

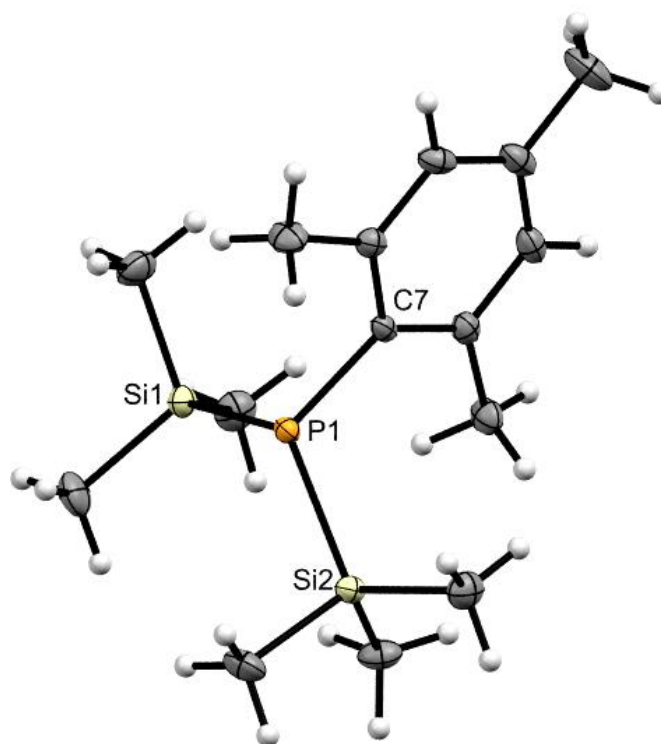
# Phosphacycloalkyldiones: Synthesis and coordinative behaviour of 6- and 7-member cyclic diketophosphanyls

Kyle G. Pearce, Vladimir Simenok and Ian R. Crossley\*

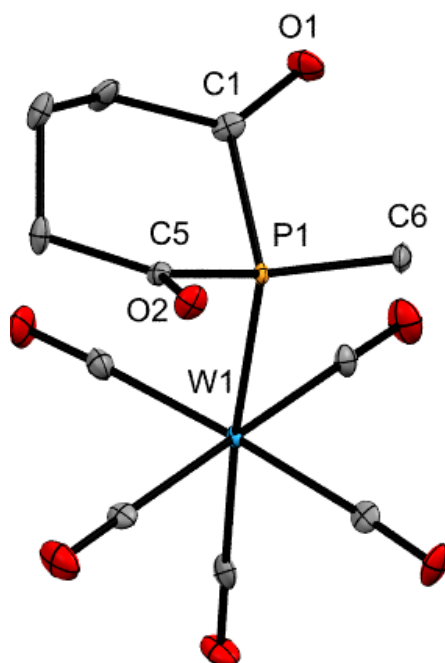
*Department of Chemistry, University of Sussex, Falmer, Brighton, UK*

## Electronic Supplementary Information

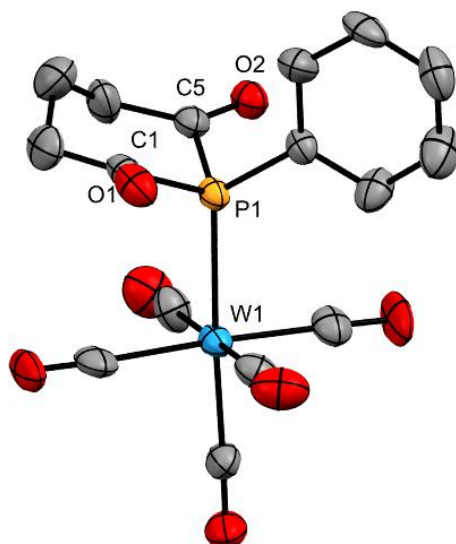
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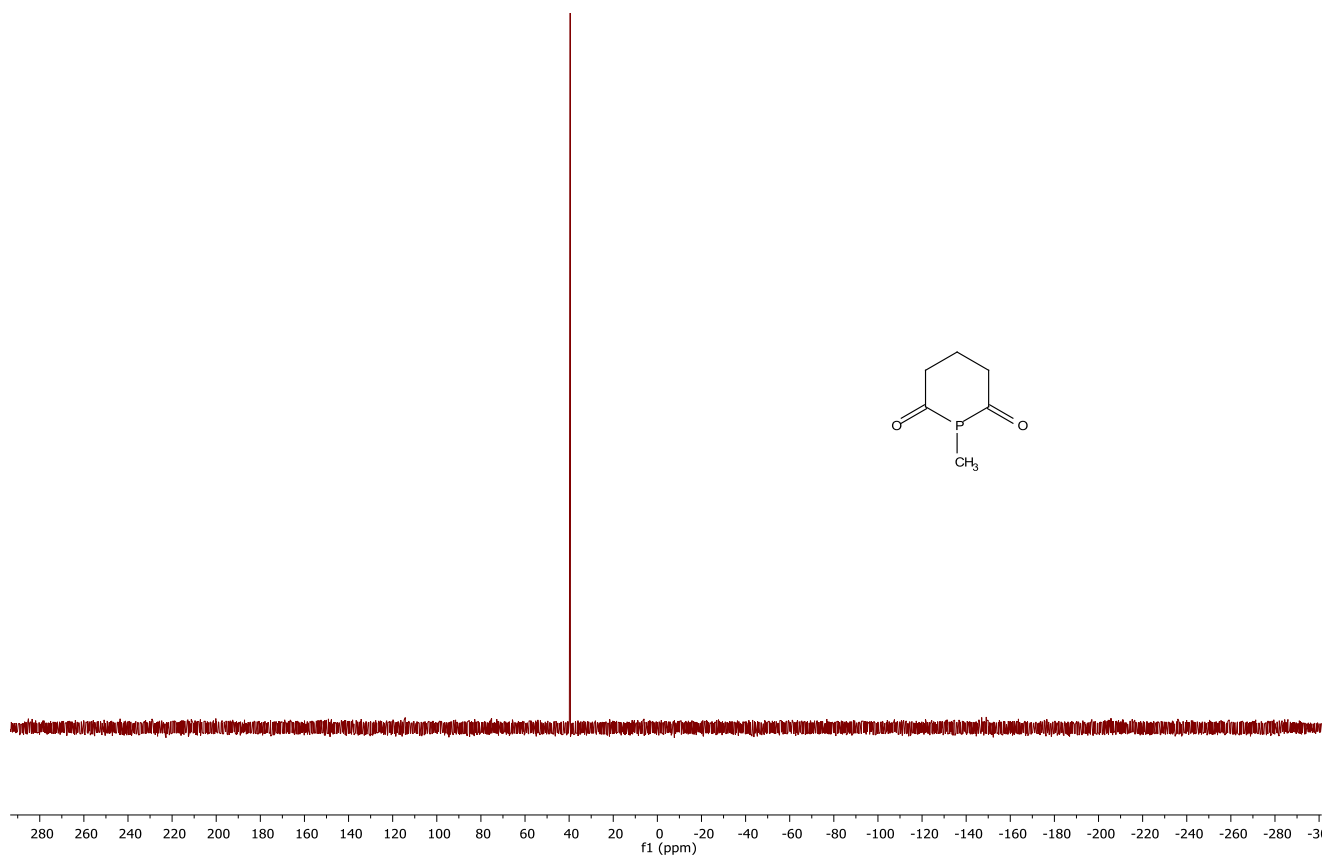
**Figure S1.** Molecular structure of  $\text{MesP}(\text{SiMe}_3)_2$  with displacement ellipsoids at 50 %. The asymmetric unit contains two independent molecules of comparable geometry. Selected geometric parameters ( $\text{\AA}$ , deg.): P1–Si1 2.2465(5), P1–Si2 2.2529(5), P1–C7 1.8505(13), Si–P–Si 111.04(2), Si1–P1–C7 103.07(5), Si2–P1–C7 114.58(4)



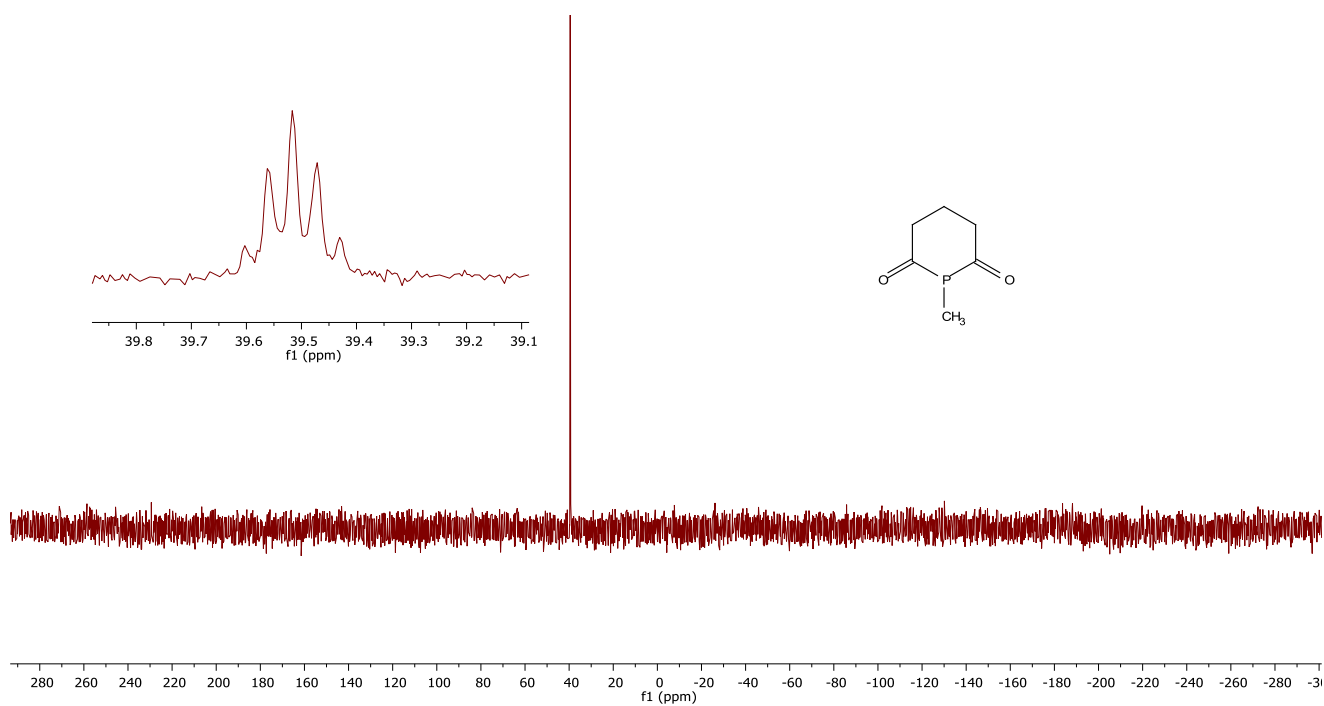
**Figure S2.** Molecular structure of **5a** with displacement ellipsoids at 50 % and hydrogen atoms omitted for clarity. Selected geometric parameters ( $\text{\AA}$ ): W1–P1 2.5065(7), W1–C<sub>trans</sub> 1.998(3), W1–C<sub>cis</sub> 2.045(3)–2.064(4), C–O<sub>trans</sub> 1.145(4), C–O<sub>cis</sub> 1.133(5)–1.143(4), P1–C6 1.815(3), P1–C5 1.881(4), P1–C1 1.880(3), C1–O1 1.202(4) C5–O2 1.206(4).



**Figure S3.** Molecular structure of **5d** with displacement ellipsoids at 50 % and hydrogen atoms omitted for clarity. The asymmetric unit includes a second molecule that is subject to disorder. Selected geometric parameters (Å): W1–P1 2.490(3), W1–C<sub>trans</sub> 2.009(11), W1–C<sub>cis</sub> 2.02(1) – 2.05(1), C–O<sub>trans</sub> 1.14 (2), C–O<sub>cis</sub> 1.13(1) – 1.17(2), P1–C6 1.818(10), P1–C5 1.889(12), P1–C1 1.882(10), C1–O1 1.212(13) C5–O2 1.205(10).



**Figure S4.**  $^{31}\text{P}\{^1\text{H}\}$  NMR Spectrum ( $\text{CDCl}_3$ , 303 K, 161.72 MHz) for compound **2a**.



**Figure S5.**  $^{31}\text{P}$  NMR Spectrum ( $\text{CDCl}_3$ , 303 K, 161.72 MHz) for compound **2a**.

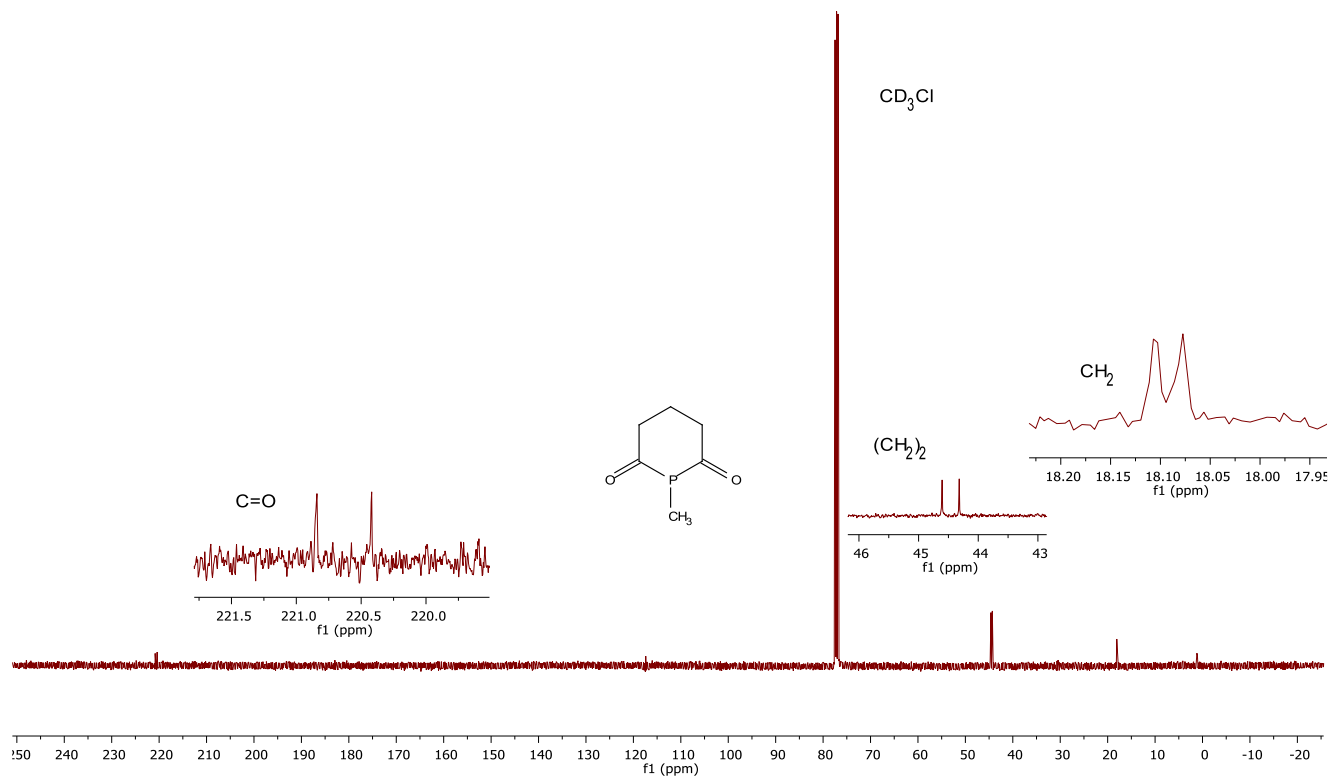


Figure S6.  $^{13}\text{C}\{^1\text{H}\}$  NMR Spectrum ( $\text{CDCl}_3$ , 303 K, 100.46 MHz) for compound **2a**.

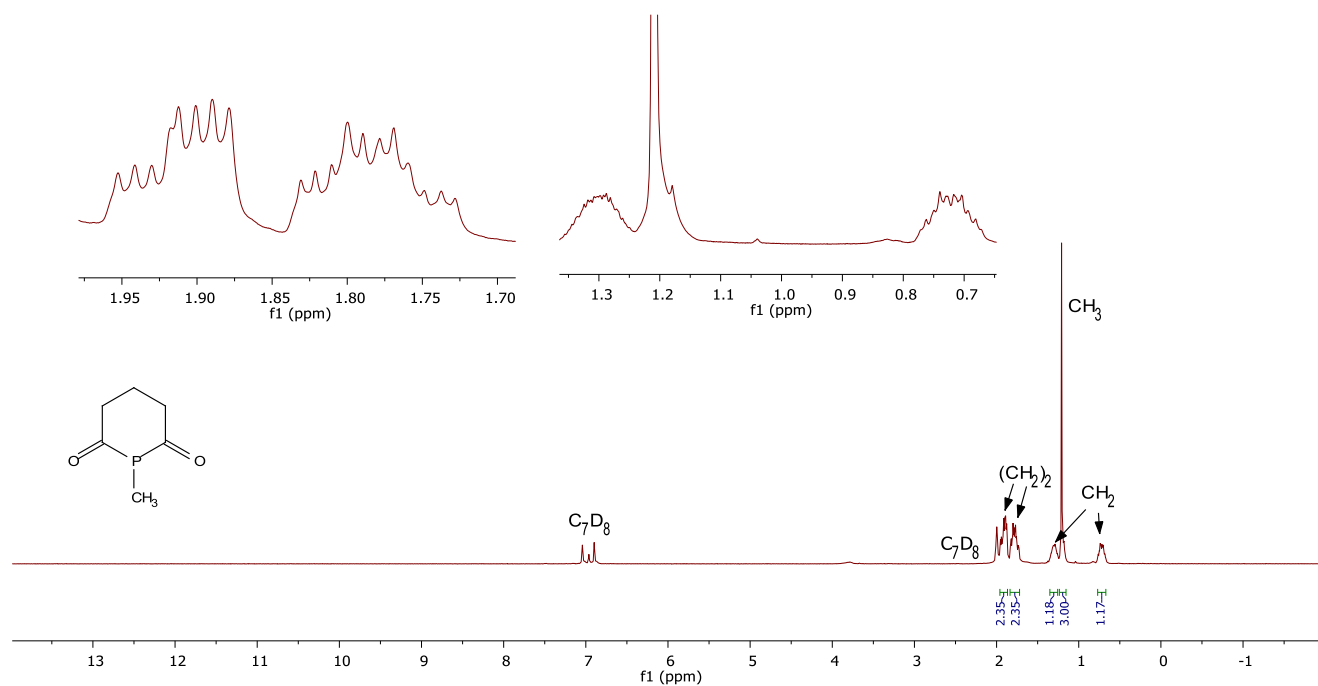
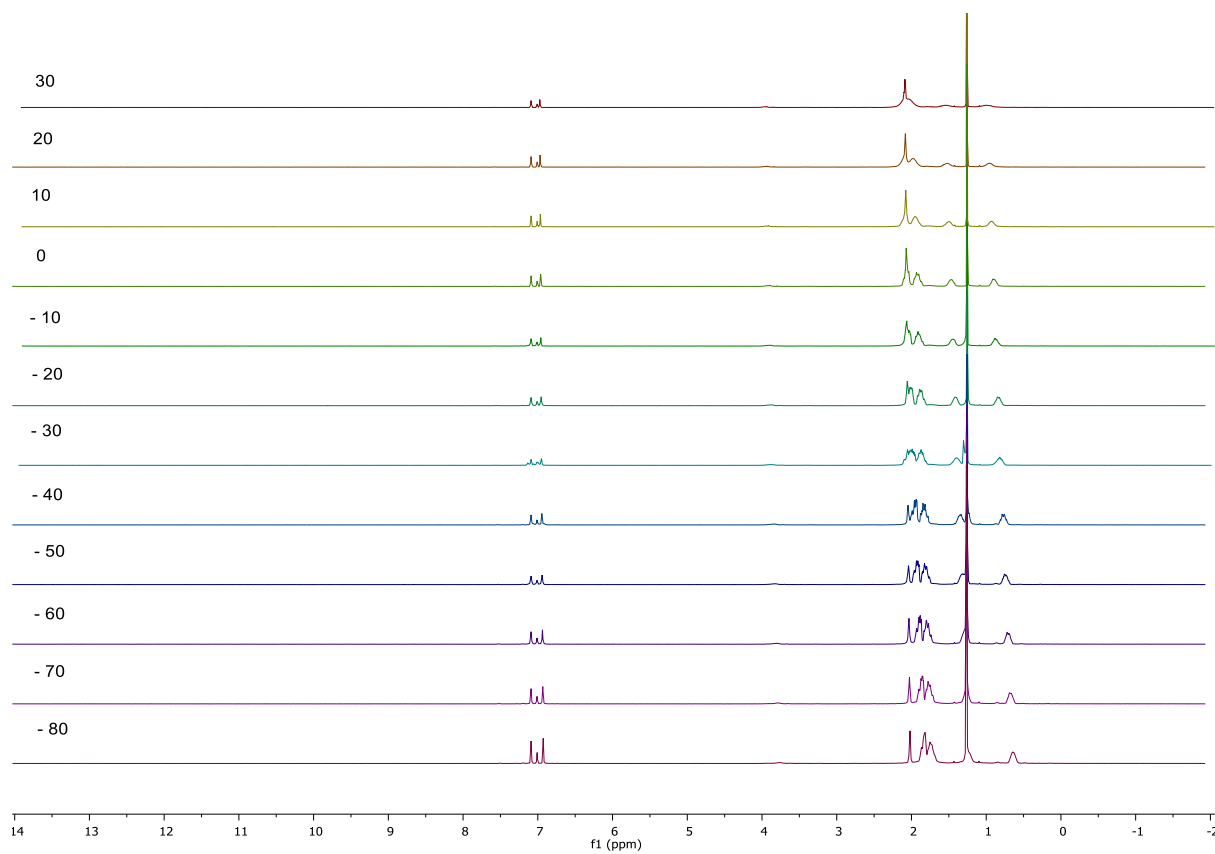
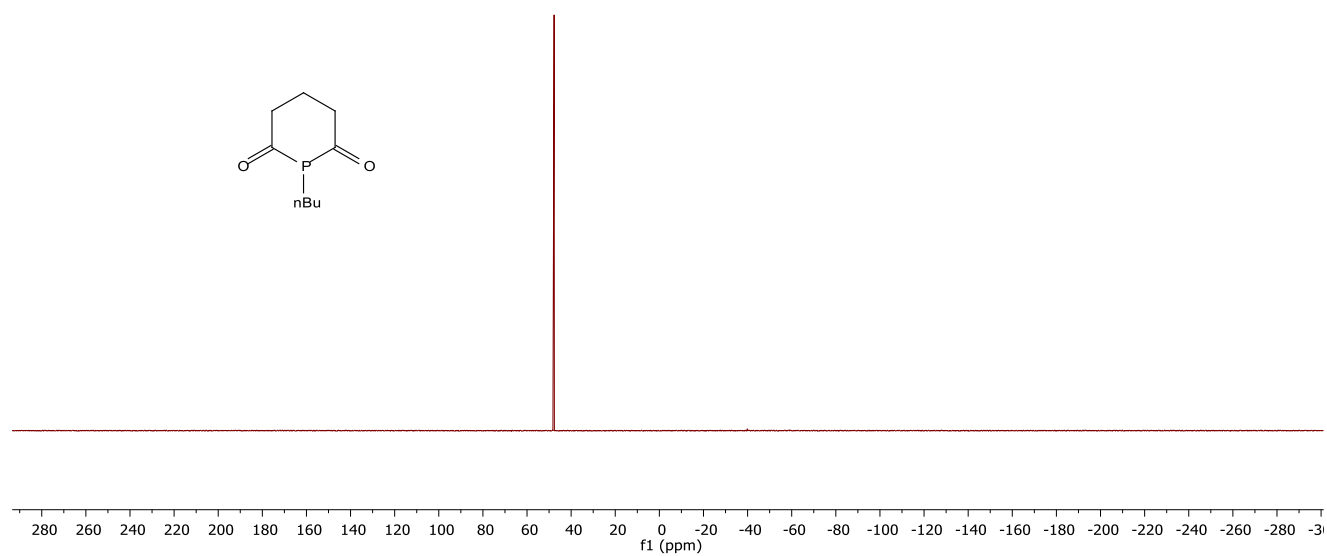


Figure S7.  $^1\text{H}$  NMR Spectrum ( $\text{C}_7\text{D}_8$ , 238 K, 399.49 MHz) for compound **2a**.



**Figure S8.** Stacked Variable Temperature  $^1\text{H}$  NMR Spectra ( $\text{C}_7\text{D}_8$ , 303 K – 193 K, 399.49 MHz) for compound **2a**.



**Figure S9.**  $^{31}\text{P}\{^1\text{H}\}$  NMR Spectrum ( $\text{C}_6\text{D}_6$ , 303 K, 161.72 MHz) for compound **2b**.

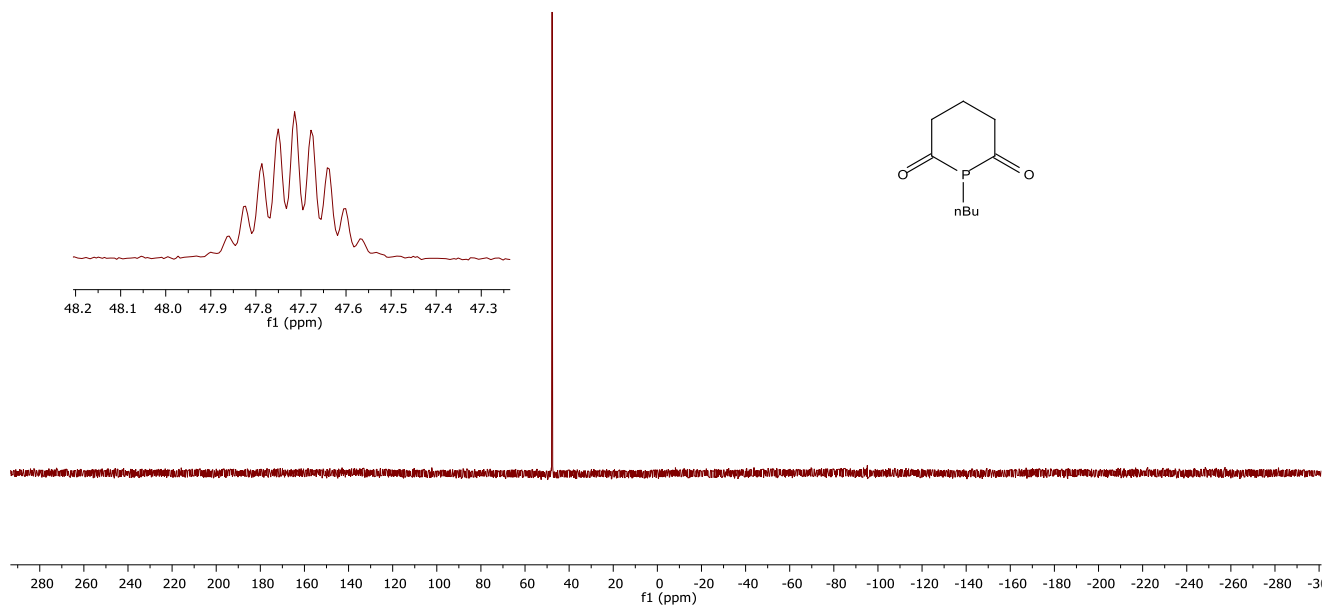


Figure S10.  $^{31}\text{P}$  NMR Spectrum ( $\text{C}_6\text{D}_6$ , 303 K, 161.72 MHz) for compound **2b**.

