receiver Gain   193     Relaxation Delay   2.0000     Pulse Width   8.0000     Acquisition Time   0.5112     Acquisition Date   2016-12-08T20:58:06     Spectrometer   161.97     Frequency	Solvent	CDCl3										
Pulse Sequence     zgp30       Experiment     1D       Number of Scans     64       Receiver Gain     193       Relaxation Delay     2.0000       Pulse Width     8.0000       Acquisition Time     0.5112       Acquisition Date     2016-12-08T20:58:06       Spectrometer     16.197       Frequency     -       Spectral Width     64102.6       Lowest Frequency     -       Spectral Size     31P       Acquired Size     32768       Spectral Size     65536	Pulse Sequence     zgpg30       Experiment     1D       Number of Scans     64       Receiver Gain     193       Relaxation Delay     2.0000       Pulse Width     8.0000       Acquisition Time     0.5112       Acquisition Date     2016-12-08T20:58:06       Spectrometer     161.97       Frequency	Temperature	300.0										
Experiment   1D     Number of Scans   64     Receiver Gain   193     Relaxation Delay   2.0000     Pulse Width   8.0000     Acquisition Time   0.5112     Acquisition Date   2016-12-08T20:58:06     Spectrometer   161.97     Frequency   -     Spectral Width   64102.6     Lowest Frequency   -40150.1     Nucleus   31P     Acquired Size   32768     Spectral Size   65536	Experiment   1D     Number of Scans   64     Receiver Gain   193     Relaxation Delay   2.0000     Pulse Width   8.0000     Acquisition Time   0.5112     Acquisition Date   2016-12-08T20:58:06     Spectrometer   161.97     Frequency   -     Spectral Width   64102.6     Lowest Frequency   -40150.1     Nucleus   31P     Acquired Size   32768     Spectral Size   65536	Pulse Sequence	zgpg30										
Number of Scans   64     Receiver Gain   193     Relaxation Delay   2.0000     Pulse Width   8.0000     Acquisition Time   0.5112     Acquisition Date   2016-12-08T20:58:06     Spectron Here   161.97     Frequency   -     Spectral Width   64102.6     Lowest Frequency   -40150.1     Nucleus   31P     Acquired Size   32768     Spectral Size   65536	Number of Scans   64     Receiver Gain   193     Relaxation Delay   2.0000     Pulse Width   8.0000     Acquisition Time   0.5112     Acquisition Date   2016-12-08T20:58:06     Spectrometer   161.97     Frequency   Spectral Width     Spectral Width   64102.6     Lowest Frequency   40150.1     Nucleus   31P     Acquired Size   32768     Spectral Size   65536	Experiment	1D										
Receiver Gain   193     Relaxation Delay   2.0000     Pulse Width   8.0000     Acquisition Time   0.5112     Acquisition Date   2016-12-08T20:58:06     Spectrometer   161.97     Frequency   Spectral Width     Spectral Width   64102.6     Lowest Frequency   -40150.1     Nucleus   31P     Acquired Size   32768     Spectral Size   65536	Receiver Gain     193       Relaxation Delay     2.0000       Pulse Width     8.0000       Acquisition Time     0.5112       Acquisition Date     2016-12-08T20:58:06       Spectrometer     161.97       Frequency	Number of Scans	64										