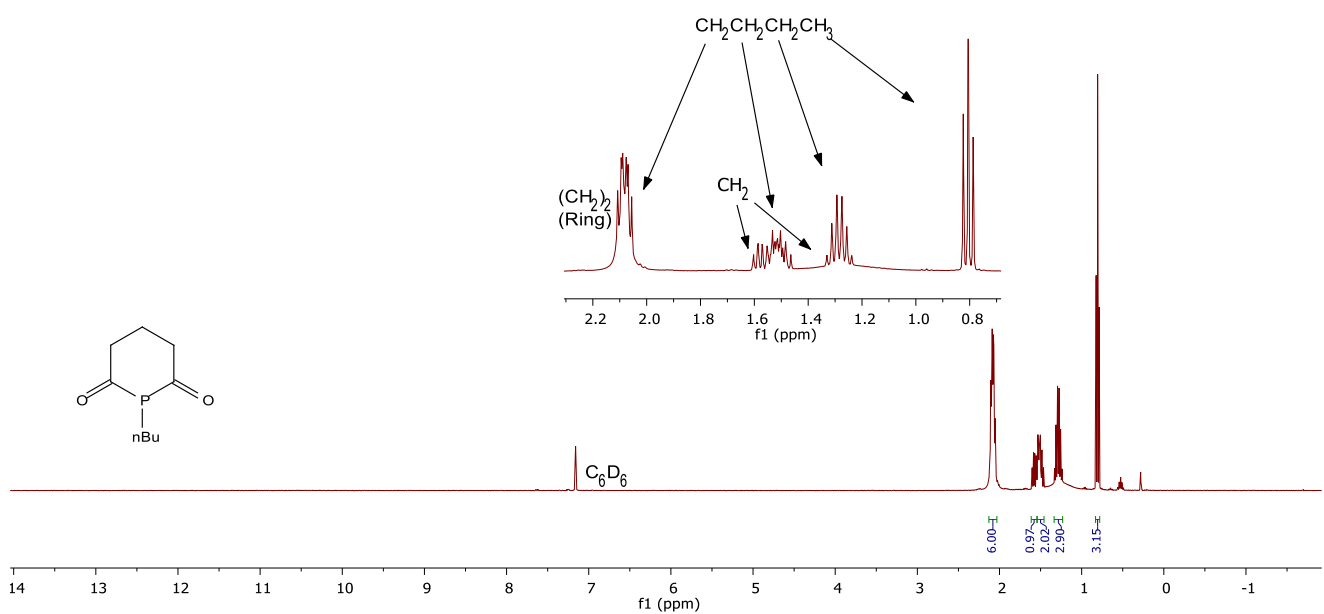
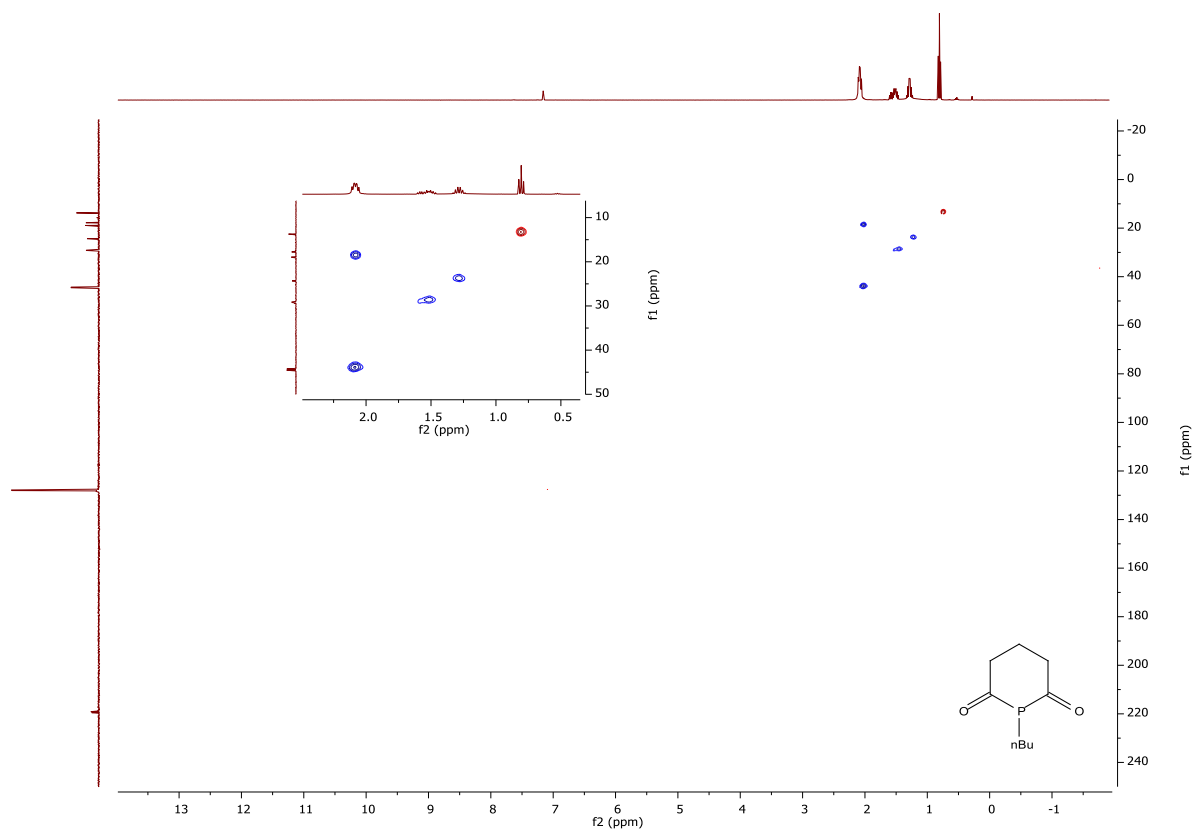
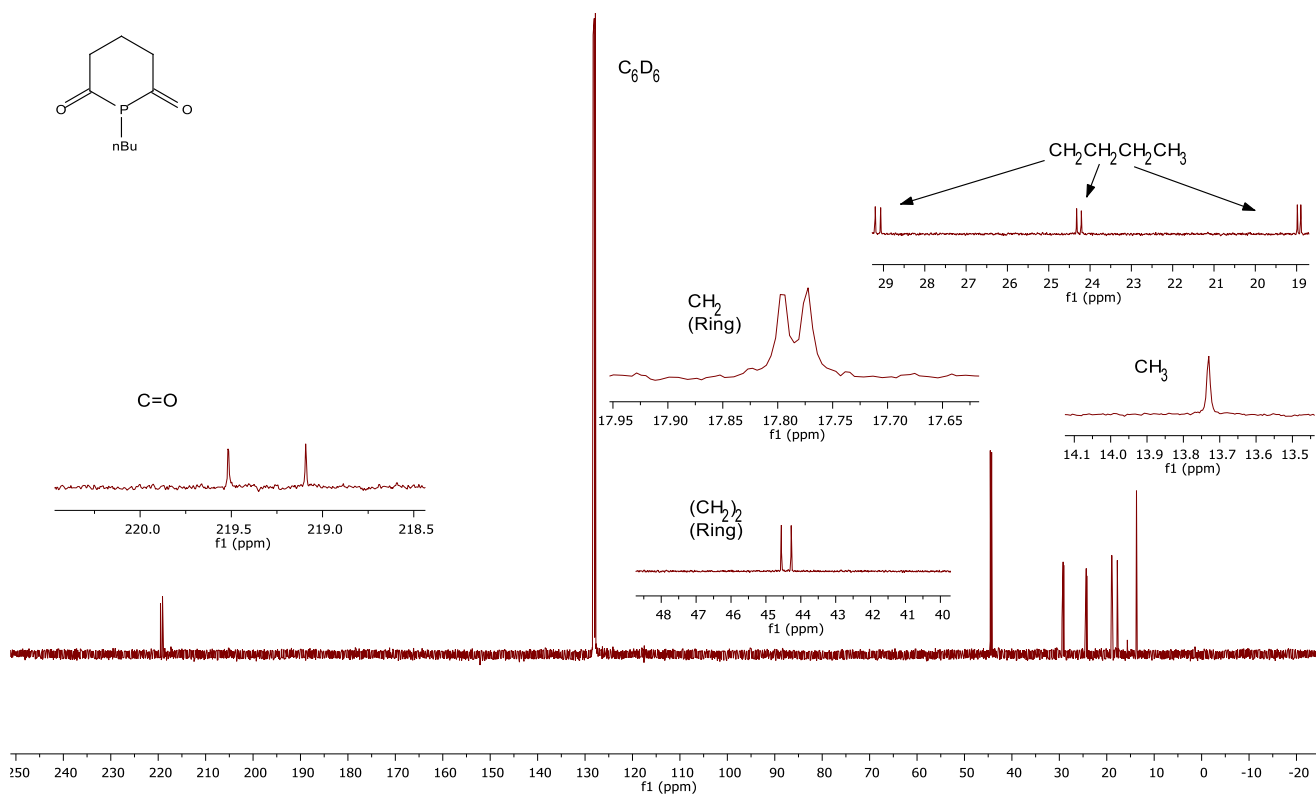
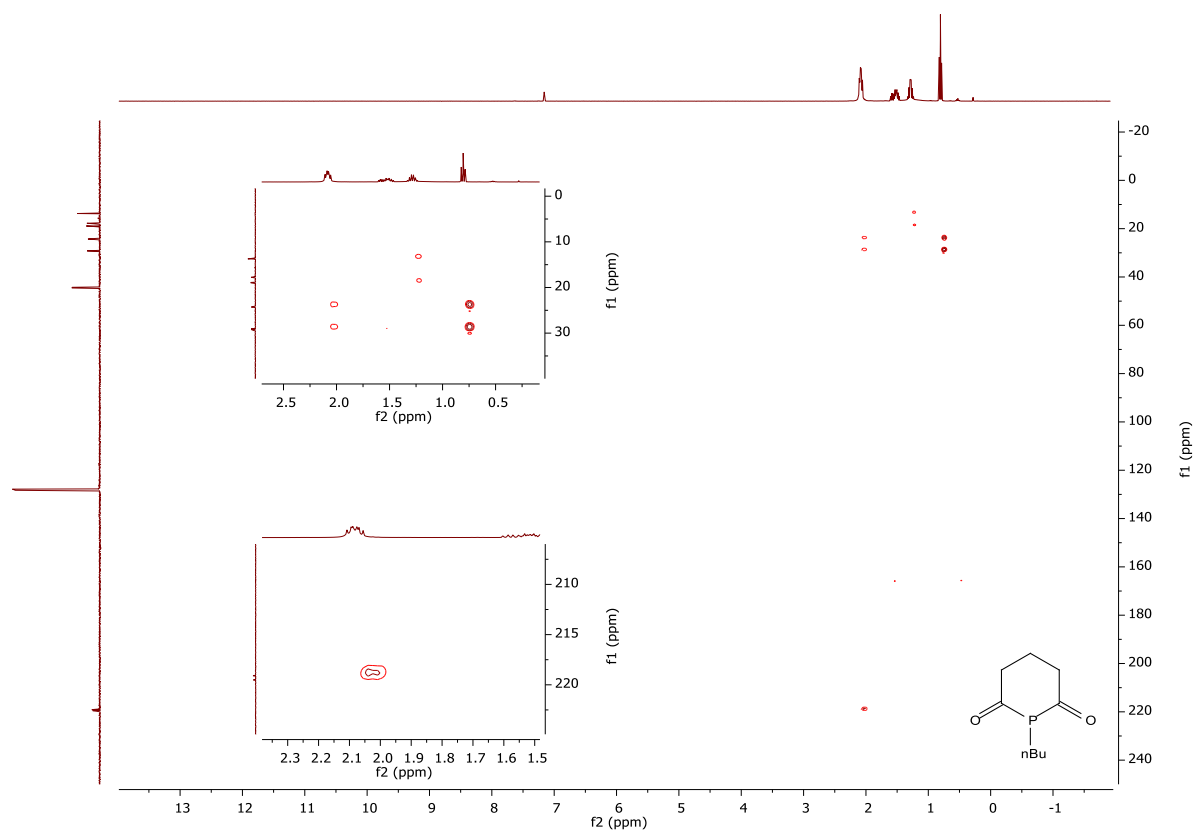


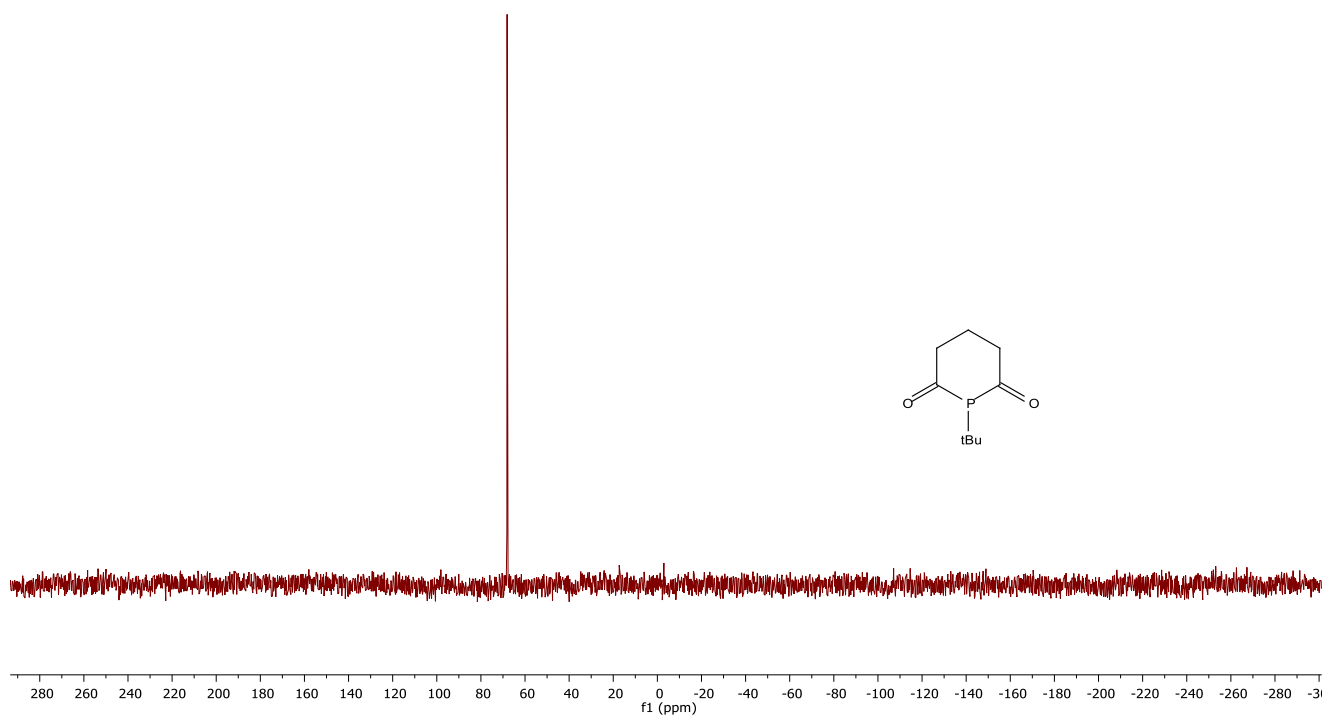
Figure S11.  $^1\text{H}$  NMR Spectrum ( $\text{C}_6\text{D}_6$ , 303 K, 399.49 MHz) for compound **2b**.



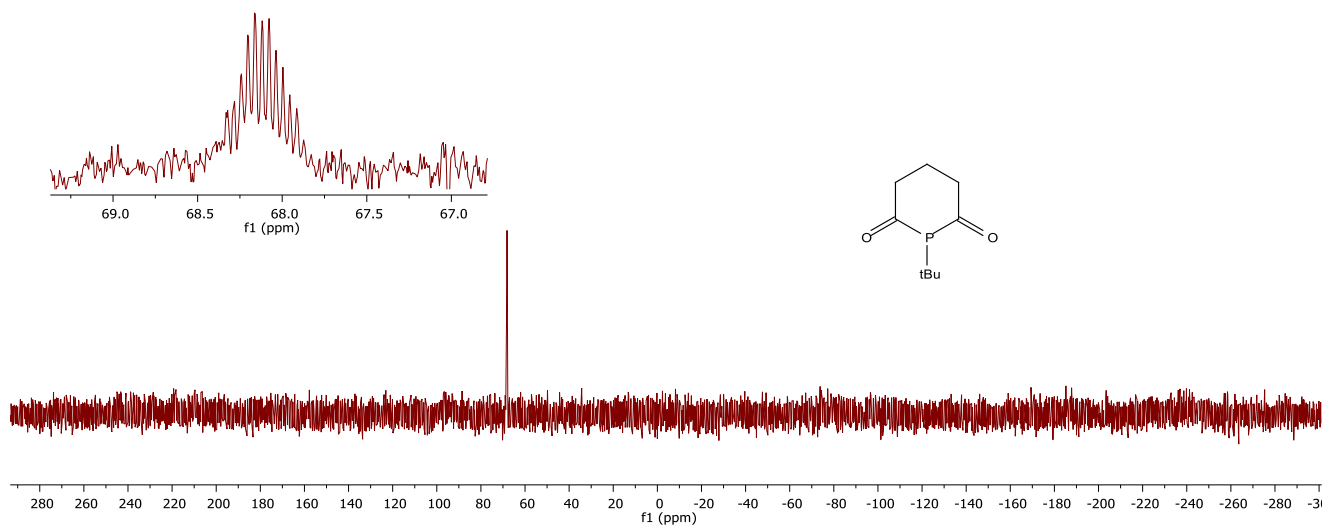




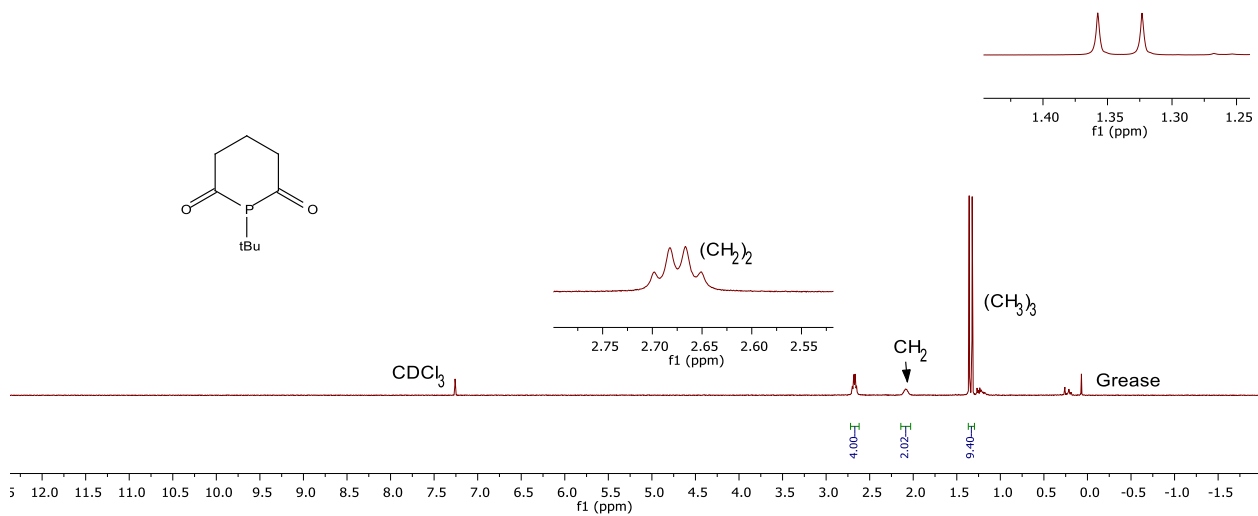
**Figure S14.**  $^1\text{H}$ - $^{13}\text{C}$  HMBC trace ( $\text{C}_6\text{D}_6$ , 303 K, 399.49, 100.46 MHz) for Compound **2b**.



**Figure S15.**  $^{31}\text{P}\{^1\text{H}\}$  NMR Spectrum ( $\text{C}_6\text{D}_6$ , 303 K, 161.72 MHz) for compound **2c**.



**Figure S16.**  $^{31}\text{P}$  NMR Spectrum ( $\text{C}_6\text{D}_6\text{O}$ , 303 K, 161.72 MHz) for compound **2c**.



**Figure S17.**  $^1\text{H}$  NMR Spectrum ( $\text{CD}_3\text{Cl}$ , 303 K, 399.49 MHz) for compound **2c**.

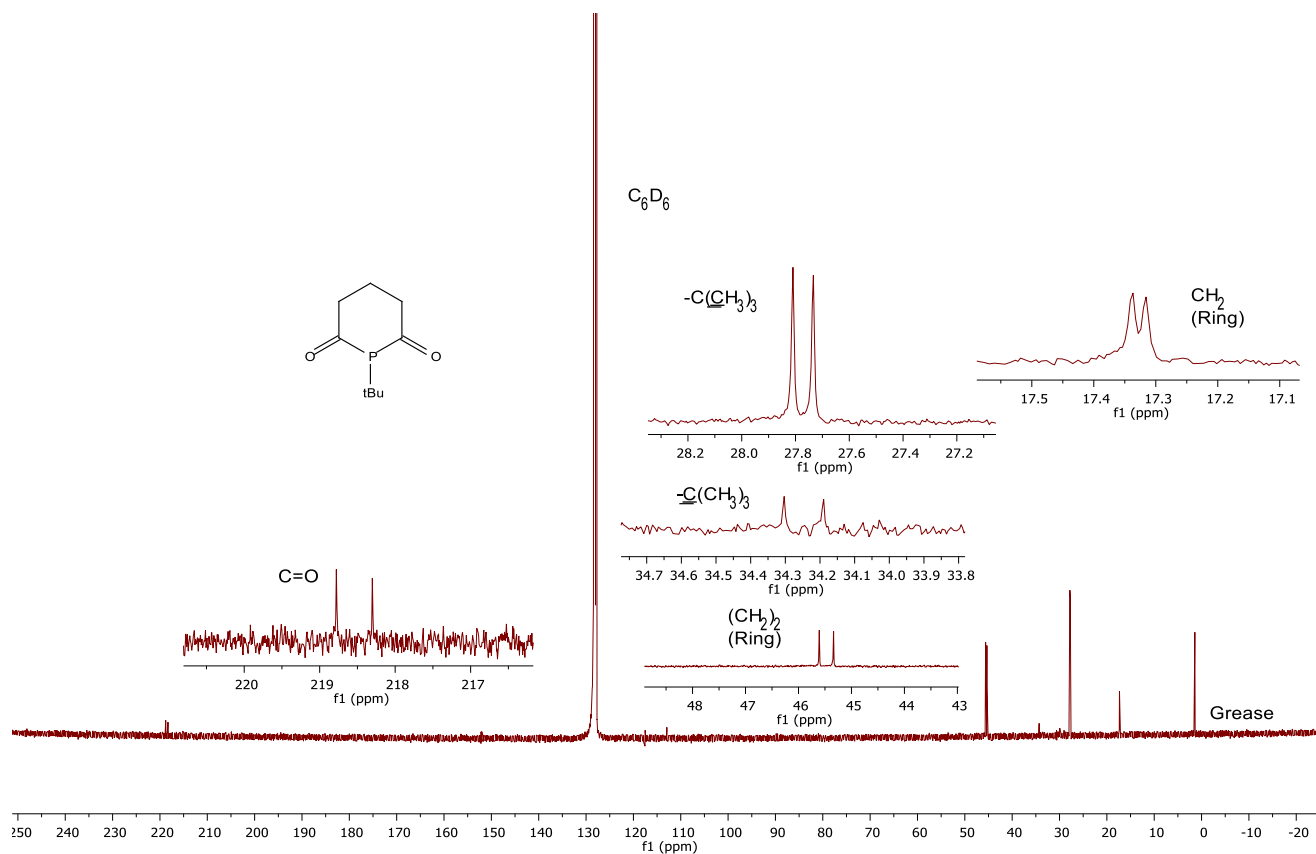


Figure S18.  $^{13}C\{^1H\}$  NMR Spectrum ( $C_6D_6$ , 303 K, 100.46 MHz) for compound **2c**.

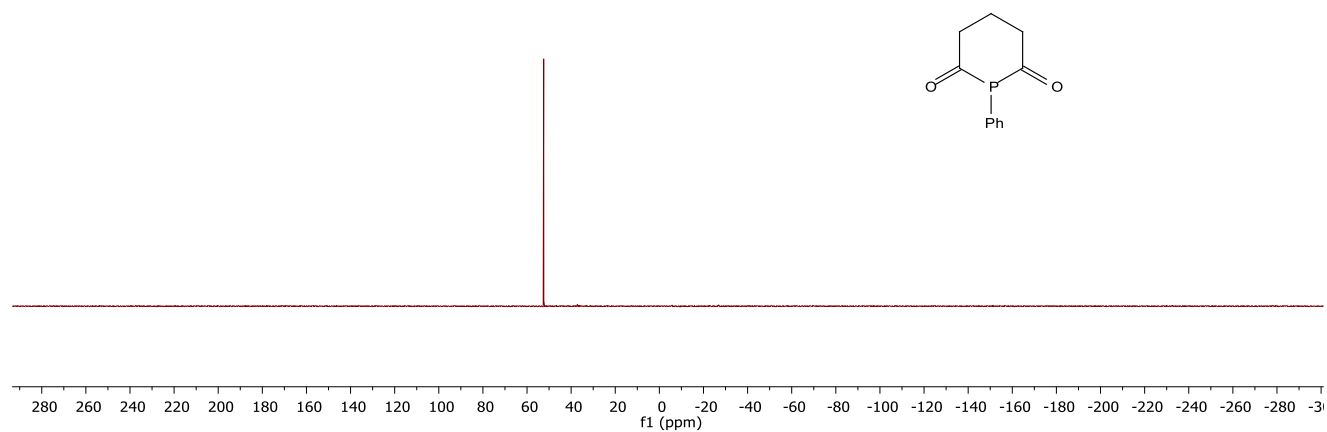


Figure S19.  $^{31}P\{^1H\}$  NMR Spectrum ( $CD_2Cl_2$ , 303 K, 161.72 MHz) for compound **2d**.

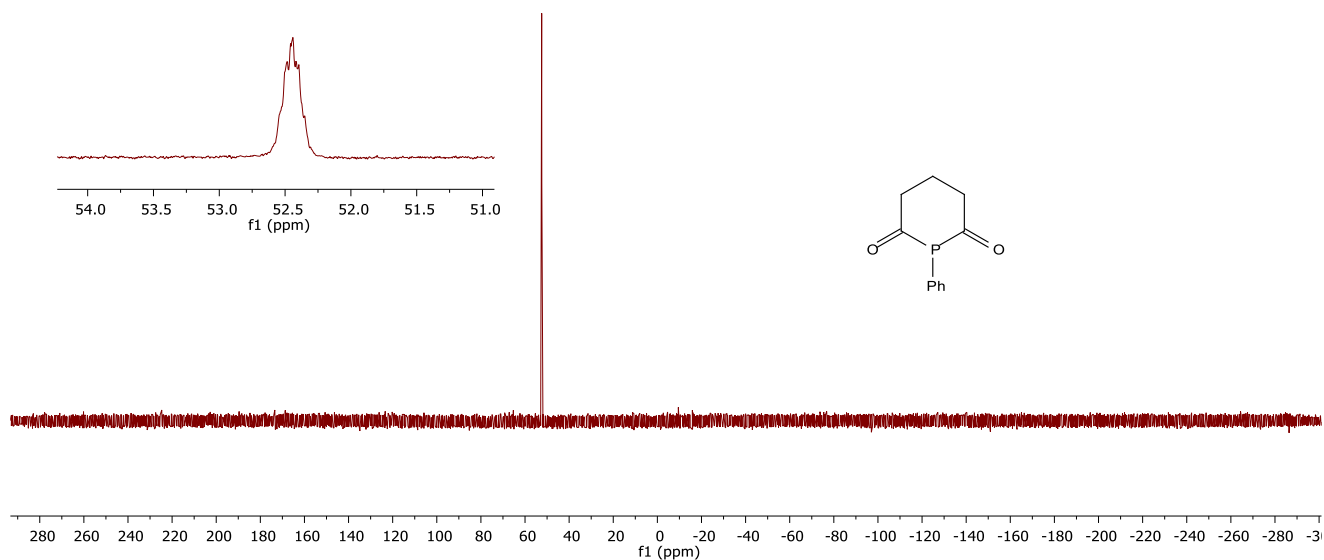


Figure S20.  $^{31}\text{P}$  NMR Spectrum ( $\text{CD}_2\text{Cl}_2$ , 303 K, 161.72 MHz) for compound 2d.

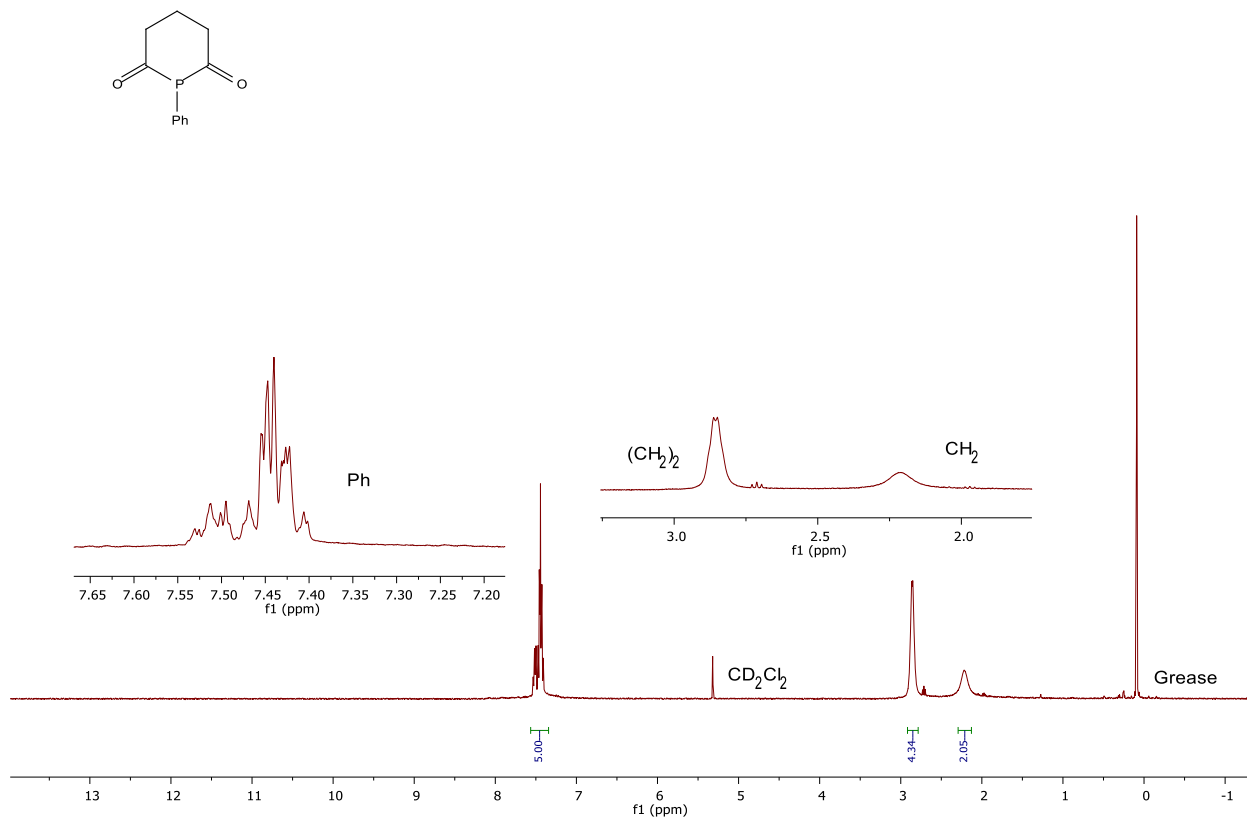
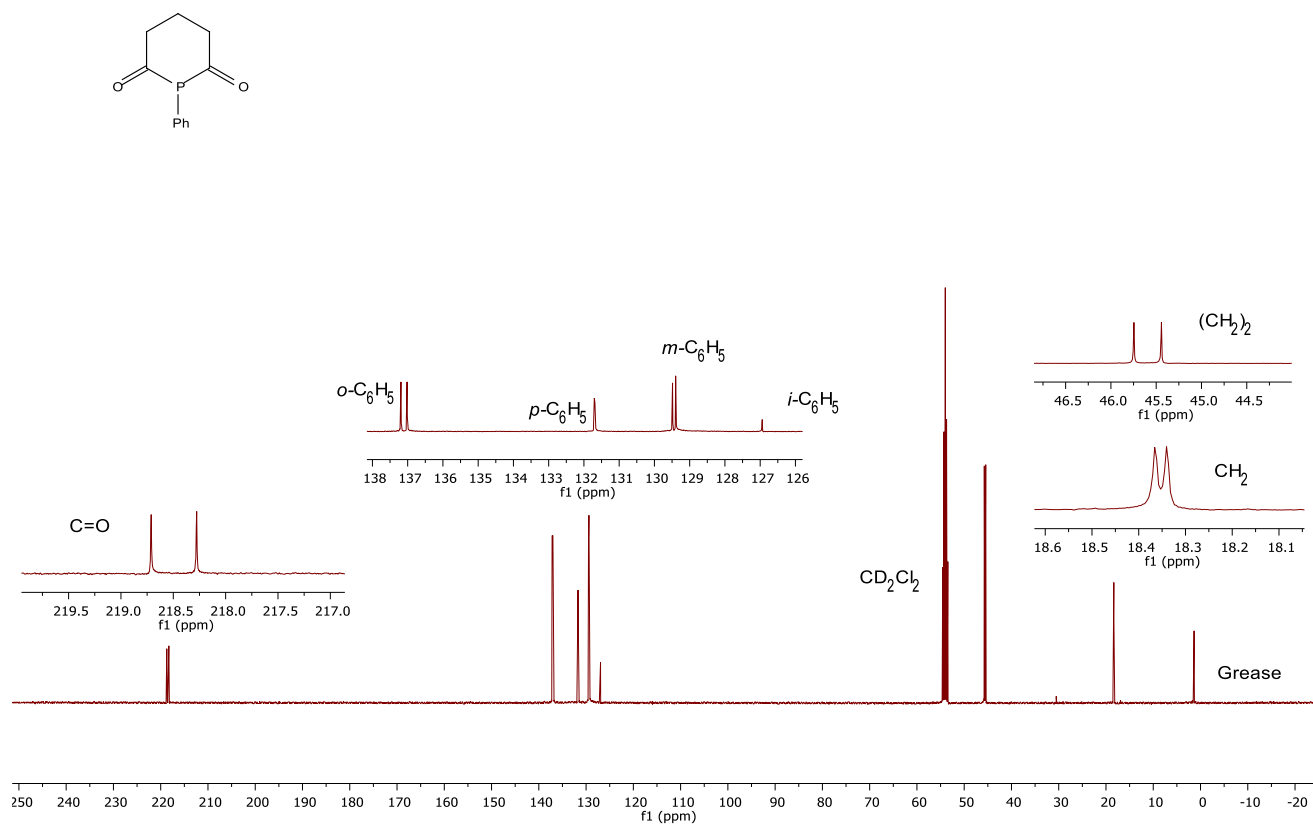
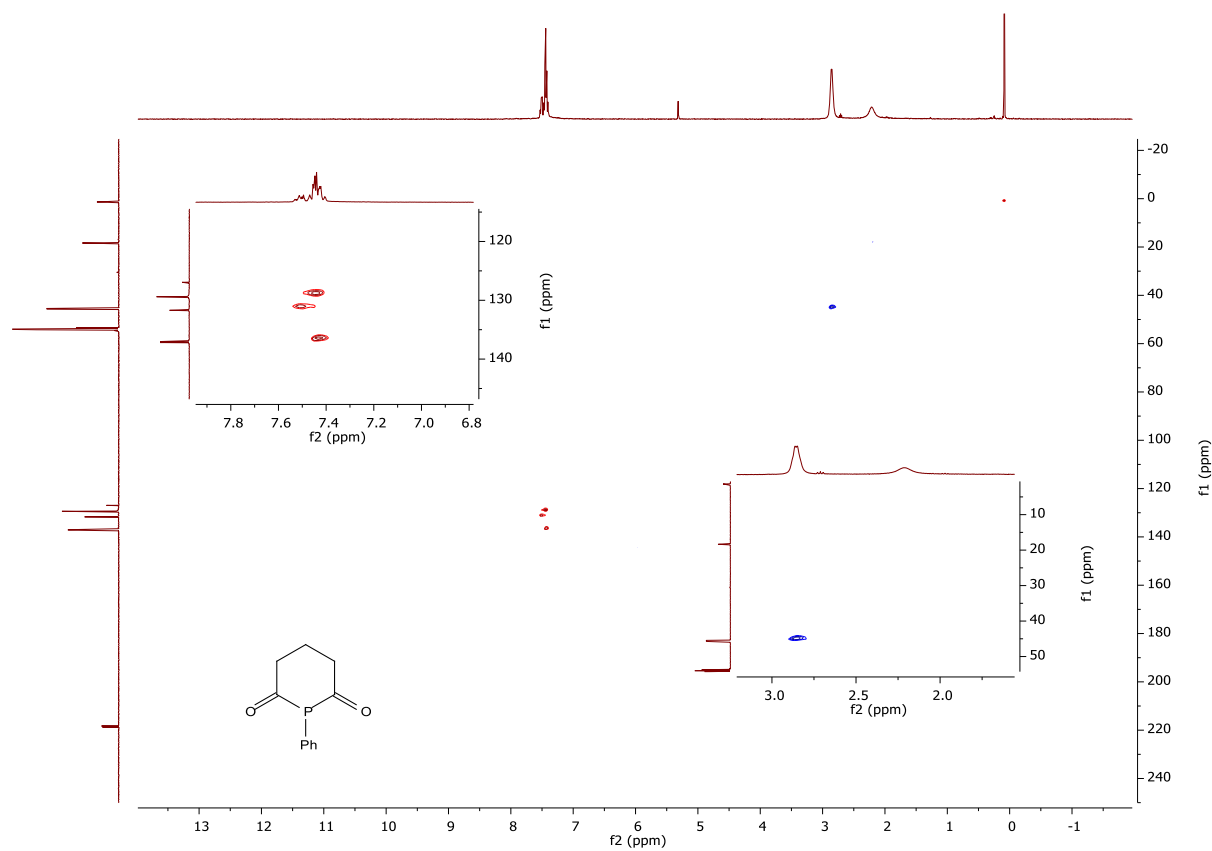


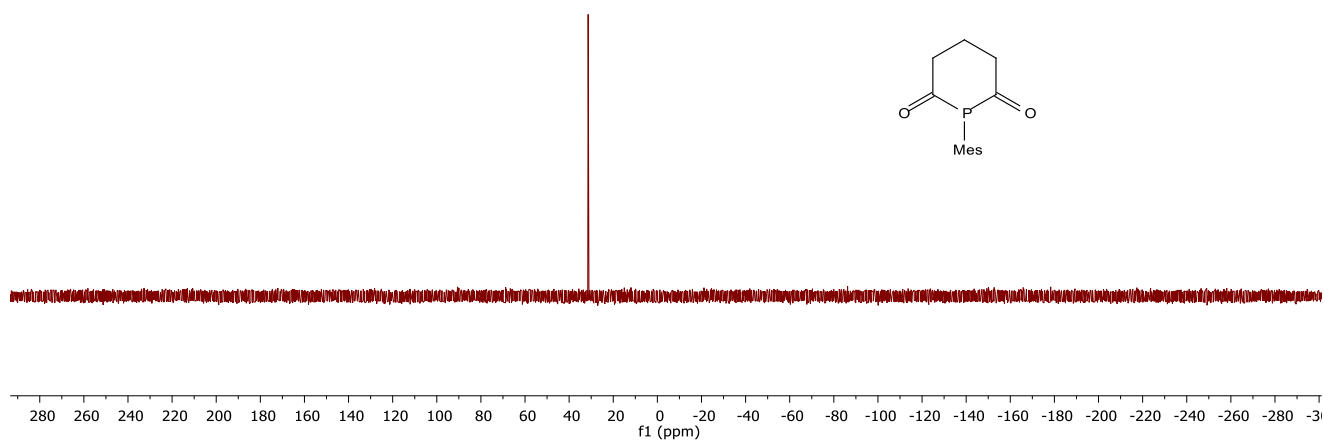
Figure S21.  $^1\text{H}$  NMR Spectrum ( $\text{CD}_2\text{Cl}_2$ , 303 K, 399.49 MHz) for compound 2d.



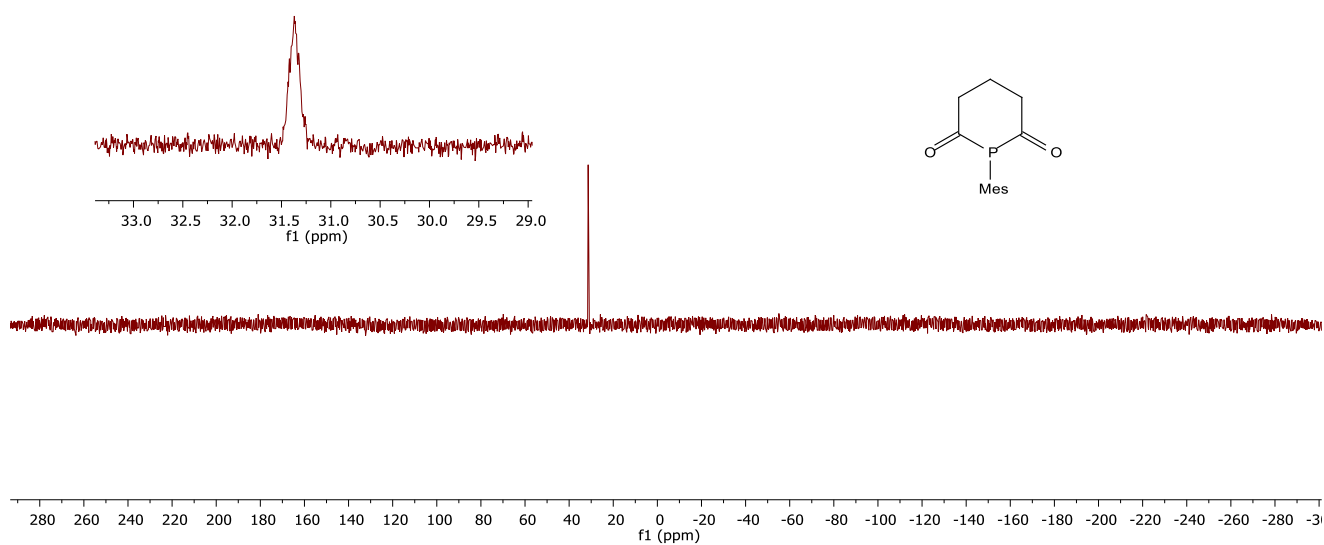
**Figure S22.**  $^{13}\text{C}\{^1\text{H}\}$  NMR Spectrum ( $\text{CD}_2\text{Cl}_2$ , 303 K, 100.46 MHz) for compound **2d**.



**Figure S23.**  $^1\text{H}\text{-}^{13}\text{C}$  HSQC trace ( $\text{CD}_2\text{Cl}_2$ , 303 K, 399.49, 100.46 MHz) for Compound **2d**.



**Figure S24.**  $^{31}\text{P}\{^1\text{H}\}$  NMR Spectrum ( $\text{C}_6\text{D}_6$ , 303 K, 161.72 MHz) for compound **2e**.



**Figure S25.**  $^{31}\text{P}$  NMR Spectrum ( $\text{C}_6\text{D}_6$ , 303 K, 161.72 MHz) for compound **2e**.

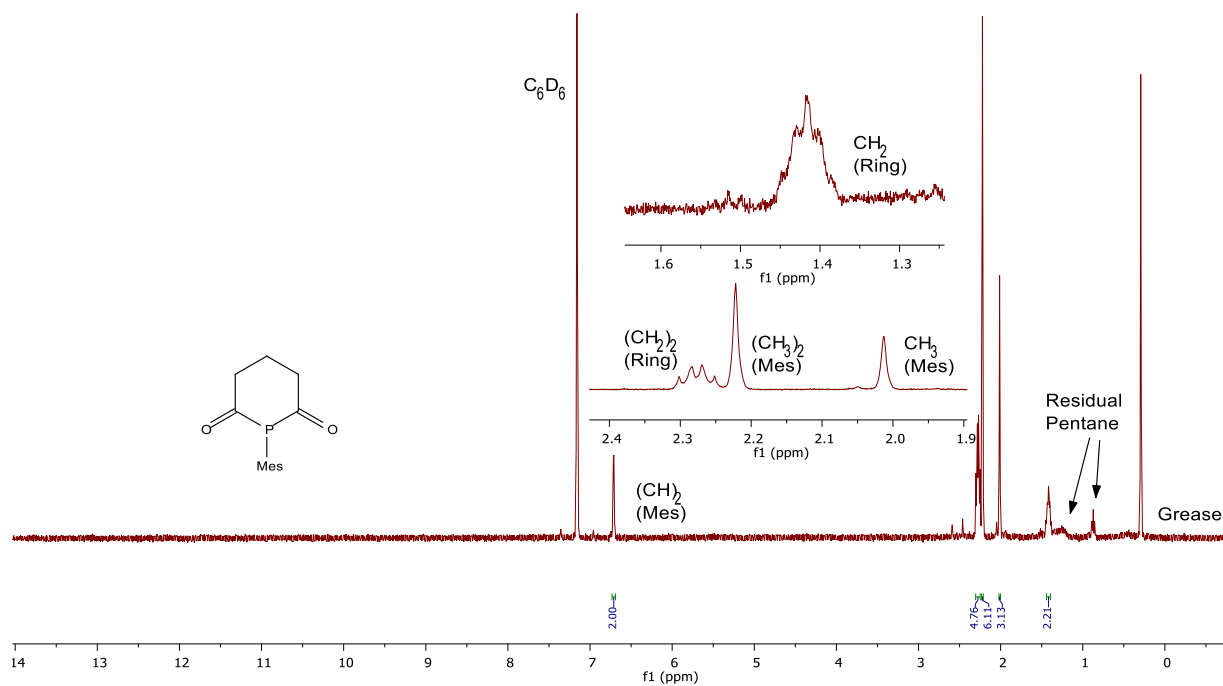


Figure S26.  $^1\text{H}$  NMR Spectrum ( $\text{C}_6\text{D}_6$ , 303 K, 399.49 MHz) for compound 2e.

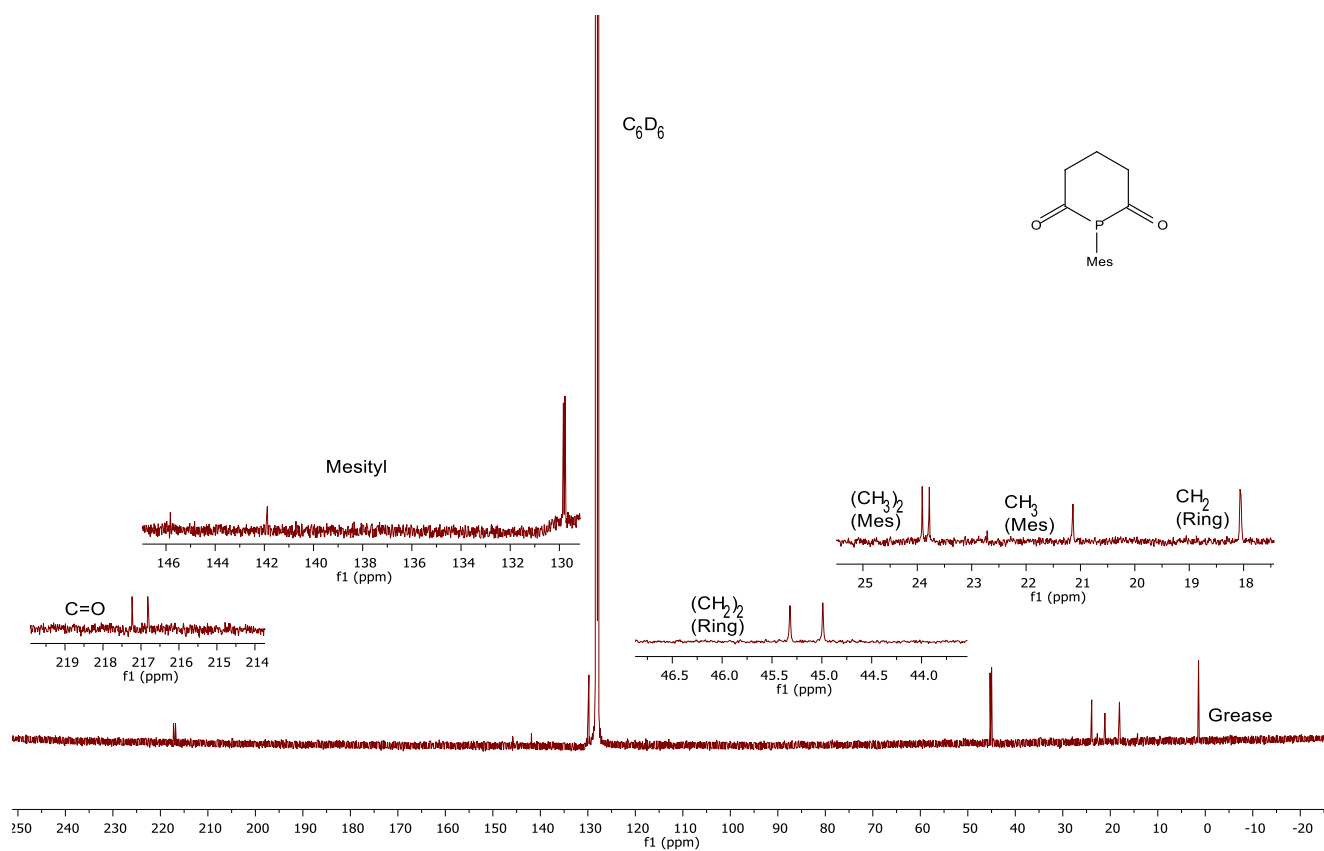
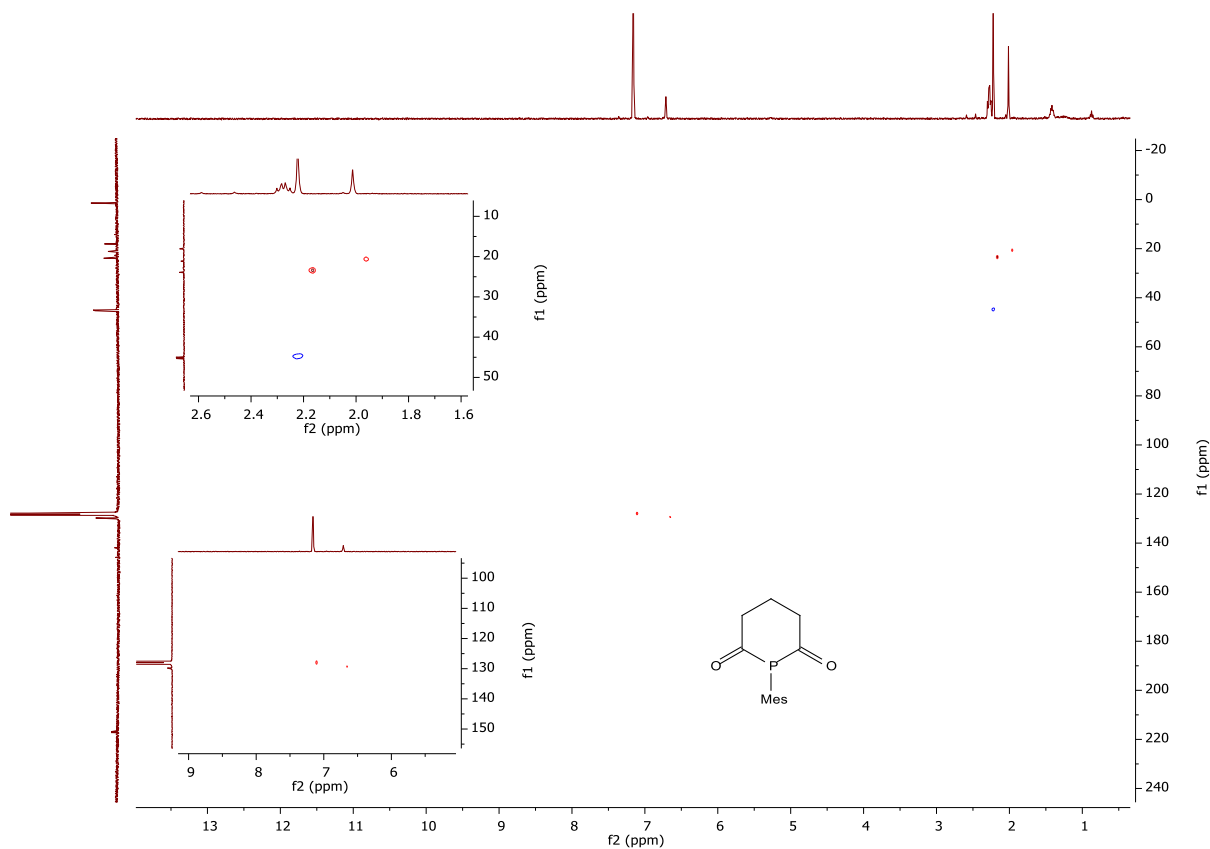
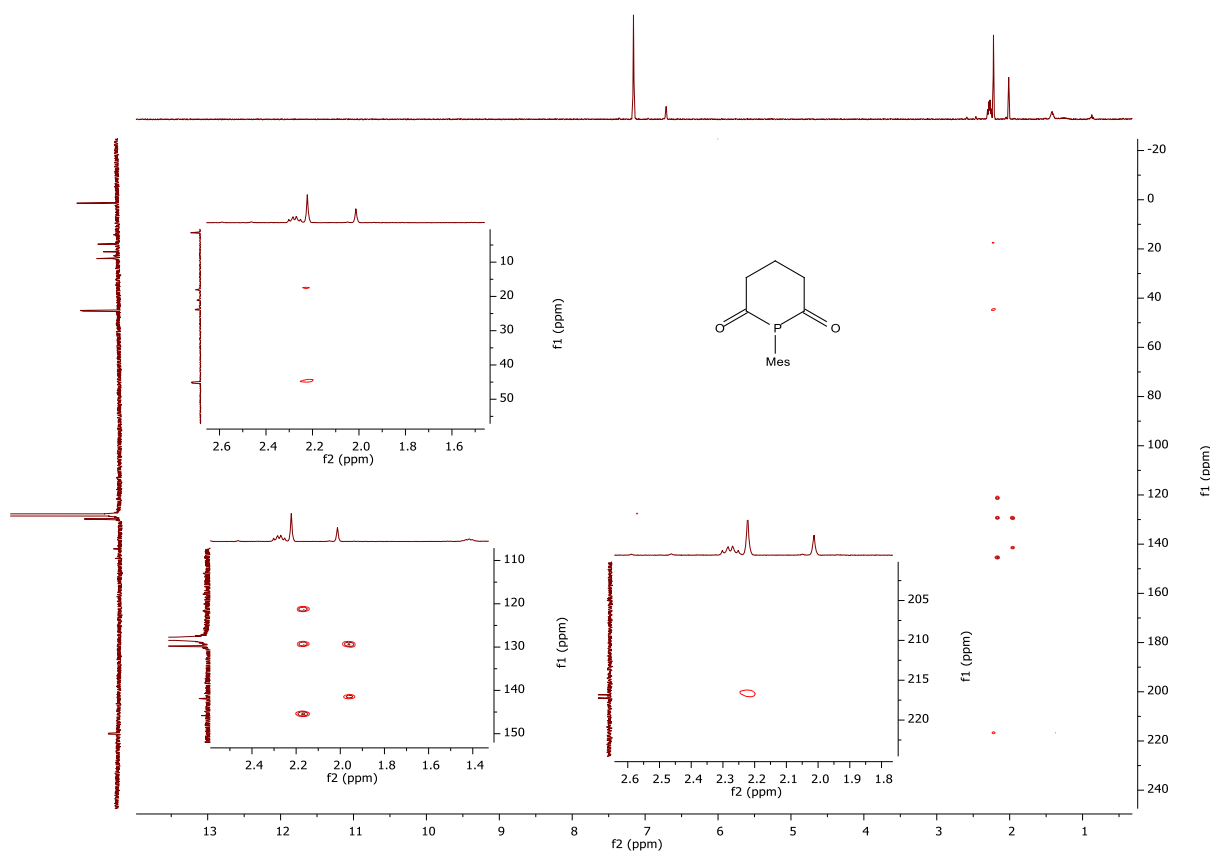


Figure S27.  $^{13}\text{C}\{^1\text{H}\}$  NMR Spectrum ( $\text{C}_6\text{D}_6$ , 303 K, 100.46 MHz) for compound 2e.