Relaxation Delay   2.0000     Pulse Width   8.0000     Acquisition Time   0.5112     Acquisition Date   2016-12-08T20:58:06     Spectrometer   161.97     Frequency   Spectral Width     Spectral Width   64102.6     Lowest Frequency   -40150.1     Nucleus   31P     Acquired Size   32768     Spectral Size   65536	Relaxation Delay   2.0000     Pulse Width   8.0000     Acquisition Time   0.5112     Acquisition Date   2016-12-08T20:58:06     Spectrometer   16.197     Frequency   -     Spectral Width   64102.6     Lowest Frequency   -40150.1     Nucleus   31P     Acquired Size   32768     Spectral Size   65536	Receiver Gain	193										
Pulse Width8.0000Acquisition Time0.5112Acquisition Date2016-12-08T20:58:06Spectrometer161.97Frequency-Spectral Width64102.6Lowest Frequency-40150.1Nucleus31PAcquired Size32768Spectral Size65536	Pulse Width 8.000 Acquisition Time 0.5112 Acquisition Date 2016-12-08T20:58:06 Spectrometer 161.97 Frequency 5 Spectral Width 64102.6 Lowest Frequency -40150.1 Nucleus 31P Acquired Size 32768 Spectral Size 65536	Relaxation Delay	2.0000										
Acquisition Time0.5112Acquisition Date2016-12-08T20:58:06Spectrometer161.97Frequency	Acquisition Time0.5112Acquisition Date2016-12-08T20:58:06Spectrometer161.97Frequency	Pulse Width	8.0000										
Acquisition Date2016-12-08T20:58:06Spectrometer161.97Frequency-Spectral Width64102.6Lowest Frequency-40150.1Nucleus31PAcquired Size32768Spectral Size65536	Acquisition Date 2016-12-08T20:58:06 Spectrometer 161.97 Frequency Spectral Width 64102.6 Lowest Frequency -40150.1 Nucleus 31P Acquired Size 32768 Spectral Size 65536	Acquisition Time	0.5112										
Spectrometer161.97Frequency-Spectral Width64102.6Lowest Frequency-40150.1Nucleus31PAcquired Size32768Spectral Size65536	Spectrometer161.97FrequencySpectral Width64102.6Lowest Frequency-40150.1Nucleus31PAcquired Size32768Spectral Size65536	Acquisition Date	2016-12-08T20:58:06										
Spectral Width64102.6Lowest Frequency-40150.1Nucleus31PAcquired Size32768Spectral Size65536	Spectral Width64102.6Lowest Frequency-40150.1Nucleus31PAcquired Size32768Spectral Size65536	Spectrometer Frequency	161.97										
Lowest Frequency -40150.1 Nucleus 31P Acquired Size 32768 Spectral Size 65536	Lowest Frequency -40150.1 Nucleus 31P Acquired Size 32768 Spectral Size 65536	Spectral Width	64102.6										
Nucleus 31P Acquired Size 32768 Spectral Size 65536	Nucleus 31P Acquired Size 32768 Spectral Size 65536	Lowest Frequency	-40150.1										
Acquired Size 32768 Spectral Size 65536	Acquired Size 32768 Spectral Size 65536	Nucleus	31P										
Spectral Size 65536	Spectral Size 65536	Acquired Size	32768										
		Spectral Size	65536										
		Spectral Size	65536										
		130	110 90 80 7	0 60	50 40	30 20	) 10	0	-20	-40	-60	-80	-1