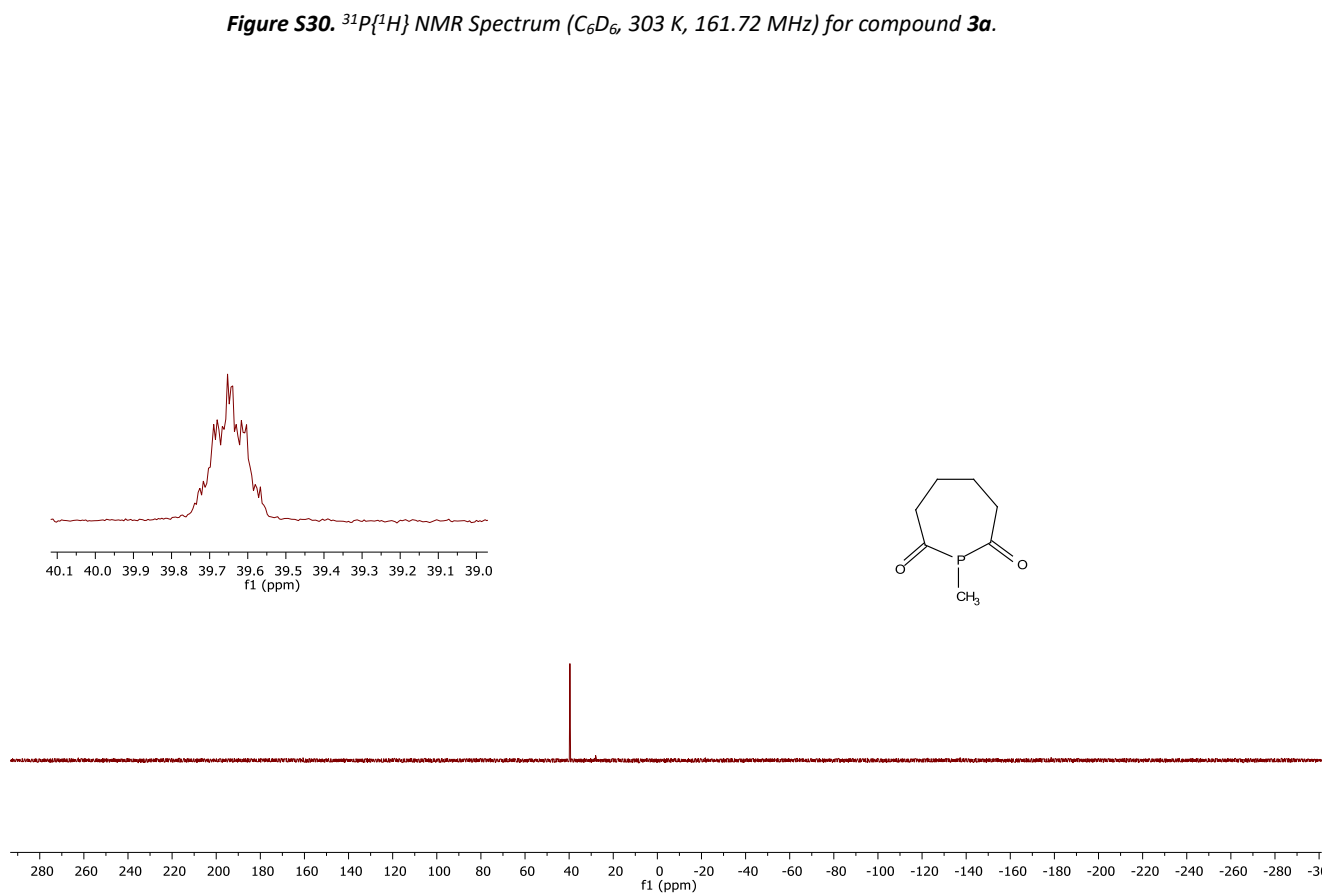
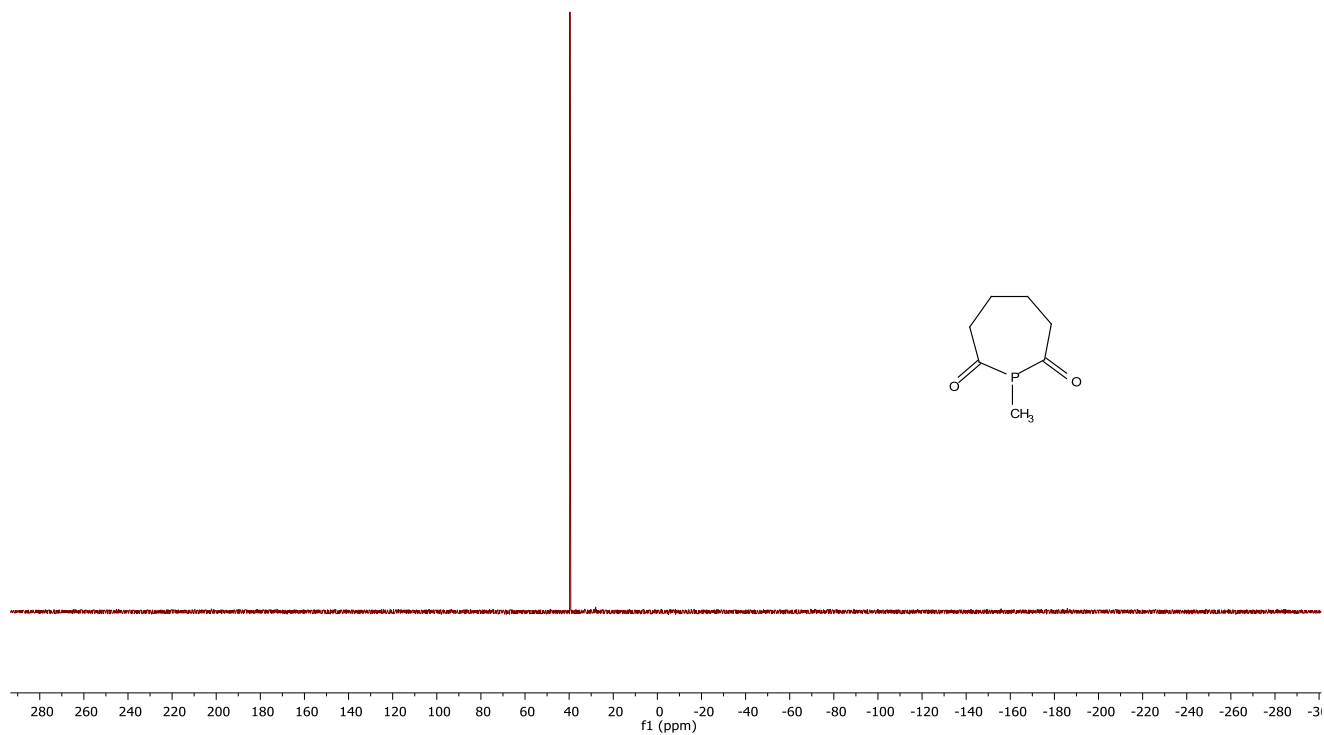


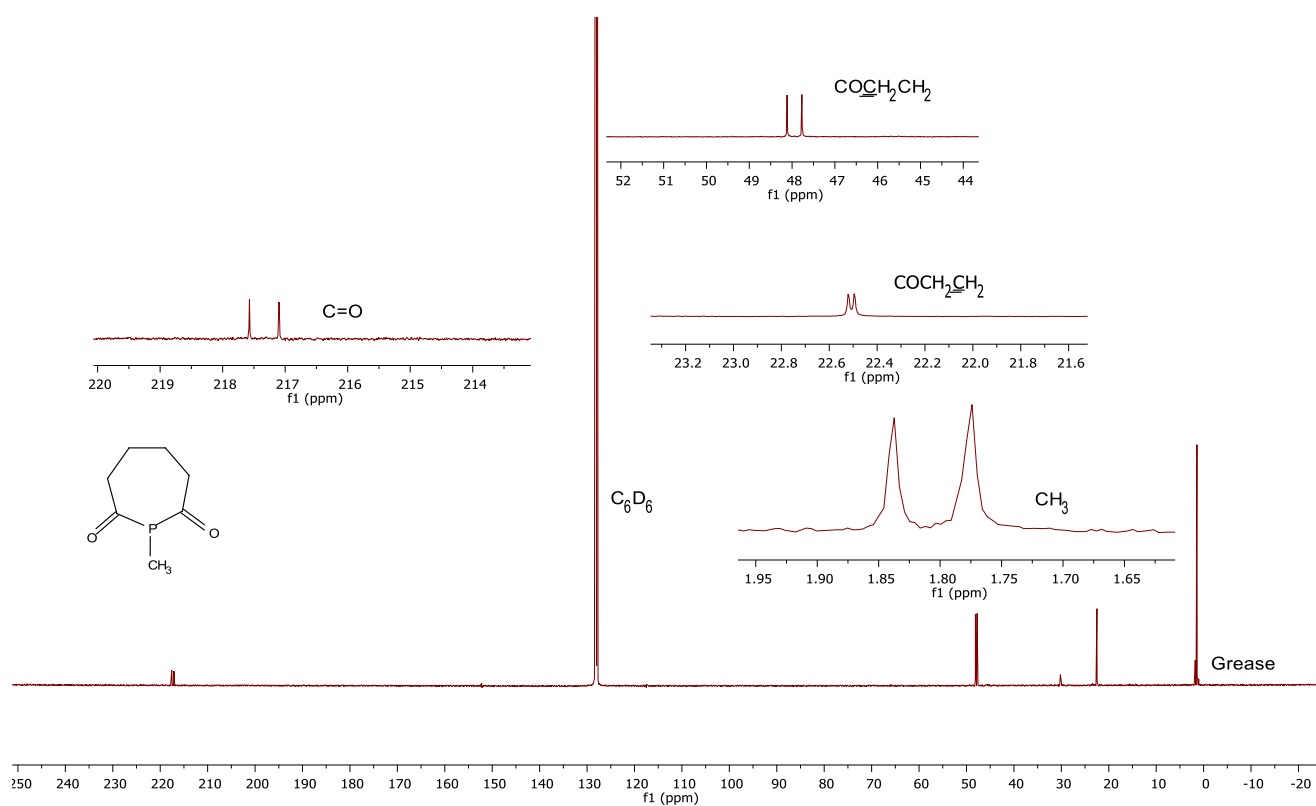
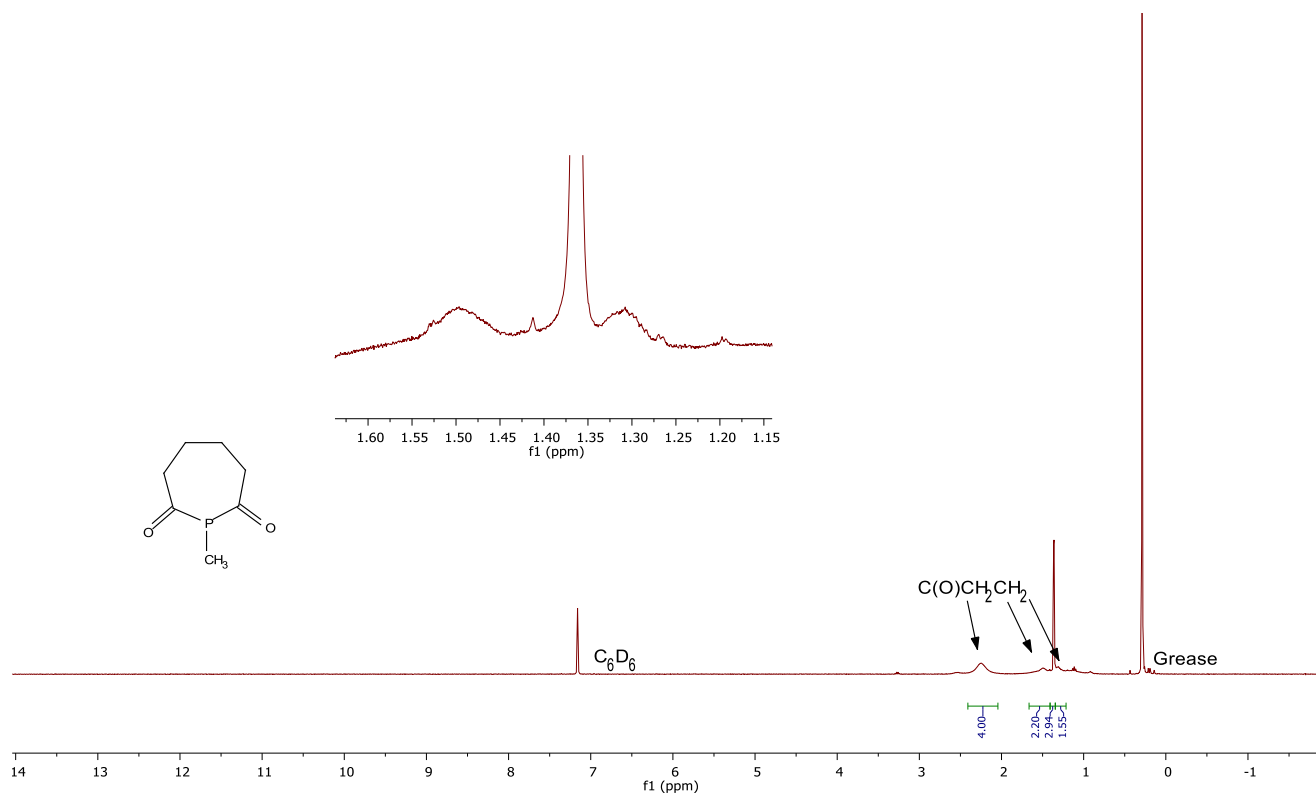
**Figure S28.**  $^1\text{H}$ - $^{13}\text{C}$  HSQC trace ( $\text{C}_6\text{D}_6$ , 303 K, 399.49, 100.46 MHz) for Compound **2e**.

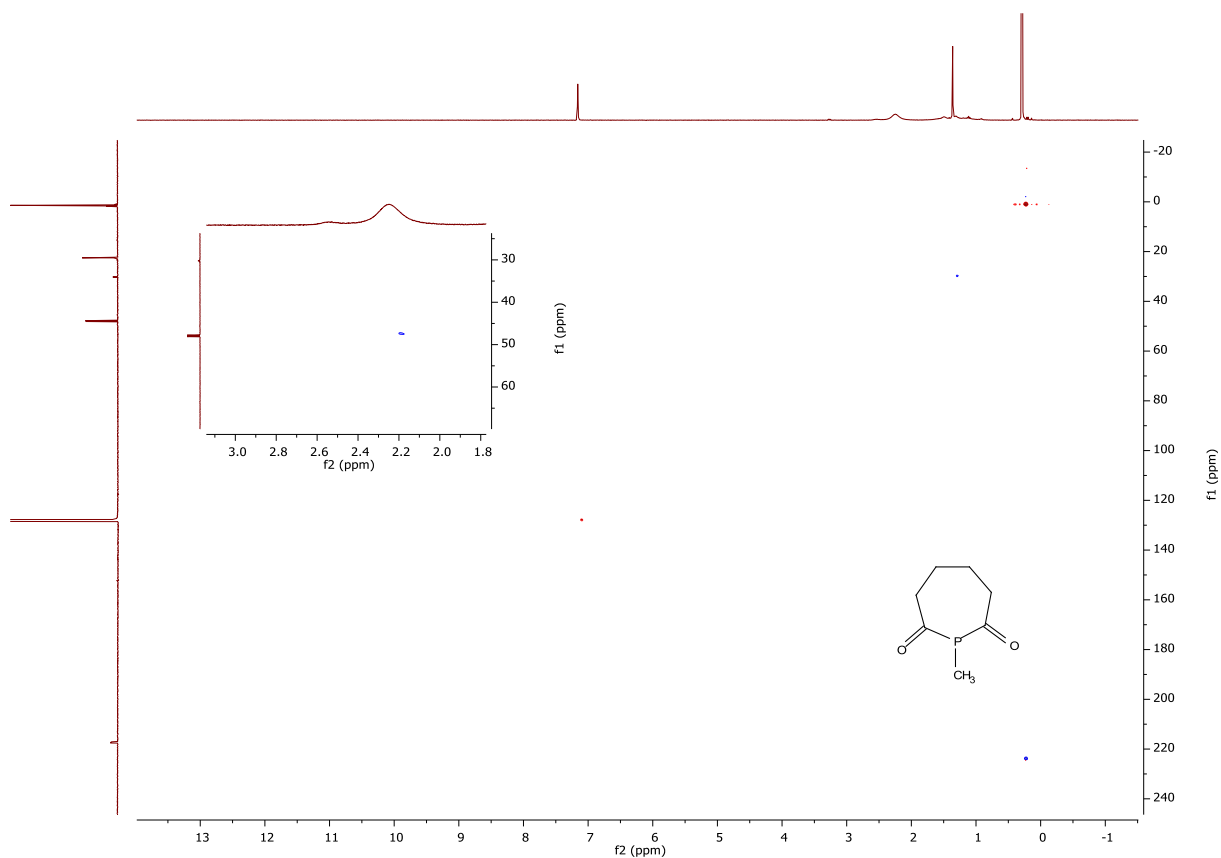


**Figure S29.**  $^1\text{H}$ - $^{13}\text{C}$  HMBC trace ( $\text{C}_6\text{D}_6$ , 303 K, 399.49, 100.46 MHz) for Compound **2e**.

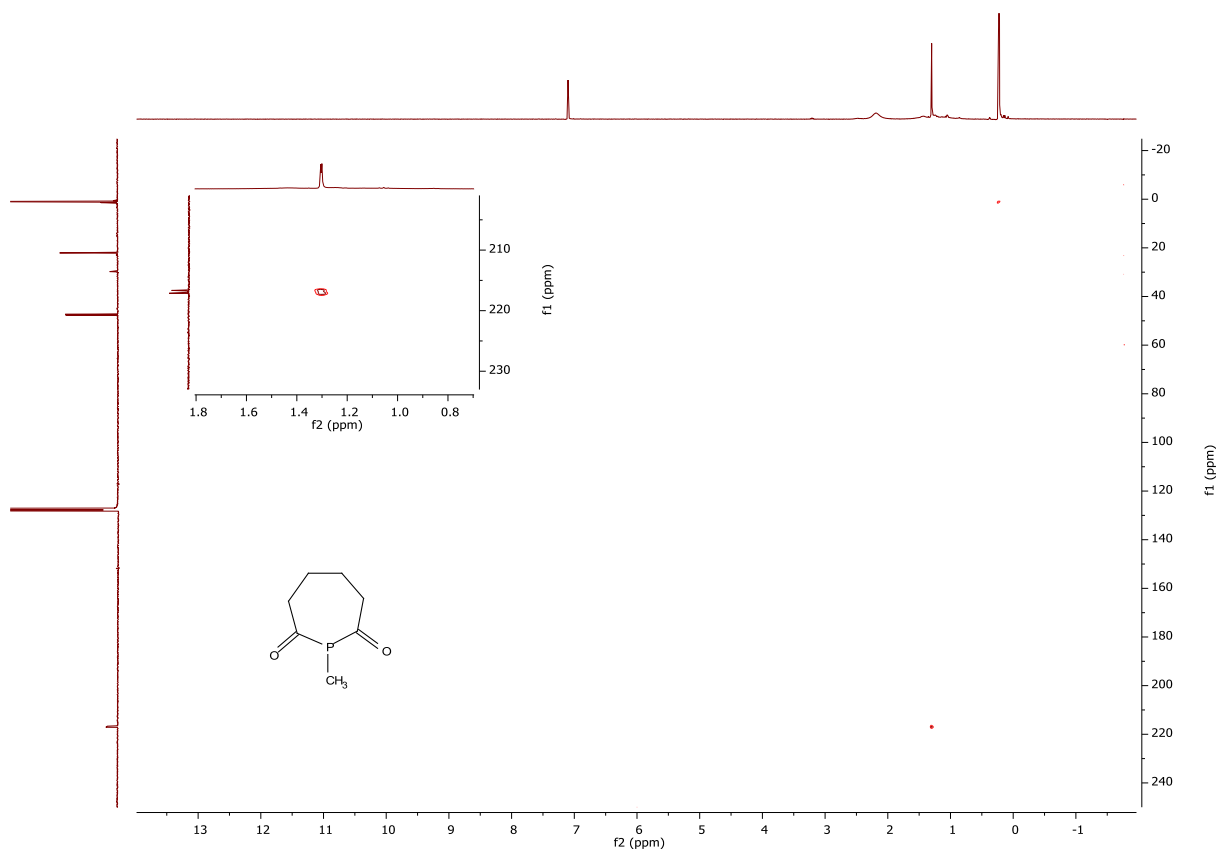




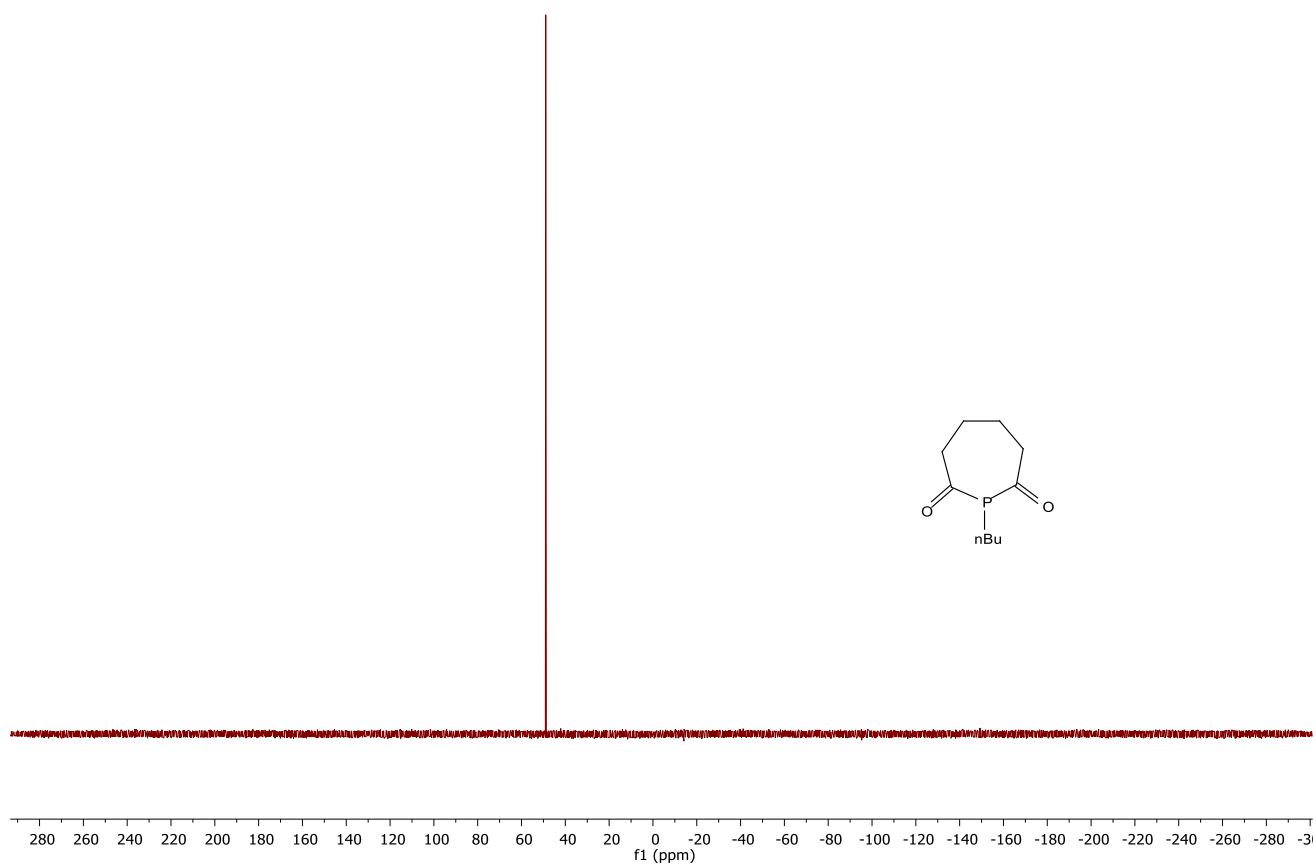




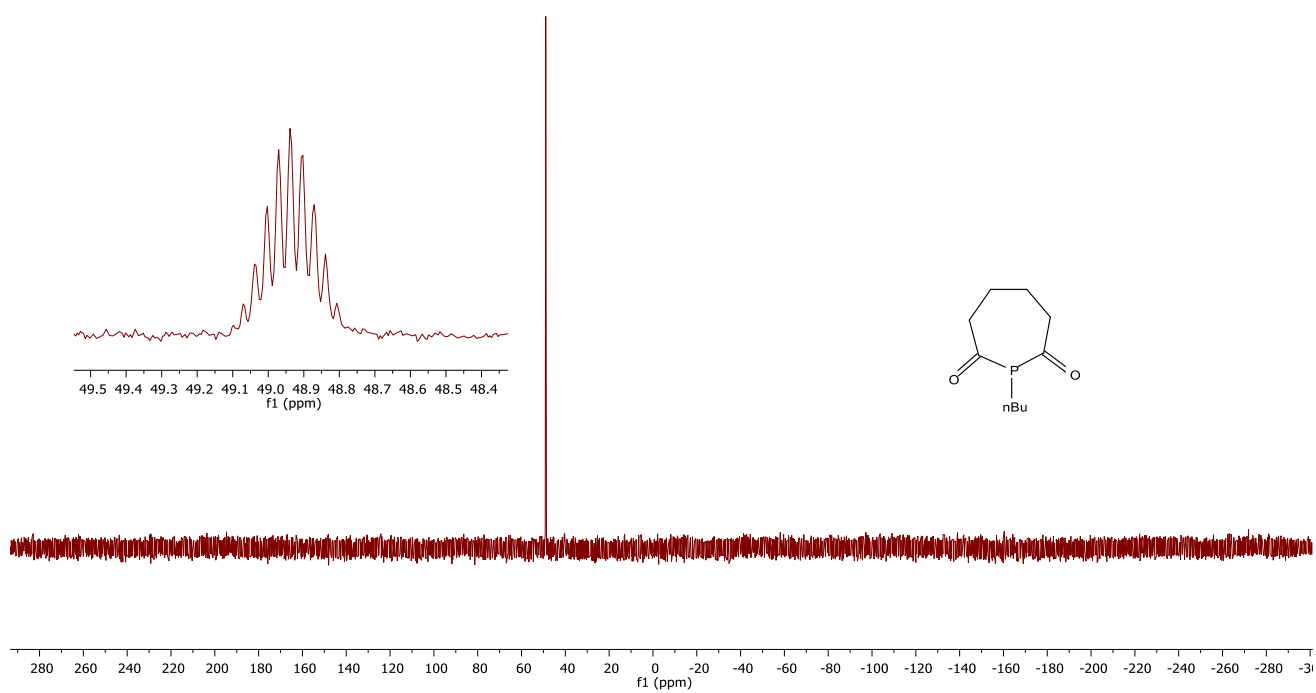
**Figure S34.**  $^1\text{H}$ - $^{13}\text{C}$  HSQC trace ( $\text{C}_6\text{D}_6$ , 303 K, 399.49, 100.46 MHz) for Compound **3a**.



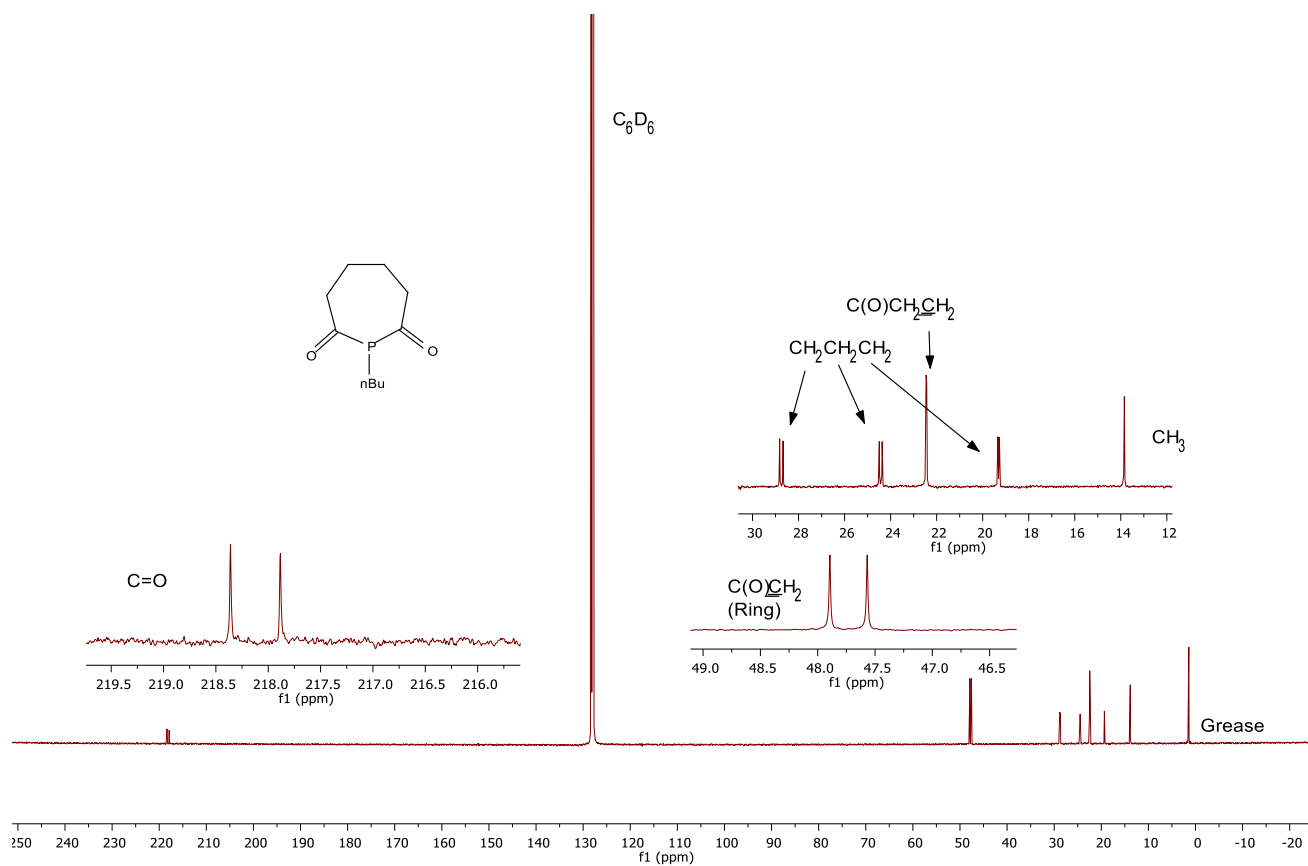
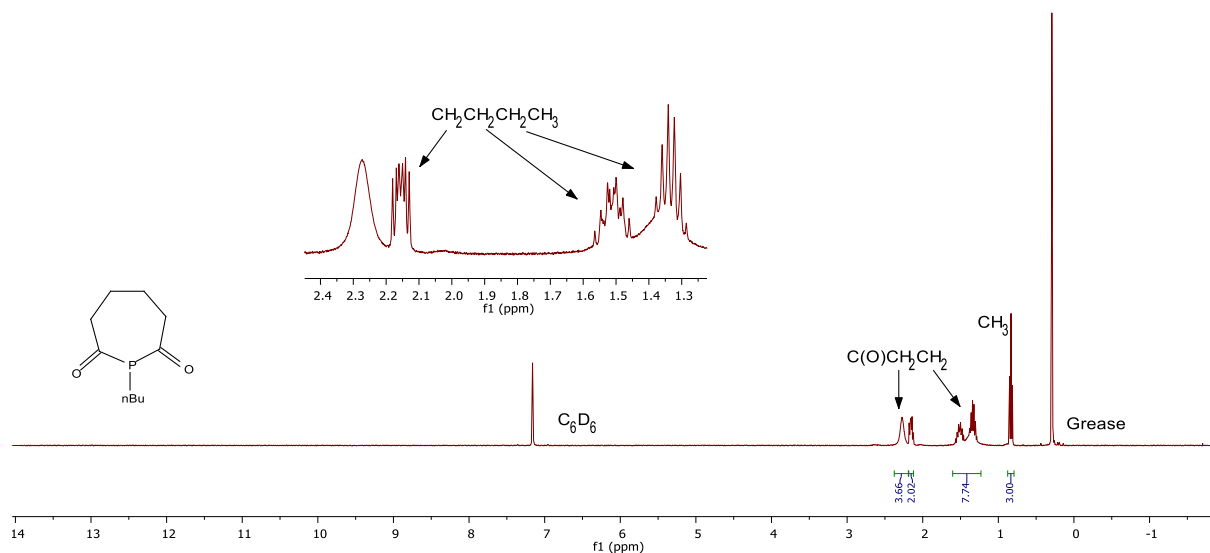
**Figure S35.**  $^1\text{H}$ - $^{13}\text{C}$  HMBC trace ( $\text{C}_6\text{D}_6$ , 303 K, 399.49, 100.46 MHz) for Compound **3a**.

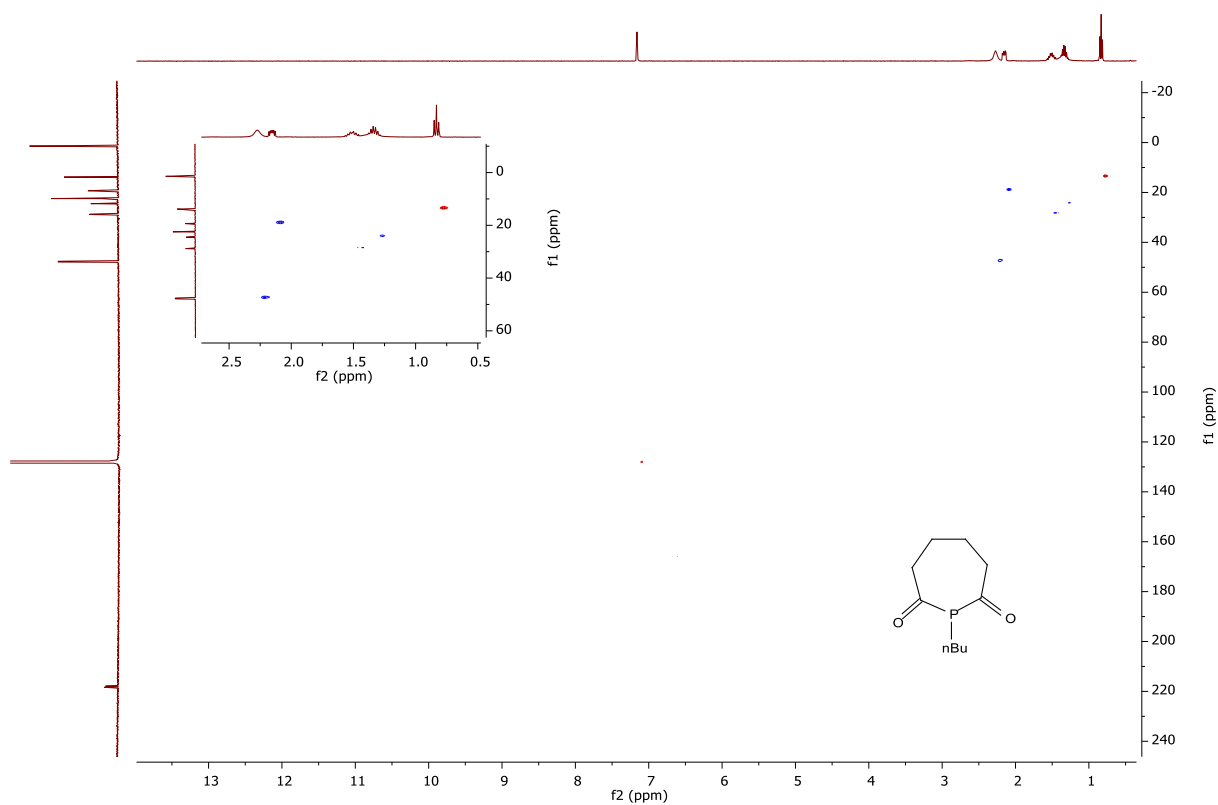


**Figure S36.**  $^{31}\text{P}\{^1\text{H}\}$  NMR Spectrum ( $\text{C}_6\text{D}_6$ , 303 K, 161.72 MHz) for compound **3b**.

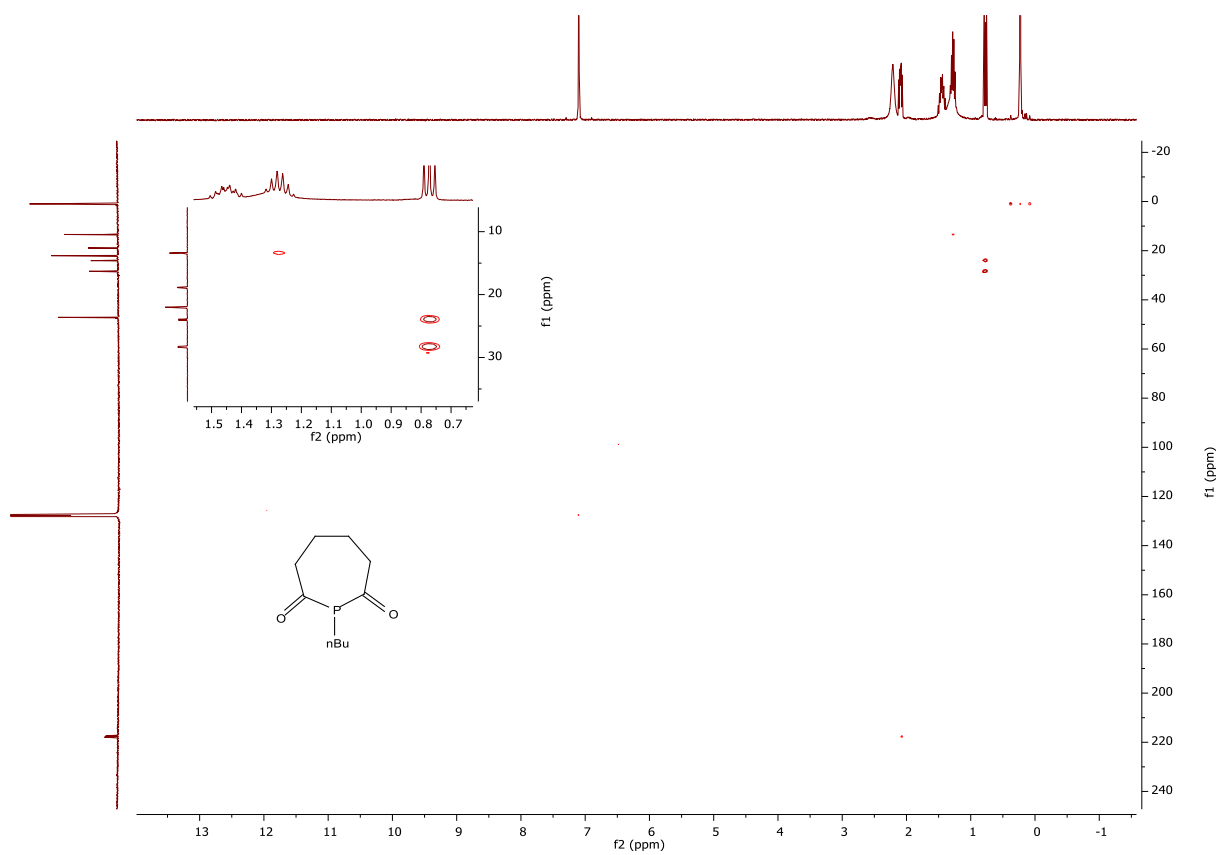


**Figure S37.**  $^{31}\text{P}$  NMR Spectrum ( $\text{C}_6\text{D}_6$ , 303 K, 161.72 MHz) for compound **3b**.

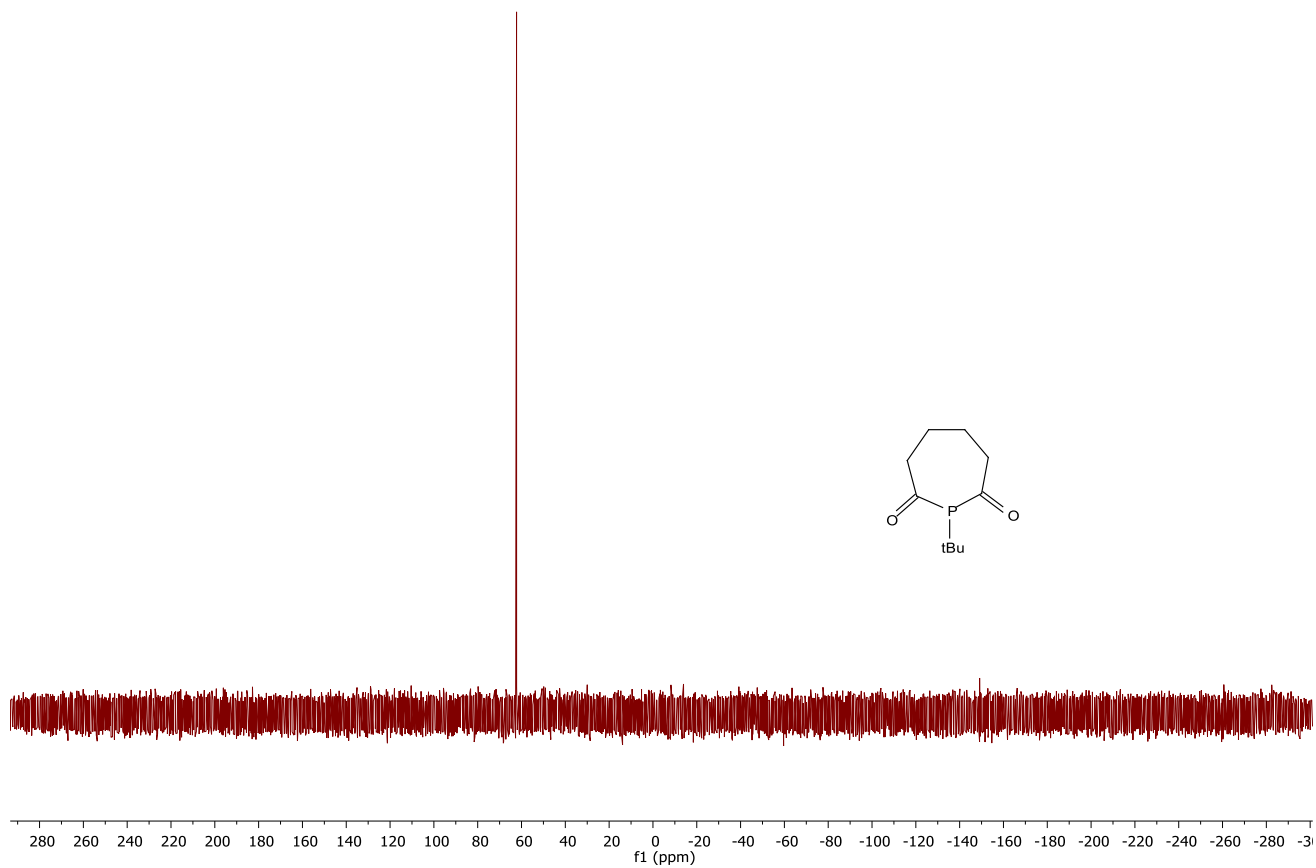




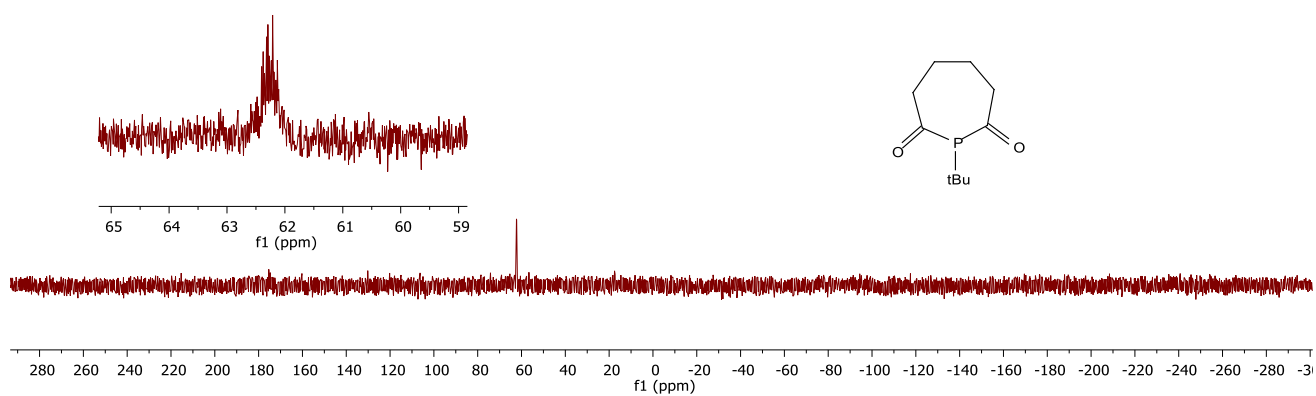
**Figure S40.**  $^1\text{H}$ - $^{13}\text{C}$  HSQC trace ( $\text{C}_6\text{D}_6$ , 303 K, 399.49, 100.46 MHz) for Compound **3b**.



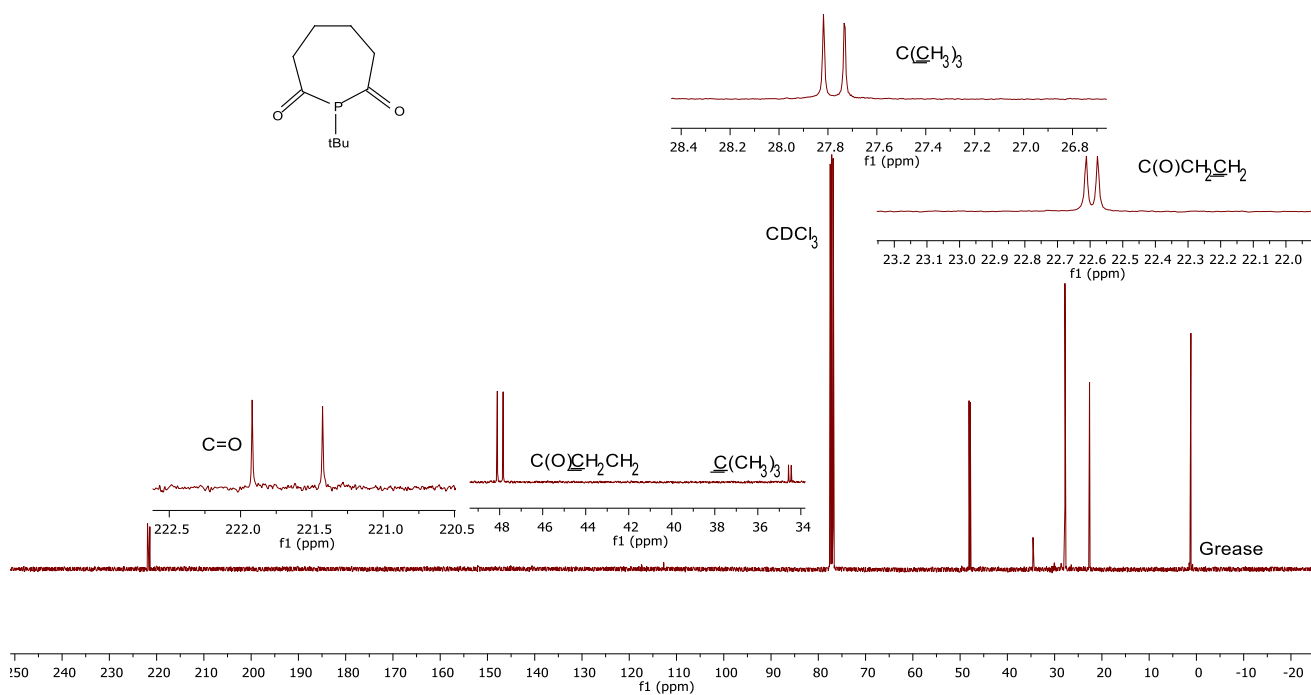
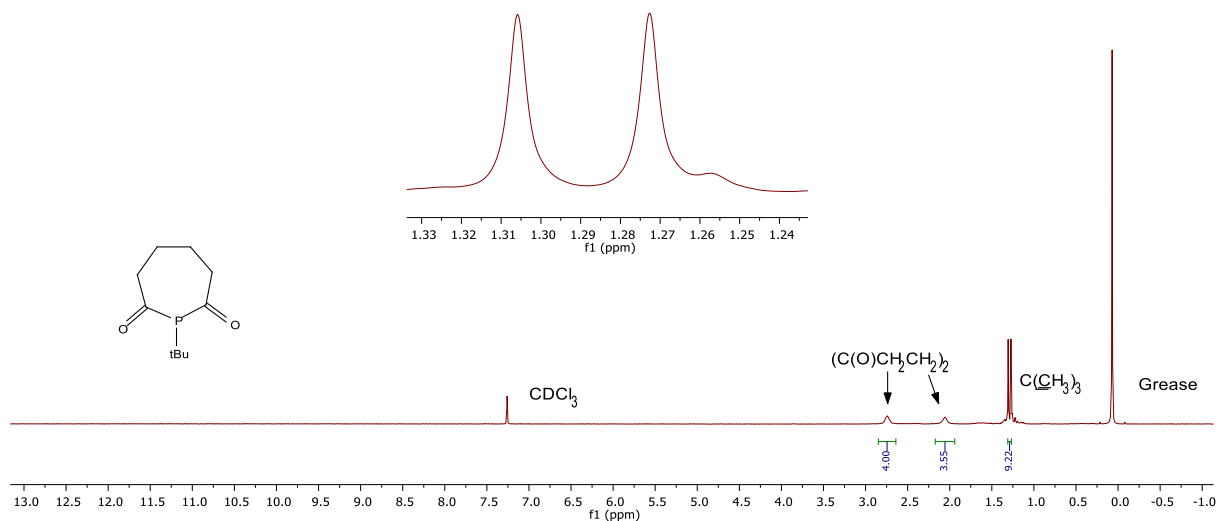
**Figure S41.**  $^1\text{H}$ - $^{13}\text{C}$  HMBC trace ( $\text{C}_6\text{D}_6$ , 303 K, 399.49, 100.46 MHz) for Compound **3b**.