#### HSQC <sup>13</sup>C-<sup>1</sup>H NMR Spectrum of 3e



#### <sup>13</sup>C{<sup>1</sup>H} NMR Spectrum of 3e





## <sup>13</sup>C{<sup>1</sup>H} NMR Spectrum of 3e (Expansion)

Data File Name / Users/ kathym/ Documents/ APhD2015/ Abragam/ KMAb135 DCMECOH workup_CDCL3/ 13/ fid Domer av400bb Solvent CDCI3 Femperature 300.0 Pulse Sequence 2pp30 Experiment 1D Number of Scans 6000 Receiver Gain 193 Relaxation Delay 2.0000 Pulse Width 10.0000 Acquisition Time 1.3631 Acquired Size 32768 Spectral Vidth 24038.5 Lowest Frequency 1943.1 Nucleus 13C Acquired Size 32768	Parameter	Value	.15	.76		46	37.37.37.37.37.37.37.37.37.37.37.37.37.3	.85
Drigin     Bruker BioSpin GmbH       Wmer     av400bb       Solvent     CDC3       Temperature     300.0       Values Sequence     zzpg30       xizyperiment     ID       Number of Scans     6000       teceiver Gain     193       telaxation Delay     2.0000       Pulse Width     10.0000       kcquisition Time     1.3631       kcquisition Date     2016-12-09T02:58:40       pectral Width     24038.5       cowset Frequency     -1943.1       values     32768       spectral Size     65336	Data File Name	/ Users/ kathym/ Documents/ APhD2015/ Abragam/ KMAb135_DCMEtOH workup_CDCL3/ 13/ fid	×136	133 133	$\begin{matrix} 129\\128\\127\\127\end{matrix}$	101,	121 121 120 120 120	-110
Dvner     av400bb       Solvent     CDC3       Temperature     300.0       Pulse Sequence     zgpg30       Experiment     1D       Number of Scans     6000       Receiver Gain     193       Relaxation Delay     2.0000       Pulse Width     10.0000       Acquisition Time     1.3631       Acquisition Time     1.062       Frequency     1943.1       Vucleus     13C       Acquired Size     25536	Drigin	Bruker BioSpin GmbH						
Solvent     CDCl3       Fernperature     300.0       Vulse Sequence     zpg30       Experiment     1D       Jumber of Scans     6000       Receiver Gain     193       Relaxation Delay     2.0000       Vulse Width     10.0000       Acquisition Time     216-12-09T02:58:40       Spectrometer     100.62       requency     - 1943.1       Vulces     13C       Acquiristion Zaita     2768       Spectral Size     65536	Dwner	av400bb						
Temperature     300.0       Yulse Sequence     2gp30       Steperiment     1D       Number of Scans     6000       Reelever Gain     193       Relaxation Delay     2.0000       Yulse Width     10.0000       Acquisition Time     1.3631       Acquisition Date     2016-12-09T02:58:40       Spectrometer     100.62       Frequency     -1943.1       Vucleus     13C       Acquired Size     32768       Spectral Size     65536	Solvent	CDCl3						
Pulse Sequence zgp30 Experiment 1D Number of Scans 6000 Receiver Gain 193 Relaxation Delay 2.0000 Pulse Width 10.0000 Acquisition Date 2016-12-09T02:58:40 Spectral Width 24038.5 Lowest Frequency - 1943.1 Nucleus 13C Acquired Size 32768 Spectral Size 65536	Temperature	300.0						
Experiment     1D       Number of Scans     6000       Receiver Gain     193       relaxation Delay     2.0000       Pulse Width     10.0000       Acquisition Time     1.3631       Acquisition Time     1.3631       Acquisition Date     2016-12-09T02:58:40       Spectronetre     100.62       Trequency	Pulse Sequence	zgpg30						
Number of Scans 6000 Receiver Gain 193 Relaxation Delay 2.0000 Pulse Width 10.0000 Acquisition Time 1.3631 Acquisition Date 2016-12-09T02:58:40 Spectral Width 24038.5 Lowest Frequency -1943.1 Nucleus 13C Acquired Size 32768 Spectral Size 65536	Experiment	1D						
Receiver Gain 193 Relaxation Delay 2.0000 Pulse Width 10.0000 Acquisition Time 1.3631 Acquisition Date 2016-12-09T02:58:40 Spectrometer 100.62 Frequency Spectral Width 24038.5 Lowest Frequency - 1943.1 Nucleus 13C Acquired Size 32768 Spectral Size 65536	Number of Scans	6000						
Relaxation Delay 2.0000 Pulse Width 10.0000 Acquisition Time 1.3631 Acquisition Date 2016-12-09T02:58:40 Spectrometer 100.62 Frequency -1943.1 Nucleus 13C Acquired Size 32768 Spectral Size 65536	Receiver Gain	193						
Pulse Width 10.0000 Acquisition Time 1.3631 Acquisition Date 2016-12-09T02:58:40 Spectrometer 100.62 Frequency 5 Spectral Width 24038.5 Lowest Frequency -1943.1 Nucleus 13C Acquired Size 32768 Spectral Size 65536	Relaxation Delay	2.0000						
Acquisition Time 1.3631 Acquisition Date 2016-12-09T02:58:40 Spectrometer 100.62 Frequency Spectral Width 24038.5 Lowest Frequency -1943.1 Nucleus 13C Acquired Size 32768 Spectral Size 65536	Pulse Width	10.0000						
Acquisition Date 2016-12-09T02:58:40 Spectrometer 100.62 Frequency Spectral Width 24038.5 Lowest Frequency -1943.1 Nucleus 13C Acquired Size 32768 Spectral Size 65536	Acquisition Time	1.3631						
Spectrometer 100.62 Frequency Spectral Width 24038.5 Lowest Frequency -1943.1 Nucleus 13C Acquired Size 32768 Spectral Size 65536	Acquisition Date	2016-12-09T02:58:40						
Spectral Width 24038.5 Lowest Frequency -1943.1 Nucleus 13C Acquired Size 32768 Spectral Size 65536	Spectrometer Frequency	100.62						
Lowest Frequency -1943.1 Nucleus 13C Acquired Size 32768 Spectral Size 65536	Spectral Width	24038.5						
Nucleus 13C Acquired Size 32768 Spectral Size 65536	Lowest Frequency	-1943.1						
Acquired Size 32768 Spectral Size 65536	Nucleus	13C						
Spectral Size 65536	Acquired Size	32768					10	
	Spectral Size	65536						