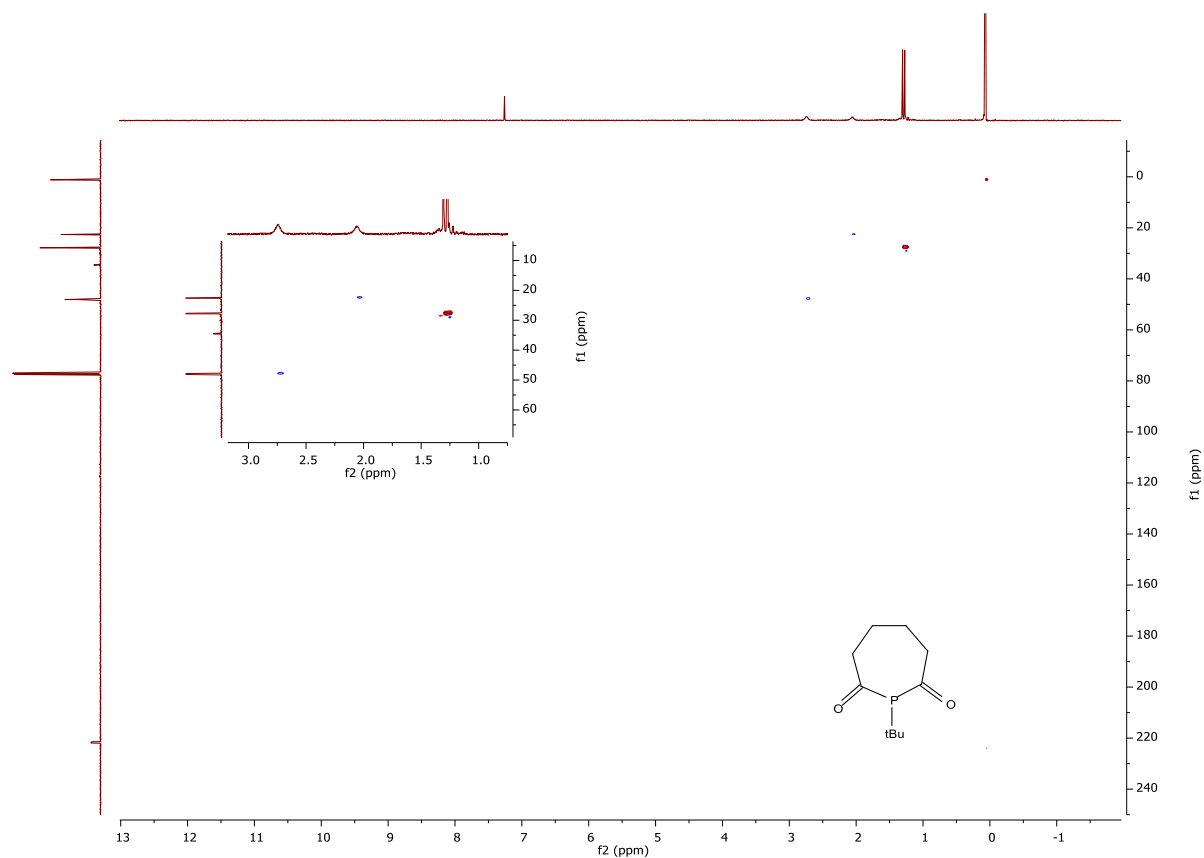
**Figure S42.**  $^{31}\text{P}\{^1\text{H}\}$  NMR Spectrum ( $\text{CDCl}_3$ , 303 K, 161.72 MHz) for compound **3c**.



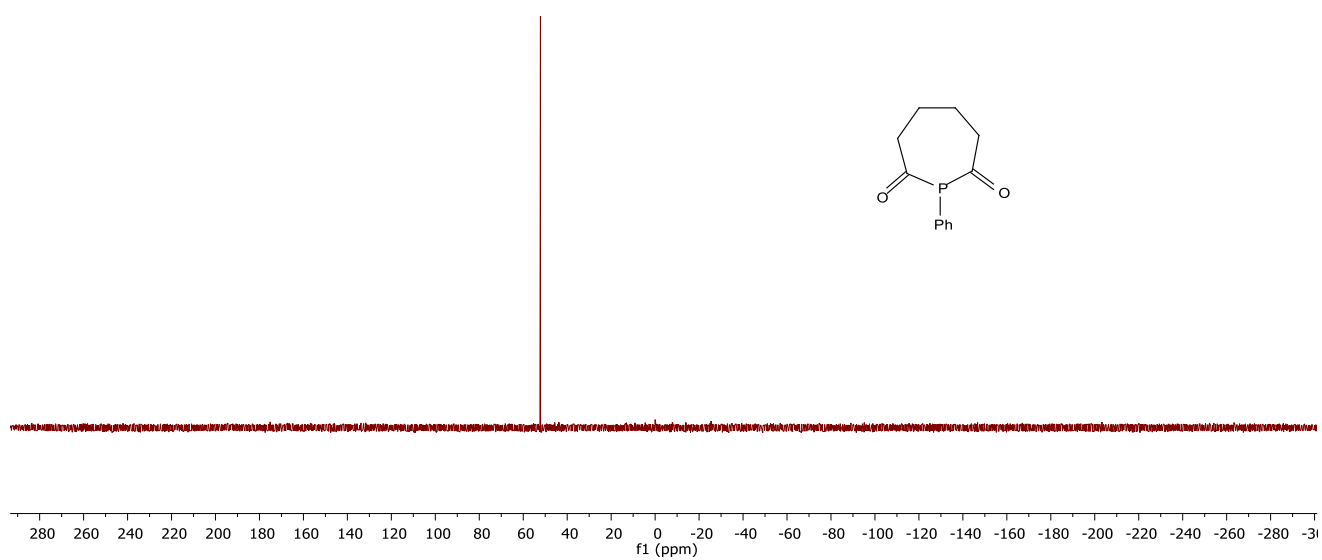
**Figure S43.**  $^{31}\text{P}$  NMR Spectrum ( $\text{CDCl}_3$ , 303 K, 161.72 MHz) for compound **3c**.



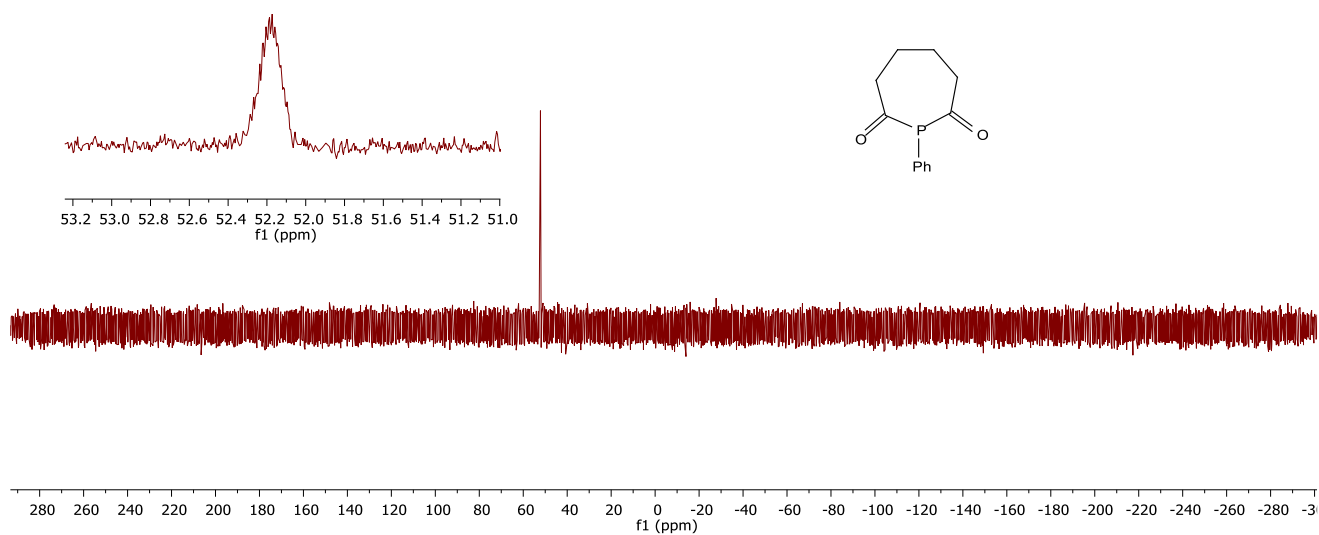




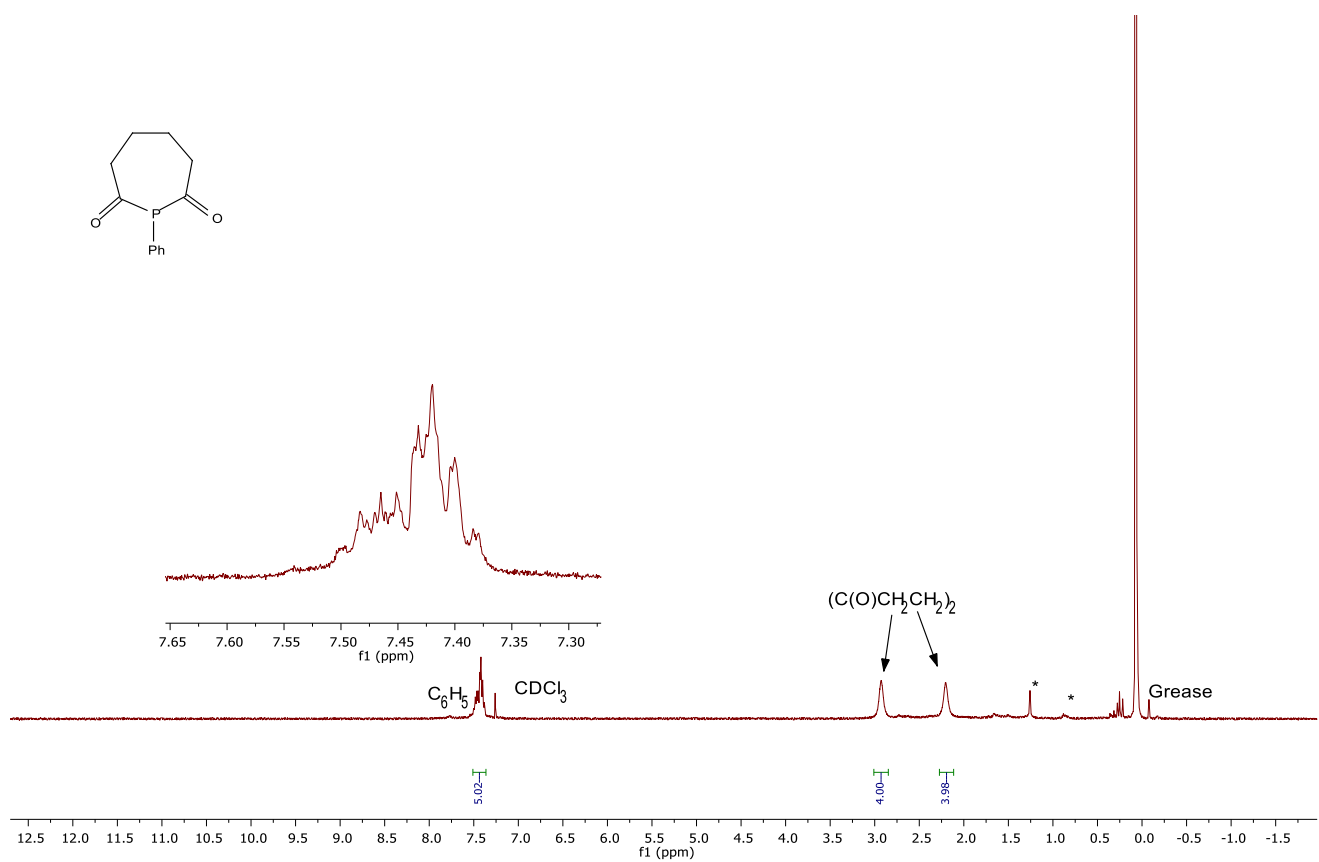
**Figure S46.**  $^1\text{H}$ - $^{13}\text{C}$  HSQC trace ( $\text{CDCl}_3$ , 303 K, 399.49, 100.46 MHz) for Compound **3c**.



**Figure S47.**  $^{31}\text{P}\{^1\text{H}\}$  NMR Spectrum ( $\text{CDCl}_3$ , 303 K, 161.72 MHz) for compound **3d**.



**Figure S48.**  $^{31}\text{P}$  NMR Spectrum ( $\text{CDCl}_3$ , 303 K, 161.72 MHz) for compound **3d**.



**Figure S49.**  $^1\text{H}$  NMR Spectrum ( $\text{CDCl}_3$ , 303 K, 399.49 MHz) for compound **3d**. \* corresponds to H Grease.

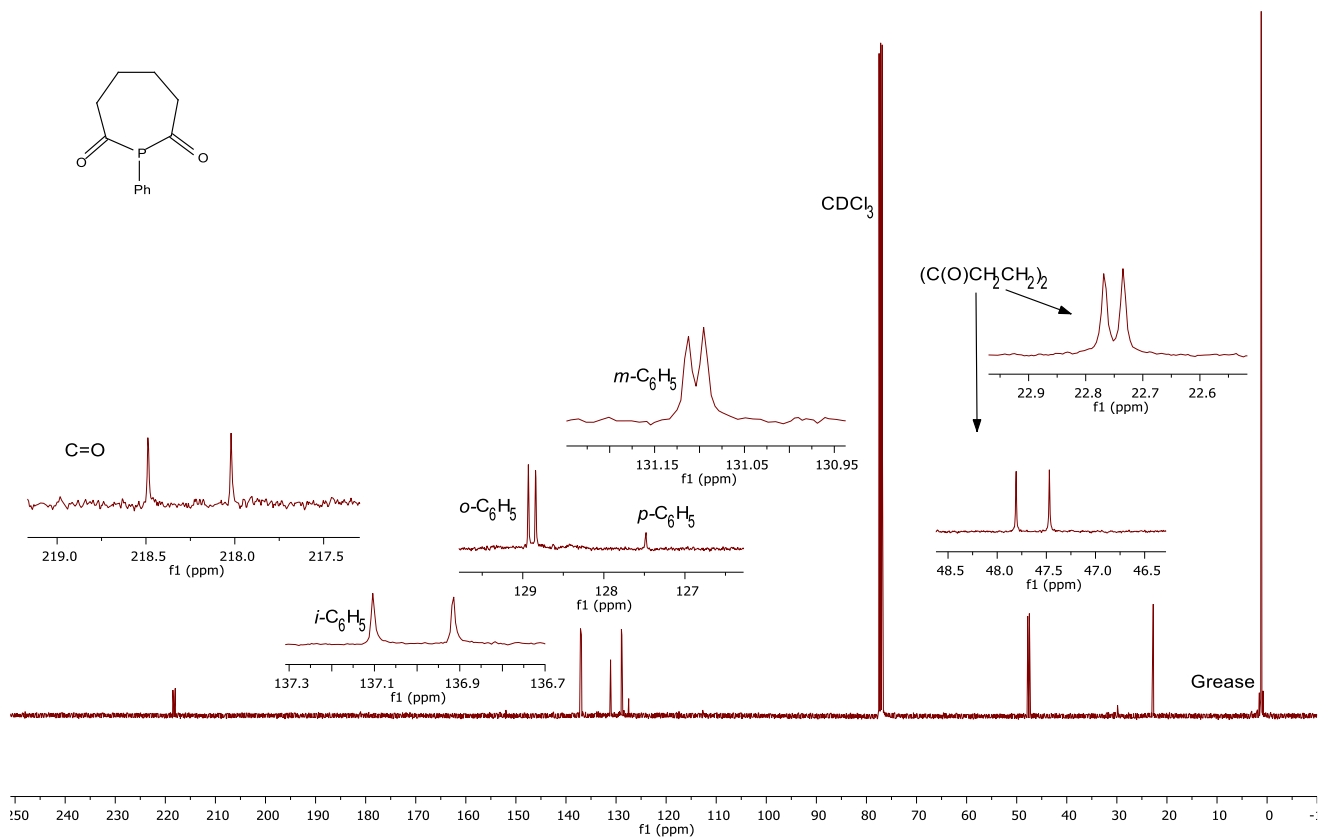


Figure S50.  $^{13}\text{C}\{^1\text{H}\}$  NMR Spectrum ( $\text{CDCl}_3$ , 303 K, 100.46 MHz) for compound 3d.

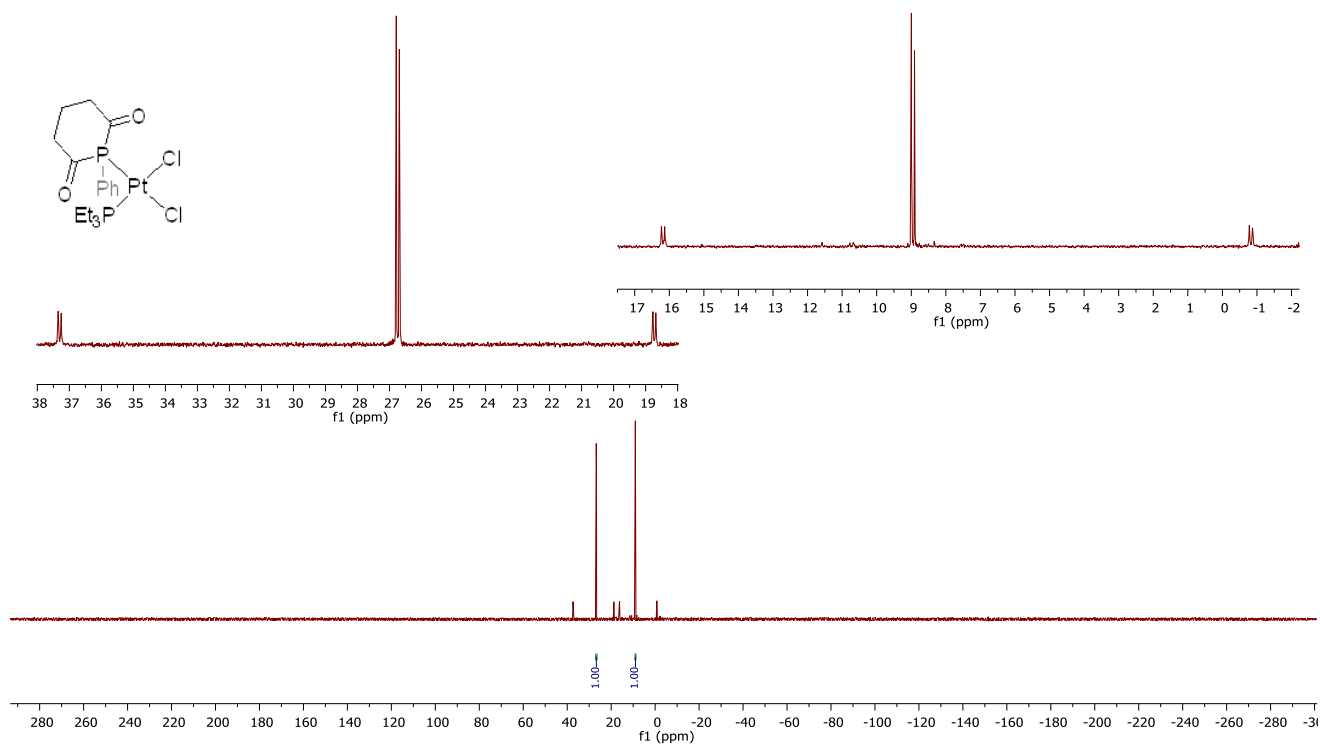


Figure S51.  $^{31}\text{P}\{^1\text{H}\}$  NMR Spectrum ( $\text{CD}_2\text{Cl}_2$ , 303 K, 161.72 MHz, D1 = 30 s) for compound 4.

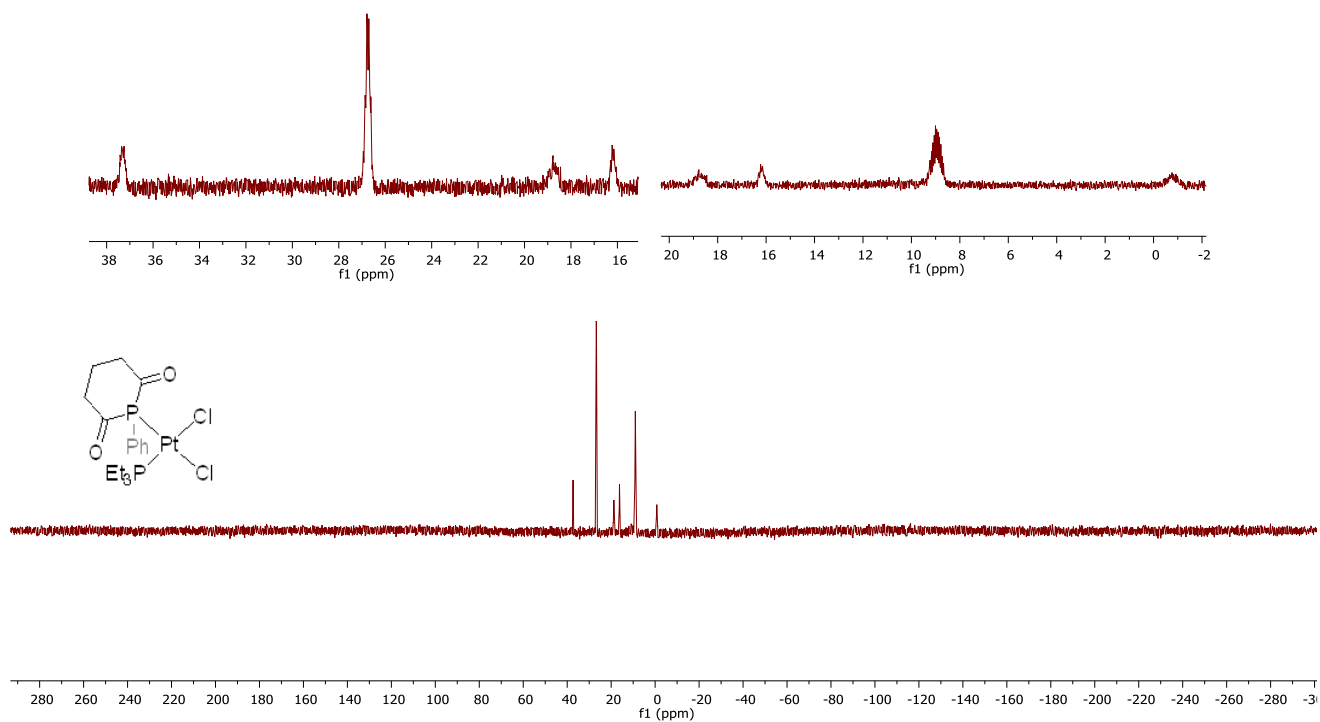


Figure S52.  $^{31}\text{P}$  NMR Spectrum ( $\text{CD}_2\text{Cl}_2$ , 303 K, 161.72 MHz,  $D1 = 30$  s) for compound 4.

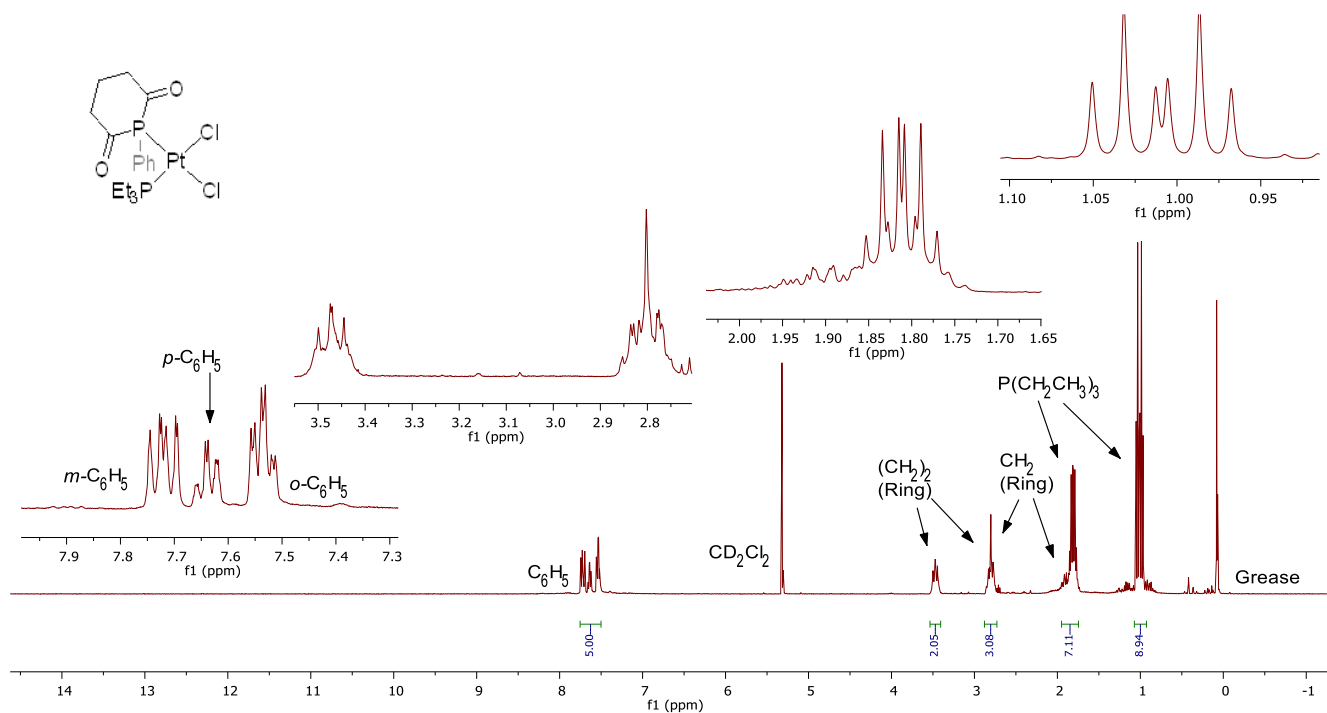
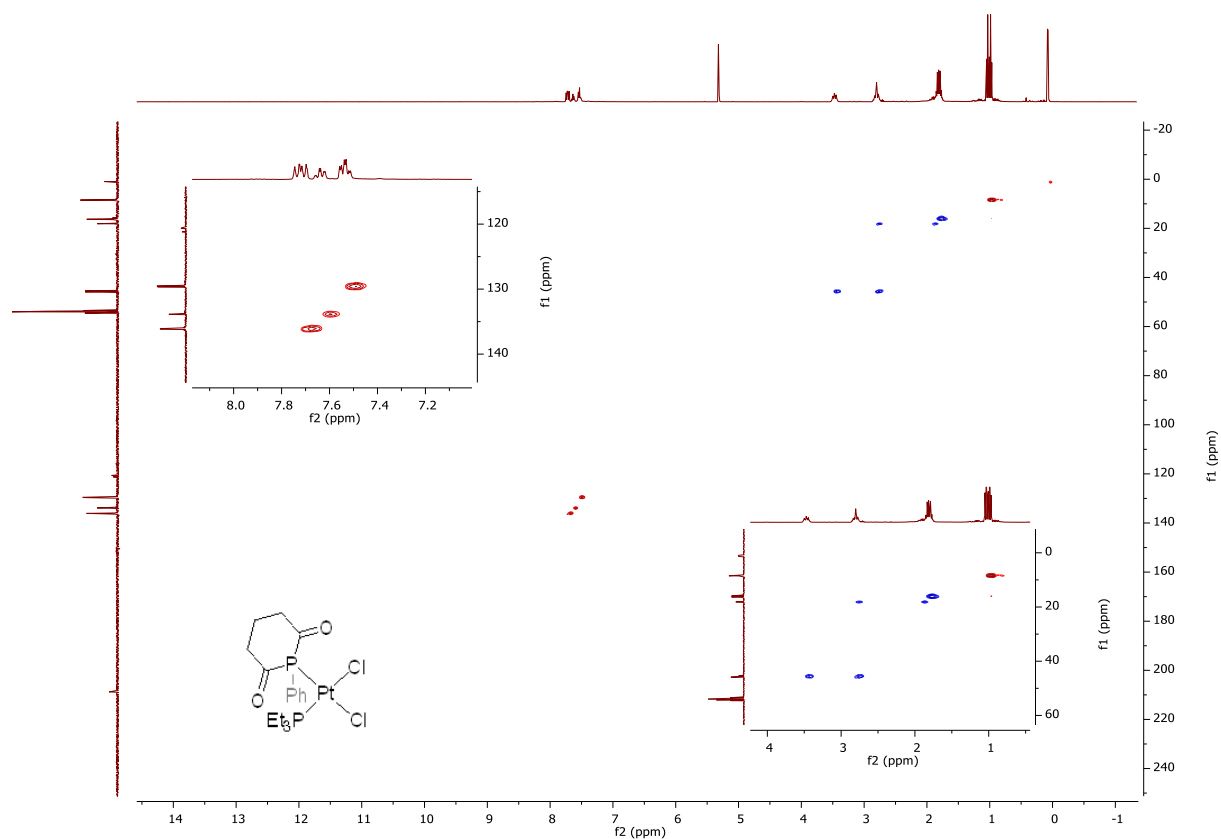
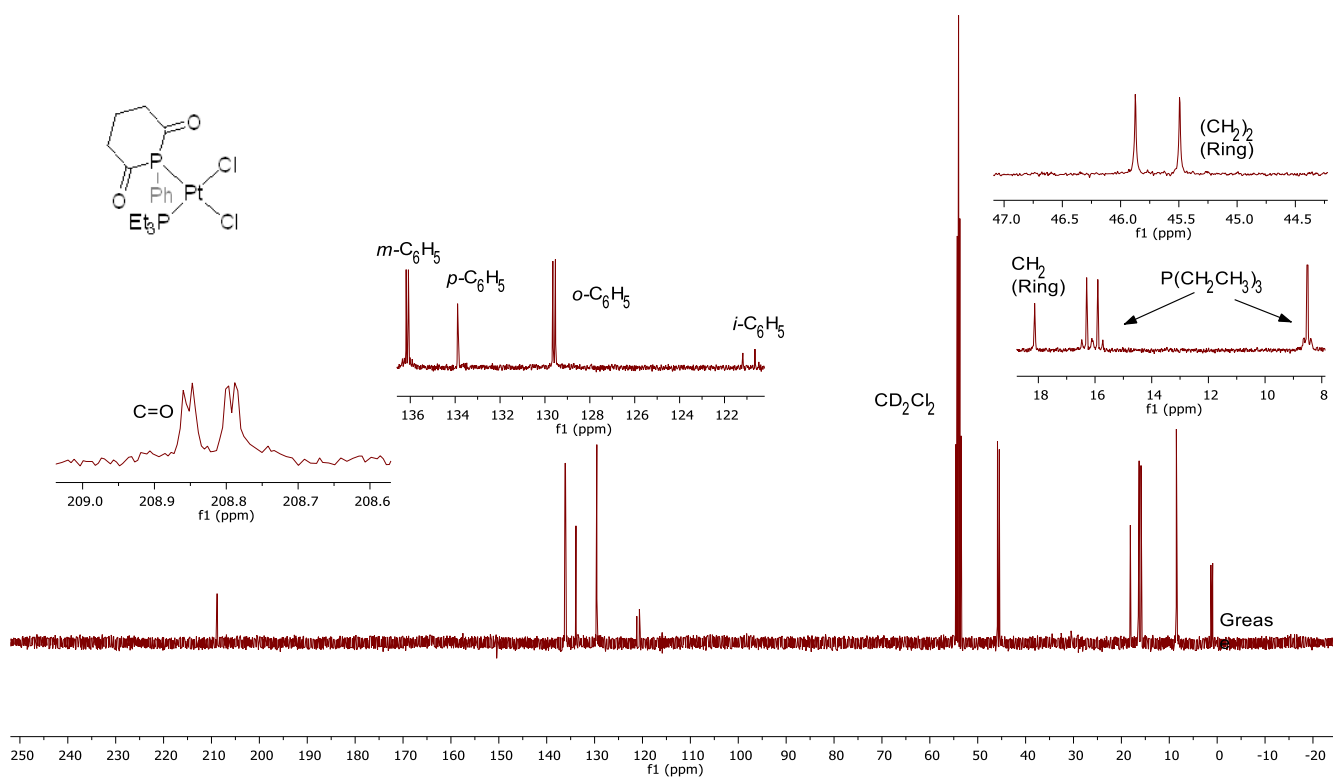
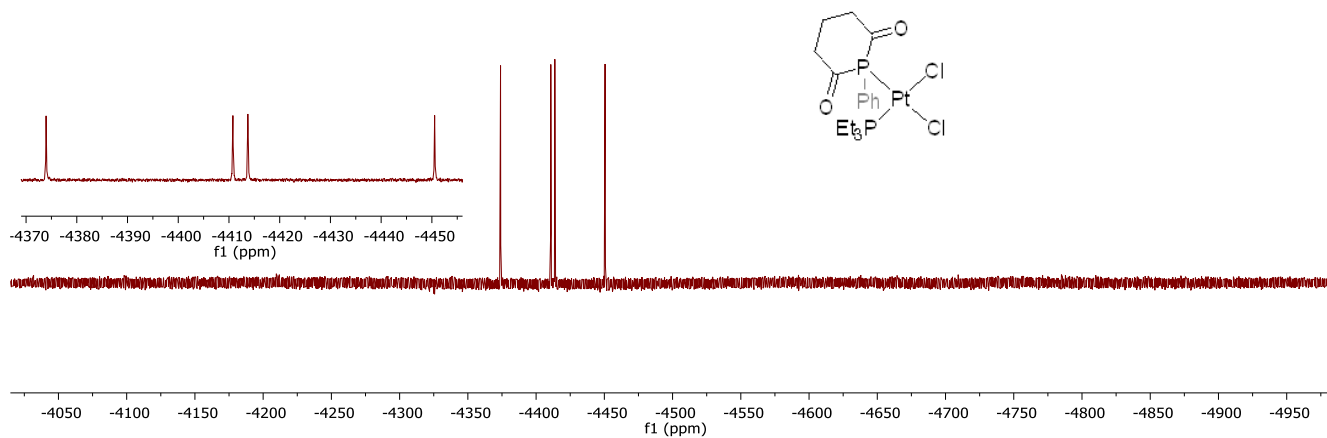
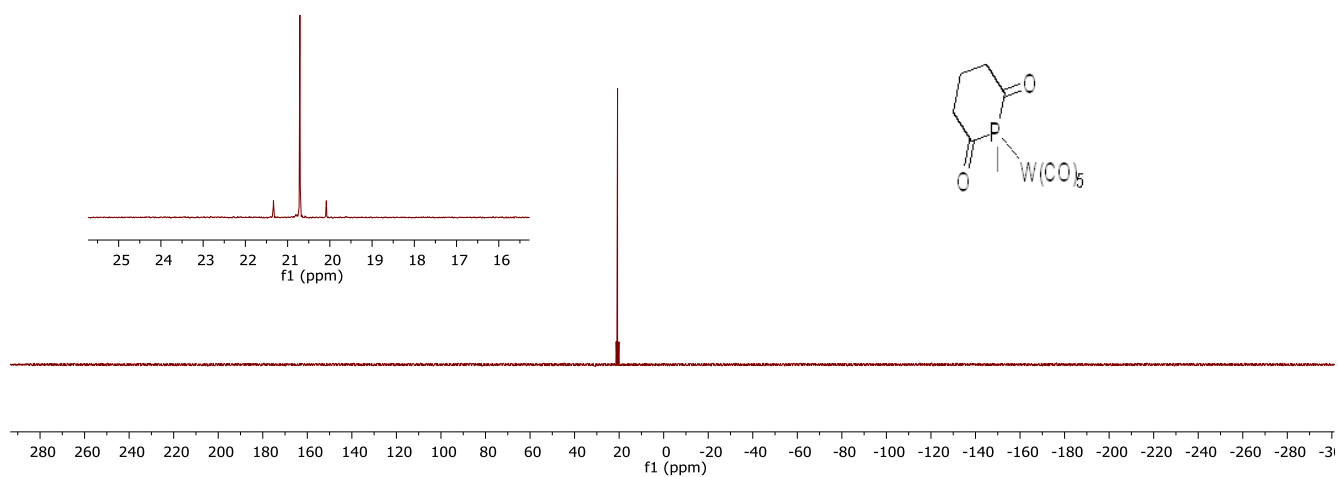


Figure S53.  $^1\text{H}$  NMR Spectrum ( $\text{CD}_2\text{Cl}_2$ , 303 K, 399.49 MHz) for compound 4.

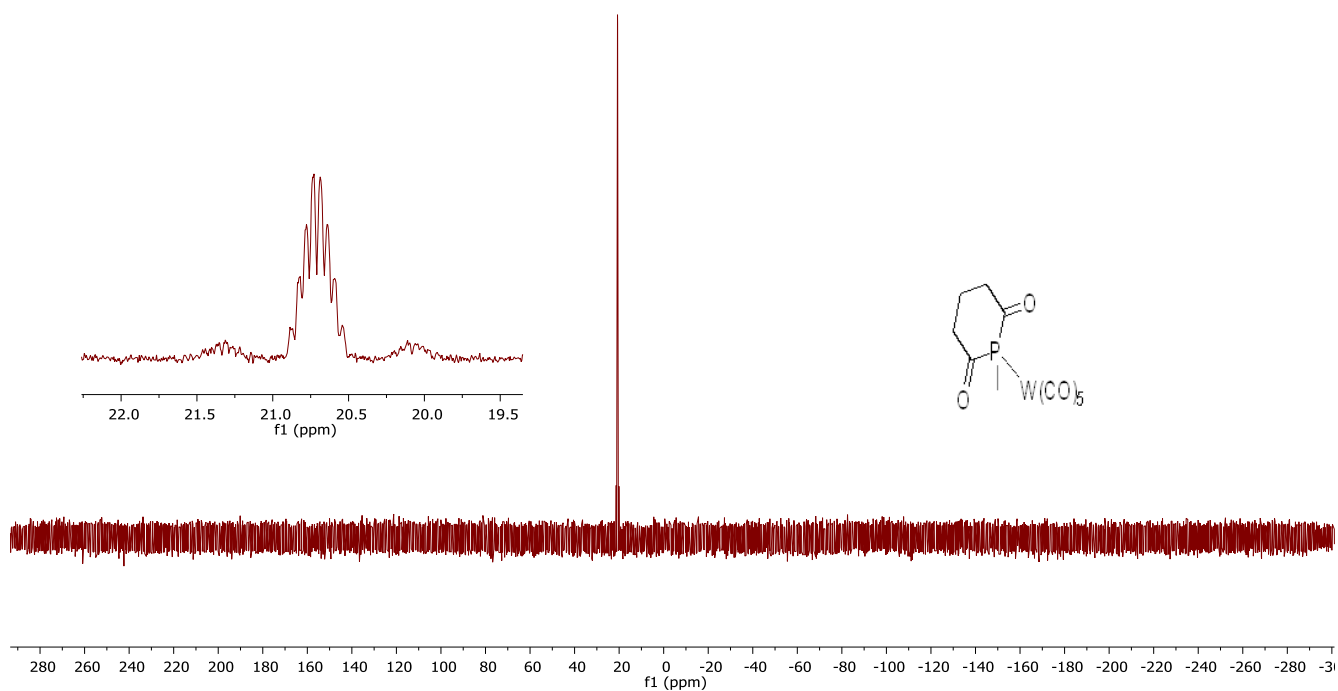




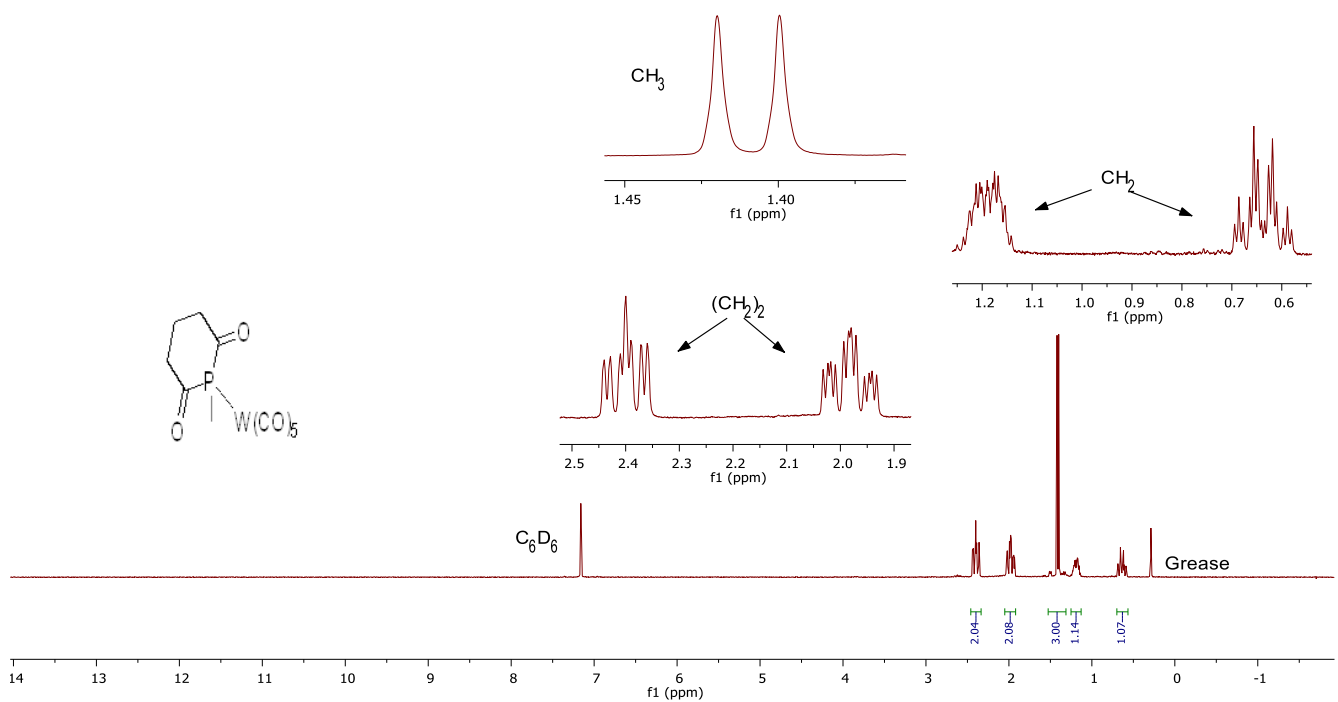
**Figure S56.**  $^{195}\text{Pt}\{^1\text{H}\}$  NMR Spectrum ( $\text{CD}_2\text{Cl}_2$ , 303 K, 85.49 MHz) for Compound 4.



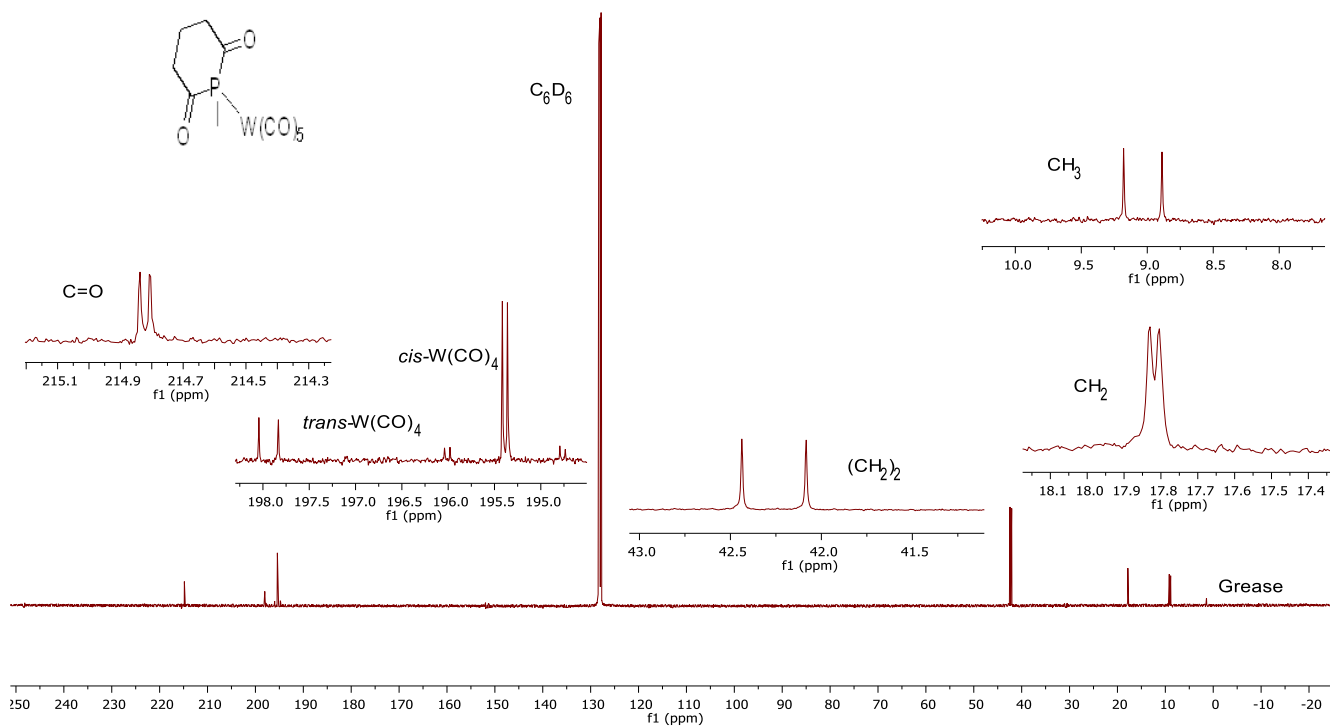
**Figure S57.**  $^{31}\text{P}\{^1\text{H}\}$  NMR Spectrum ( $\text{C}_6\text{D}_6$ , 303 K, 161.72 MHz) for compound 5a.



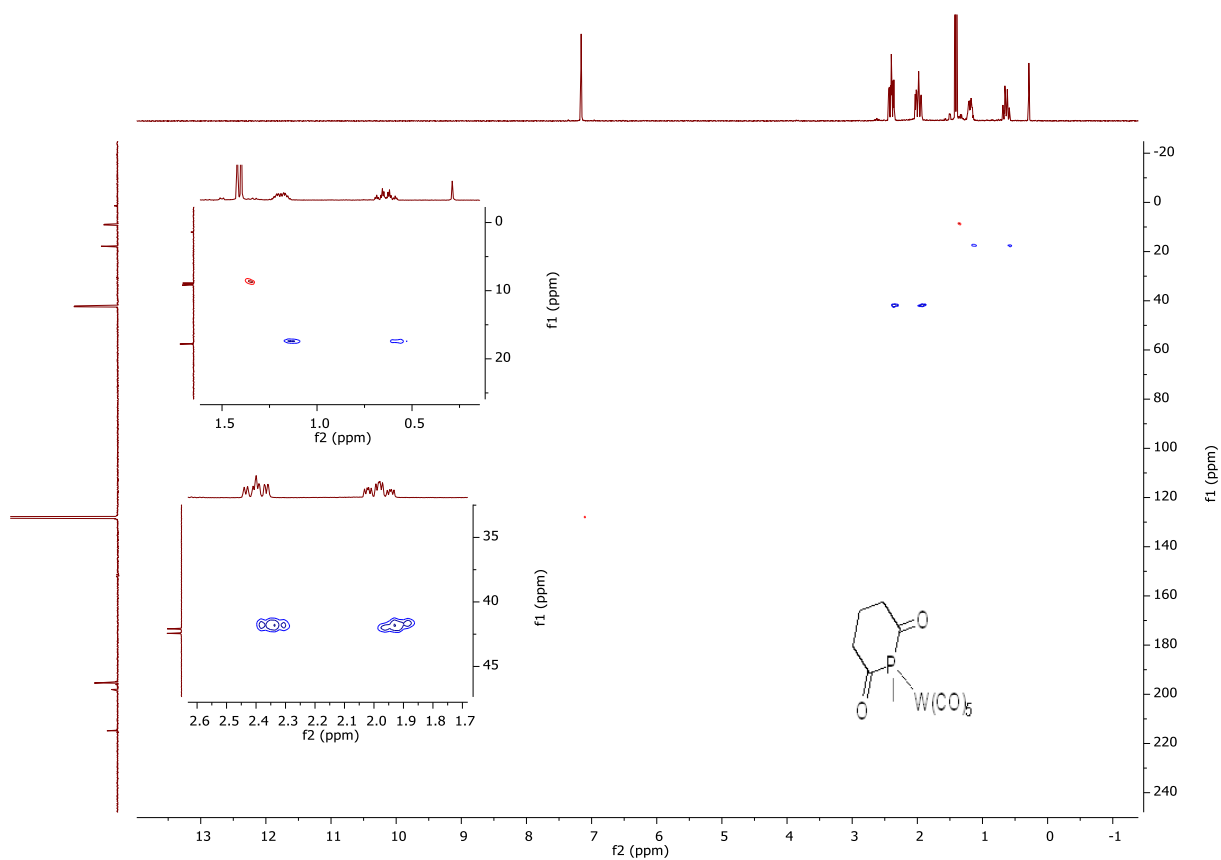
**Figure S58.**  $^{31}\text{P}$  NMR Spectrum ( $\text{C}_6\text{D}_6$ , 303 K, 161.72 MHz) for compound **5a**.



**Figure S59.**  $^1\text{H}$  NMR Spectrum ( $\text{C}_6\text{D}_6$ , 303 K, 399.49 MHz) for compound **5a**.

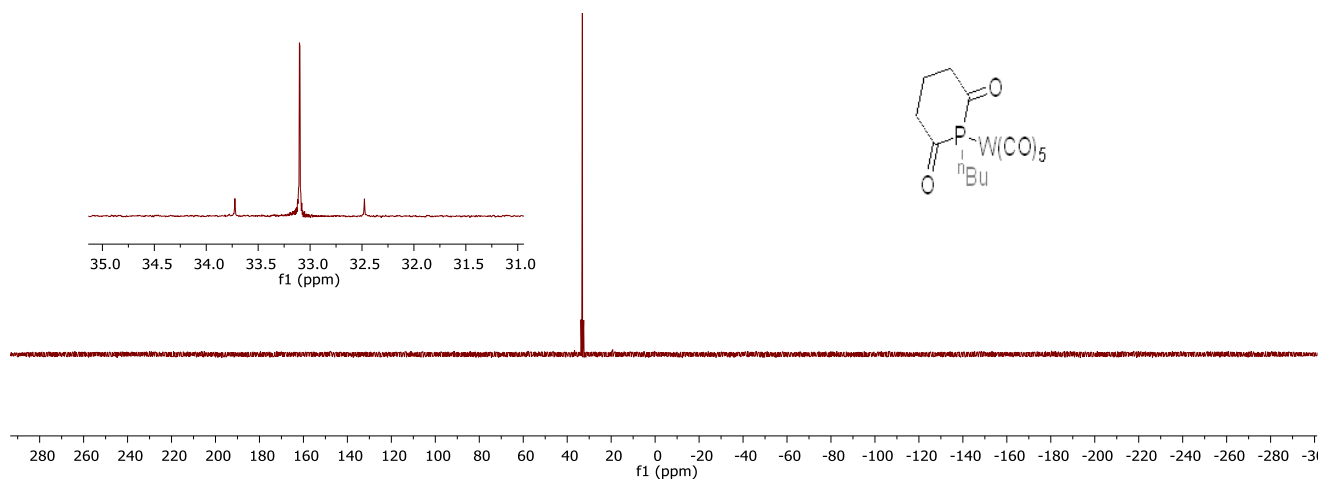
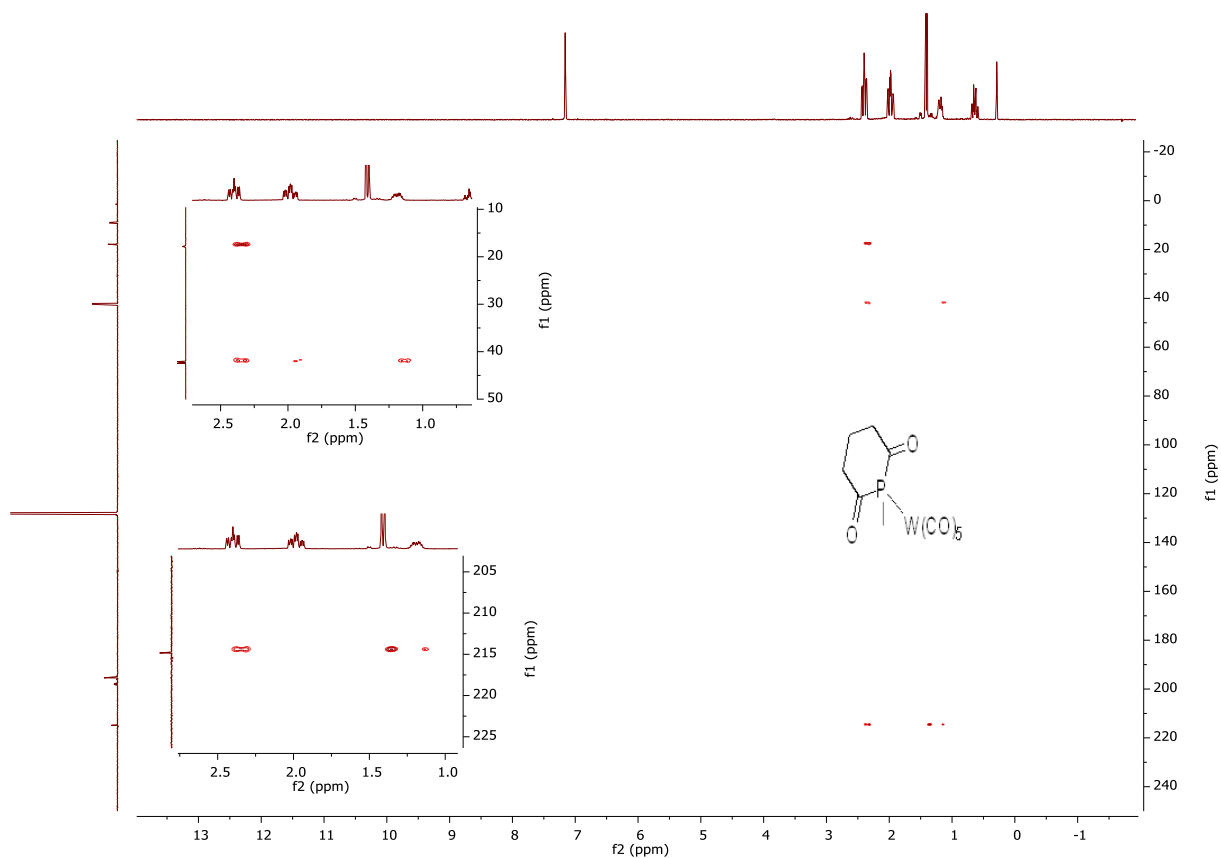