# Characterisation of [Ru(SiCl<sub>3</sub>)(CO)(PPh<sub>3</sub>){H<sub>2</sub>B(mt)<sub>2</sub>}] (3f)

#### Commentary

The infrared spectrum of **3f** included weak absorptions at 2430 and 2046 cm<sup>-1</sup> consistent with  $\kappa^3$ -*H*,*S*,*S'* coordination of the borate which was further supported by a broad resonance at -5.74 ppm in the <sup>1</sup>H NMR spectrum.

#### <sup>1</sup>H NMR Spectrum of 3f



#### <sup>1</sup>H NMR Spectrum of 3f (Expansion)



## <sup>11</sup>B{<sup>1</sup>H} NMR Spectrum of 3f

Parameter	Value
Data File Name	/ Users/ kathym/ Documents/ APhD2015/ Abragam/ KMAb136b C6D6/ 13/ fid
Origin	Bruker BioSpin GmbH
Owner	av400bb
Solvent	C6D6
Temperature	300.0
Pulse Sequence	zgpg
Experiment	1D
Number of Scans	200
Receiver Gain	193
Relaxation Delay	0.2000
Pulse Width	10.0000
Acquisition Time	1.2845
Acquisition Date	2017-02-23T13:04:33
Spectrometer Frequency	128.38
Spectral Width	25510.2
Lowest Frequency	-12754.9
Nucleus	11B
Acquired Size	32768
Spectral Size	65536

-1.35 --4.71

90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 f1 (ppm)

## <sup>31</sup>P{<sup>1</sup>H} NMR Spectrum of 3f

		40.75	19.67							
Parameter	Value									
Data File Name	/ Users/ kathym/ Documents/ APhD2015/ Abragam/ KMAb136a_workup_DCN Ethanol_rotorvap_air_C0 D6/ 11/ fid	1								
Origin	Bruker BioSpin GmbH									
Owner	av400bb									
Solvent	C6D6									
Temperature	300.0									
Pulse Sequence	zgpg30									
Experiment	1D									
Number of Scans	64									
Receiver Gain	193									
Relaxation Delay	2.0000	÷								
Pulse Width	8.0000									
Acquisition Time	0.5112									
Acquisition Date	2017-02-24T15:53:53									
Spectrometer Freque	ncy 161.97									
Spectral Width	64102.6		1							
Lowest Frequency	-40150.1		i i							
Nucleus	31P									
Acquired Size	32768									
Spectral Size	65536									
Lines Hills & confusion attenue of the base	anna a ta in an Alla bail, aist an Alla baile an Alla anna an Alla baile an Alla		g Hestin, to be dealed at the point	And the state of the		all for a share of the second	Real Property in the second state	nordea total destances	il interimite in the state	A Real Production of the second second
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	100 00	60 40			10	20	F.			
J 120	100 80	60 4C	0 20 f1	(nnm)	-10	-30	-50	-70	-90	-110

Characterisation of [Ru(SiMe<sub>3</sub>)(CO)(PPh<sub>3</sub>){H<sub>2</sub>B(mt)<sub>2</sub>}] (3f)

#### <sup>1</sup>H NMR Spectrum of 3g



#### <sup>1</sup>H NMR Spectrum of 3g (Expansion)



## <sup>11</sup>B{<sup>1</sup>H} NMR Spectrum of 3g

---4.81

Parameter	Value
Data File Name	/ Users/ kathym/ Desktop/ Fids/ KMAb149_char_CDCl3/ 13/ fid
Origin	Bruker BioSpin GmbH
Owner	av400bb
Solvent	CDCl3
Temperature	300.0
Pulse Sequence	zgpg
Experiment	1D
Number of Scans	200
Receiver Gain	193
Relaxation Delay	0.2000
Pulse Width	10.0000
Acquisition Time	1.2845
Acquisition Date	2017-04-03T12:14:51
Spectrometer	128.38
Frequency	
Spectral Width	25510.2
Lowest Frequency	-12754.9
Nucleus	11B
Acquired Size	32768
Spectral Size	65536

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երով ուրդին է երաված համորներա մերընտրել ենի հնետությելների արդեպելությունը է են մանչել ենքան է եմ են եներըներա Դուսերբը կապոր ննդրունանել իրի տան պատաննանգությունը, տունի երեկը արտաց նանարդներին ենքան էներ ենքանին էր ննդրո

10 0 f1 (ppm) 90 80 70 60 50 40 -10 -20 -40 -50 30 20 -30 -60 -70 -80 -90

## <sup>31</sup>P{<sup>1</sup>H} NMR Spectrum of 3g

Parameter	Value
Data File Name	/ Users/ kathym/ Desktop/ Fids/
	KMAb149_char_CDCl3/ 11/ fid
Origin	Bruker BioSpin GmbH
Owner	av400bb
Solvent	CDCl3
Temperature	300.1
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	64
Receiver Gain	193
Relaxation Delay	2.0000
Pulse Width	8.0000
Acquisition Time	0.5112
Acquisition Date	2017-04-03T12:01:47
Spectrometer Frequency	161.97
Spectral Width	64102.6
Lowest Frequency	-40150.1
Nucleus	31P
Acquired Size	32768
Spectral Size	65536

-48.67

## 140 120 100 80 60 40 20 0 -10 -30 -50 -70 -90 -110 f1 (ppm)

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#### <sup>13</sup>C{<sup>1</sup>H} NMR Spectrum of 3g





## Characterisation of [Os(H)(CO)(PPh<sub>3</sub>){H<sub>2</sub>B(mt)<sub>2</sub>}] (3h)

#### Commentary

As for **3a**, two weak infrared absorptions may be assigned to the terminal  $v_{BH}$  (KBr: 2398 cm<sup>-1</sup>) and 3c-2e  $v_{BHOs}$  (2100 cm<sup>-1</sup>) groups. The anticipated  $v_{OsH}$  absorption appeared as a shoulder (1949 cm<sup>-1</sup>) on the more intense  $v_{CO}$  absorption (1907 cm<sup>-1</sup>). The <sup>1</sup>H NMR spectrum includes a broad (h.h.w. = 0.44 ppm) resonance centred at -7.02 ppm due to the B– H–Os interaction in addition to a doublet at -13.23 ppm (<sup>2</sup>J<sub>PH</sub> = 18.2 Hz) due to the terminal osmium hydride. <sup>1</sup>H NMR Spectrum of 3h



#### <sup>1</sup>H NMR Spectrum of 3h (Expansion)



## <sup>11</sup>B{<sup>1</sup>H} NMR Spectrum of 3h

Parameter	Value	i	
Data File Name	/ Users/ kathym/ Documents/ APhD2015/ Abragam/ KMAb132a_pure_CDCl3/ 15/ fid		
Origin	Bruker BioSpin GmbH		
Owner	av400bb		
Solvent	CDCl3		
Temperature	300.0		
Pulse Sequence	zgpg		
Experiment	1D		
Number of Scans	200		
Receiver Gain	193		
Relaxation Delay	0.2000		
Pulse Width	10.0000		
Acquisition Time	1.2845		
Acquisition Date	2016-12-06T05:36:15		
Spectrometer Frequency	128.38		
Spectral Width	25510.2		
Lowest Frequency	-12754.9		
Nucleus	11B		
Acquired Size	32768		
Spectral Size	65536		
		11	
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#### 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 f1 (ppm)

### <sup>31</sup>P{<sup>1</sup>H} NMR Spectrum of 3h

Parameter	Value		10					
Data File Name	/ Users/ kathym/ Documents/ APhD2015/ Abragam/ KMAb132a_pure_pentanewash_CDCl3/ 11/ fid		$\checkmark$					
Origin	Bruker BioSpin GmbH							
Owner	av400bb							
Solvent	CDCl3							
Temperature	300.0							
Pulse Sequence	zgpg30							
Experiment	1D							
Number of Scans	128							
Receiver Gain	193							
Relaxation Delay	2.0000							
Pulse Width	8.0000							
Acquisition Time	0.5112							
Acquisition Date	2016-12-05T18:25:48							
Spectrometer Frequency	161.97							
Spectral Width	64102.6							
Lowest Frequency	-40044.7							
Nucleus	31P							
Acquired Size	32768							
Spectral Size	65536							
*********				****		****		 
10 100	100 00 00	40	20	0 10	20	50	70	 

#### <sup>13</sup>C{<sup>1</sup>H} NMR Spectrum of 3h