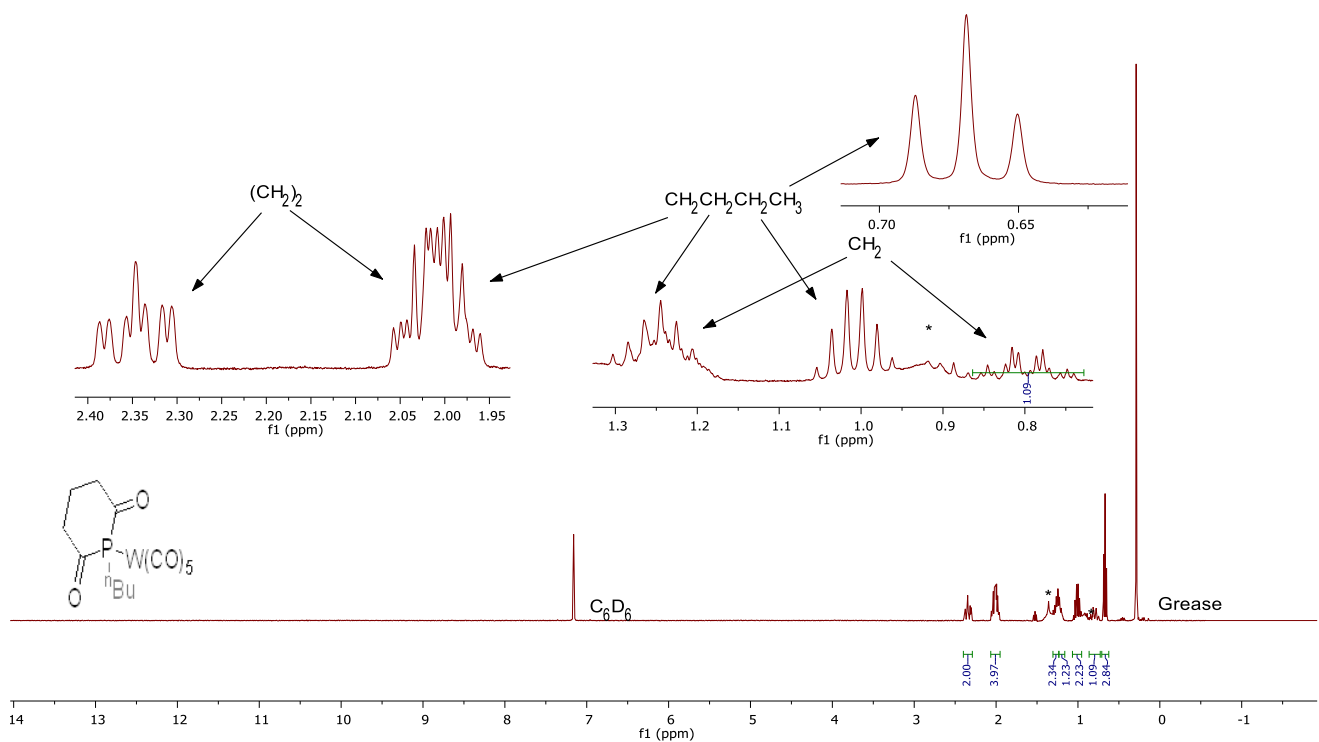
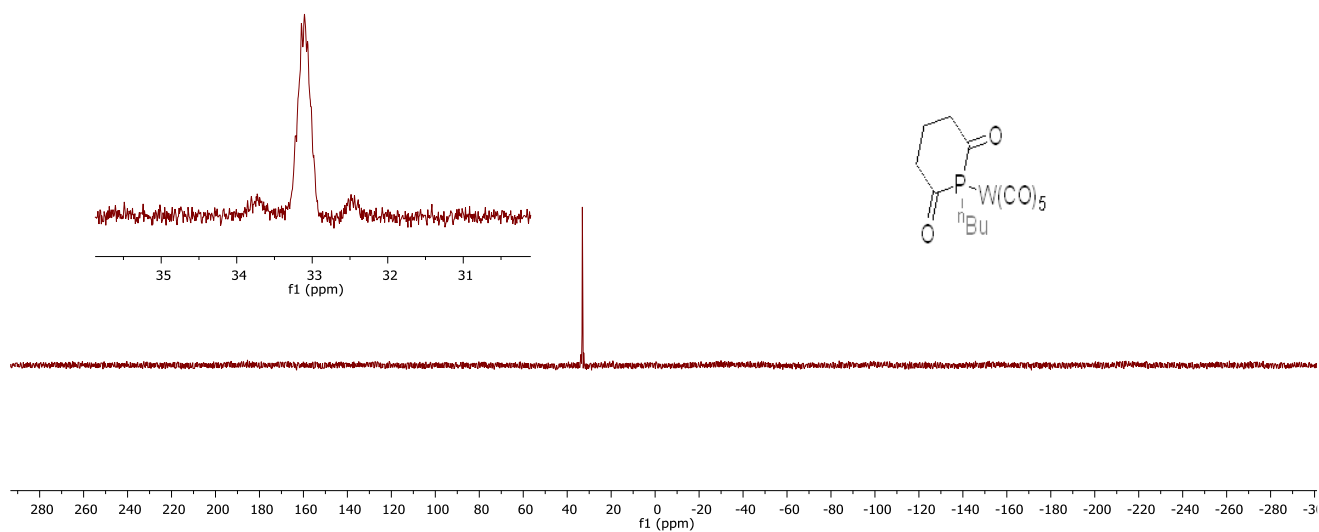
**Figure S60.**  $^{13}\text{C}\{^1\text{H}\}$  NMR Spectrum ( $\text{C}_6\text{D}_6$ , 303 K, 100.46 MHz) for compound **5a**.

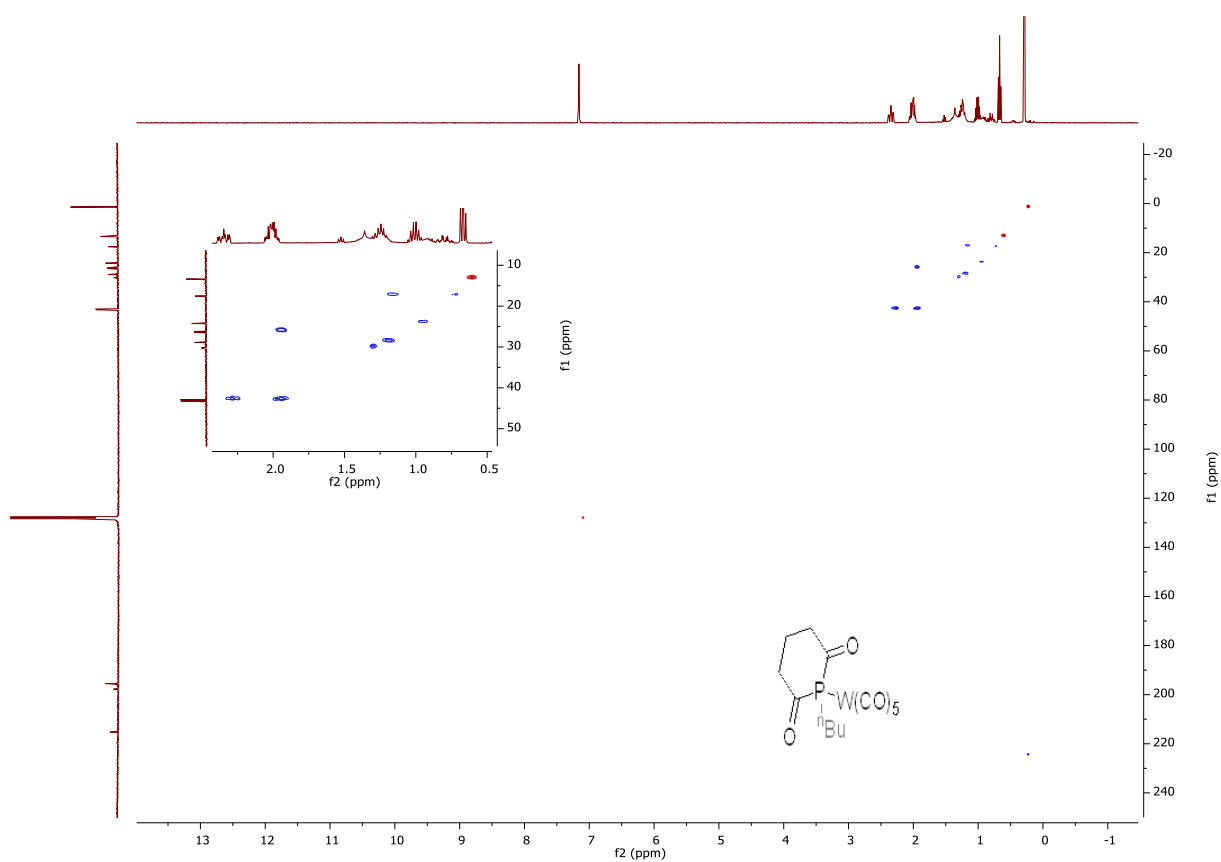
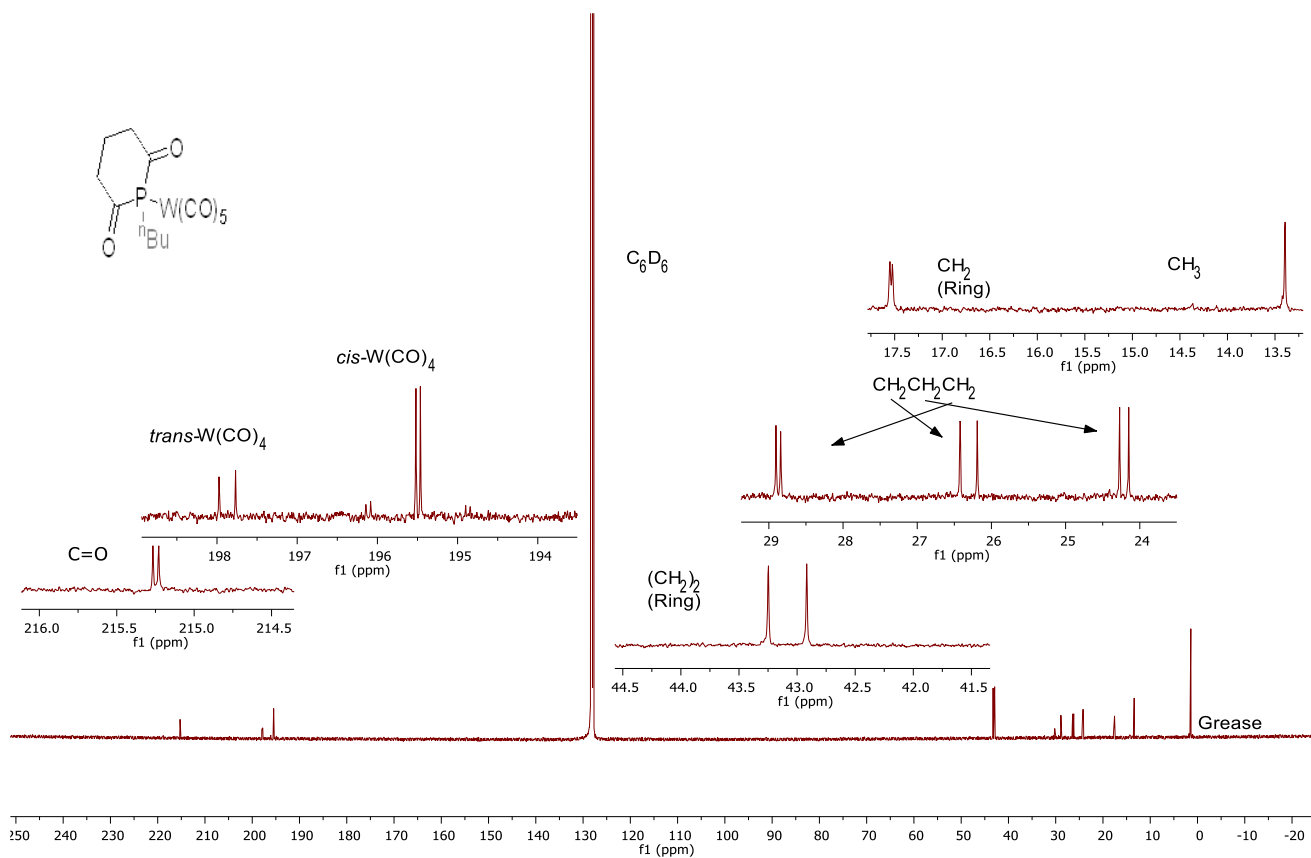


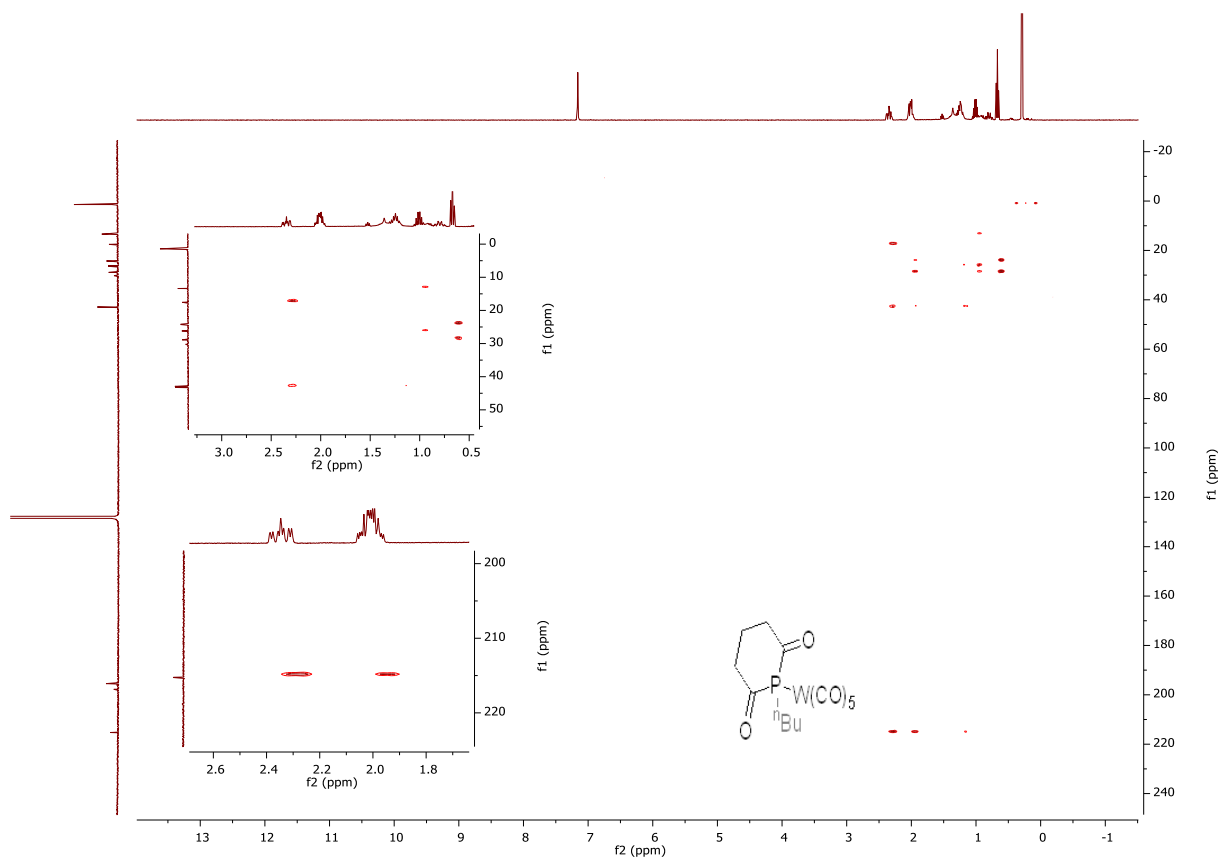
**Figure S61.**  $^1\text{H}$ - $^{13}\text{C}$  HSQC trace ( $\text{C}_6\text{D}_6$ , 303 K, 399.49, 100.46 MHz) for Compound **5a**.



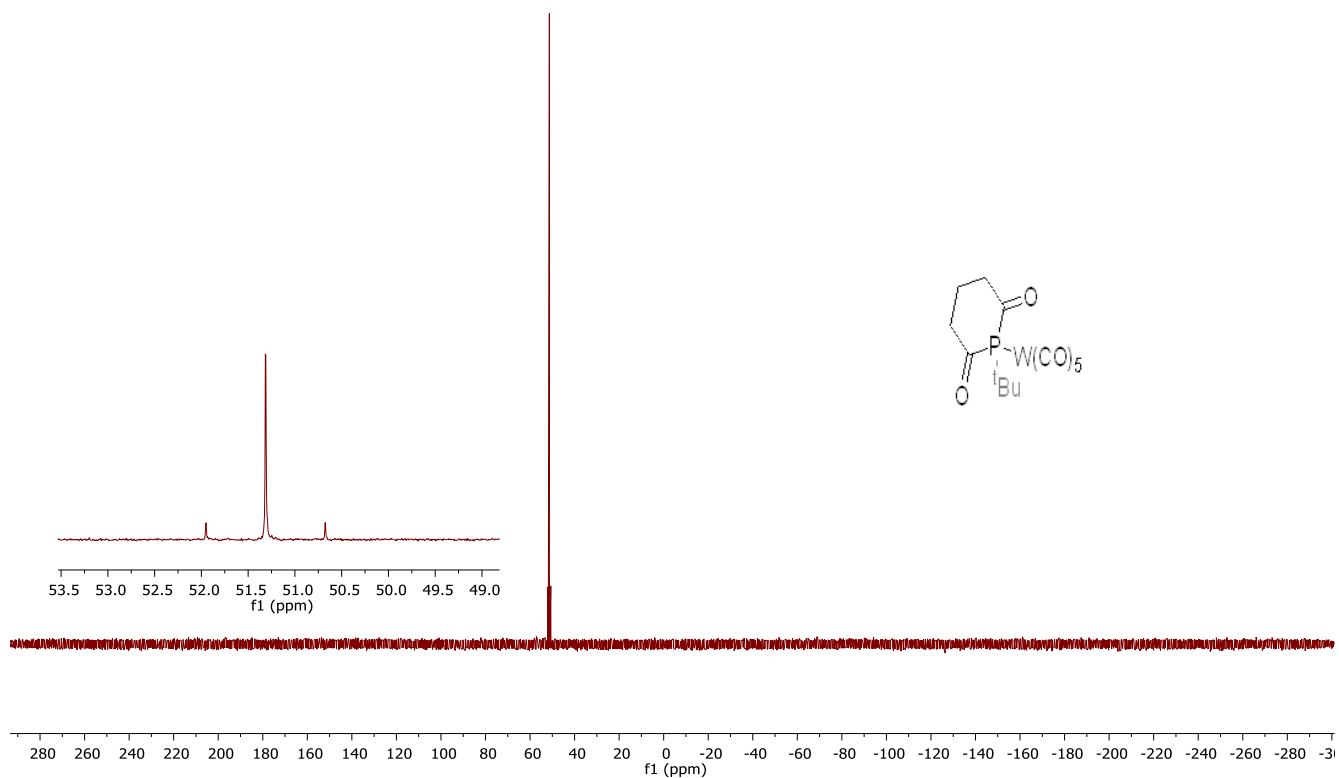








**Figure S68.**  $^1\text{H}$ - $^{13}\text{C}$  HMBC trace ( $\text{C}_6\text{D}_6$ , 303 K, 399.49, 100.46 MHz) for Compound **5b**.



**Figure S69.**  $^{31}\text{P}\{^1\text{H}\}$  NMR Spectrum ( $\text{C}_6\text{D}_6$ , 303 K, 161.72 MHz) for compound **5c**.

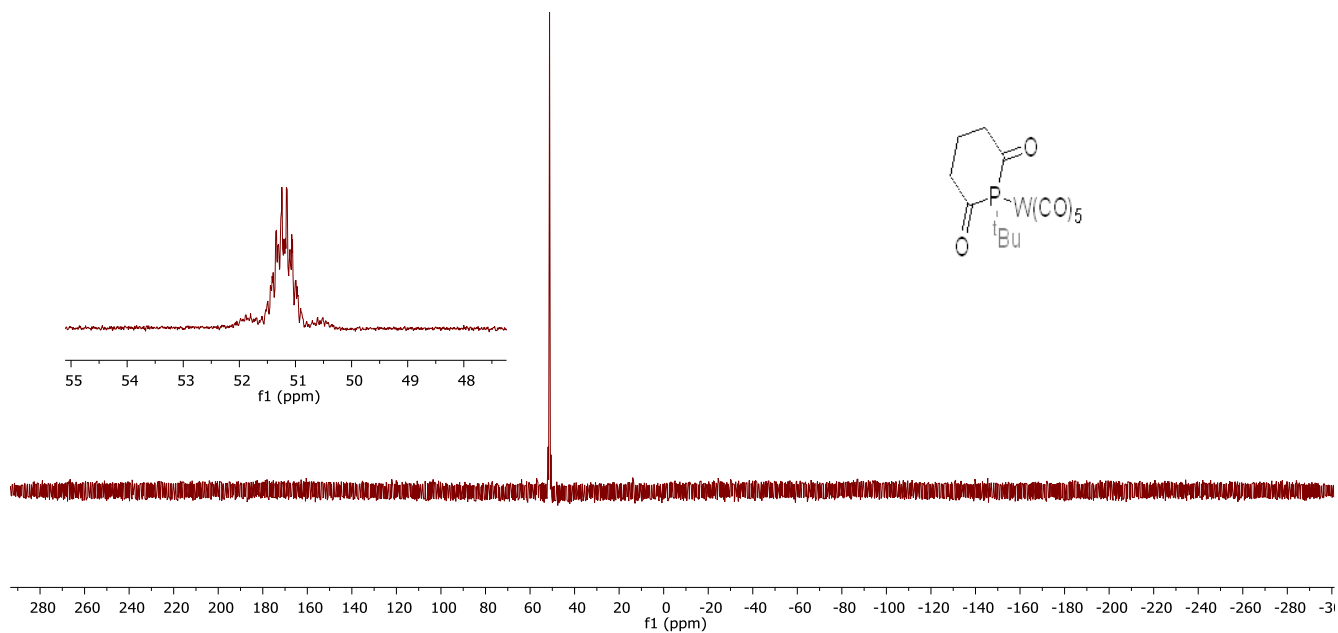


Figure S70.  $^{31}\text{P}$  NMR Spectrum ( $\text{C}_6\text{D}_6$ , 303 K, 161.72 MHz) for compound 5c.

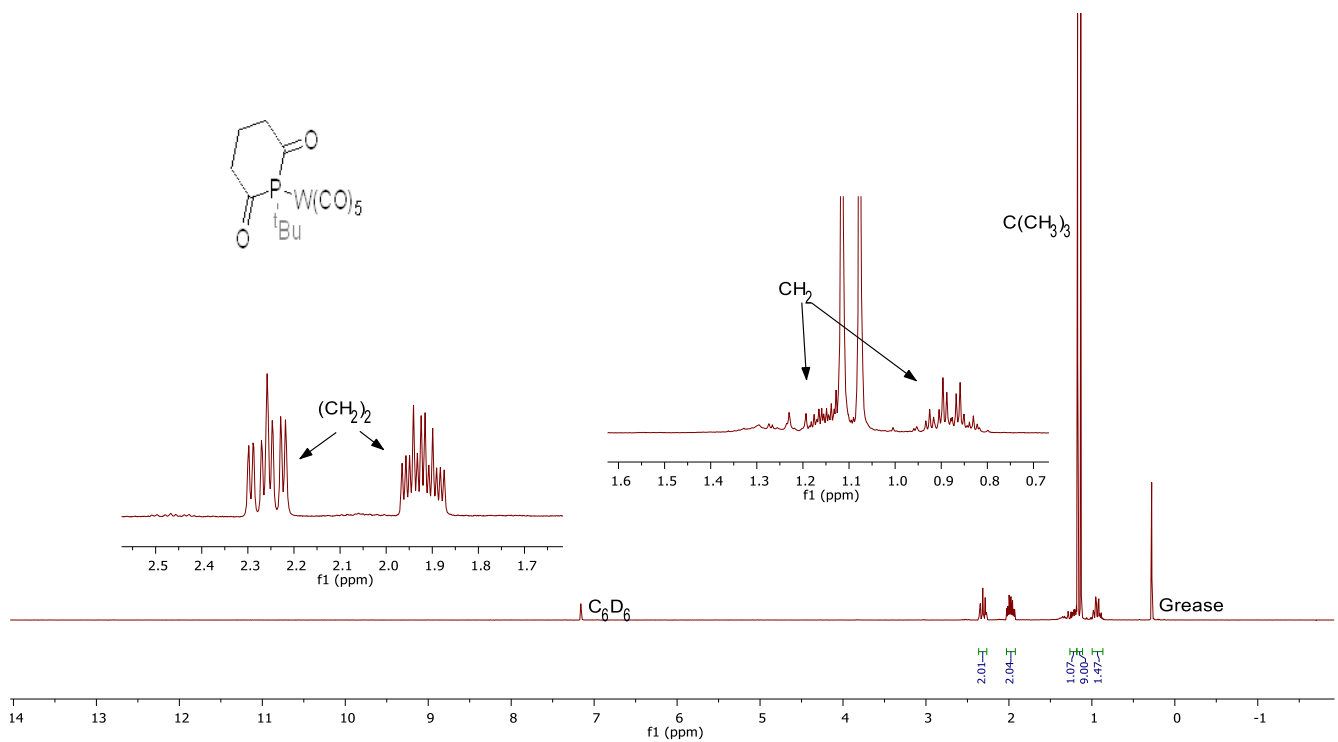
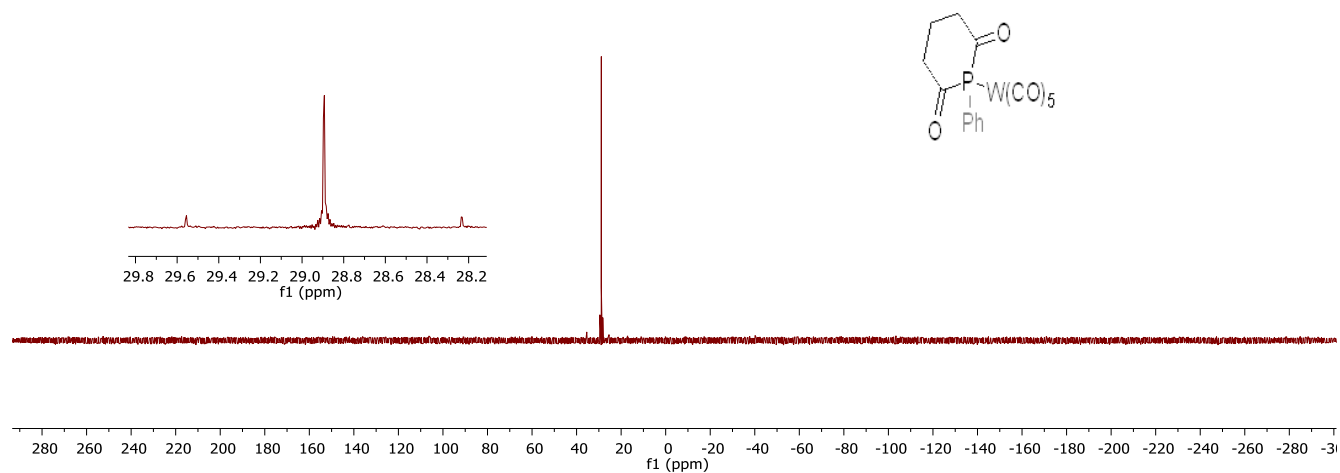
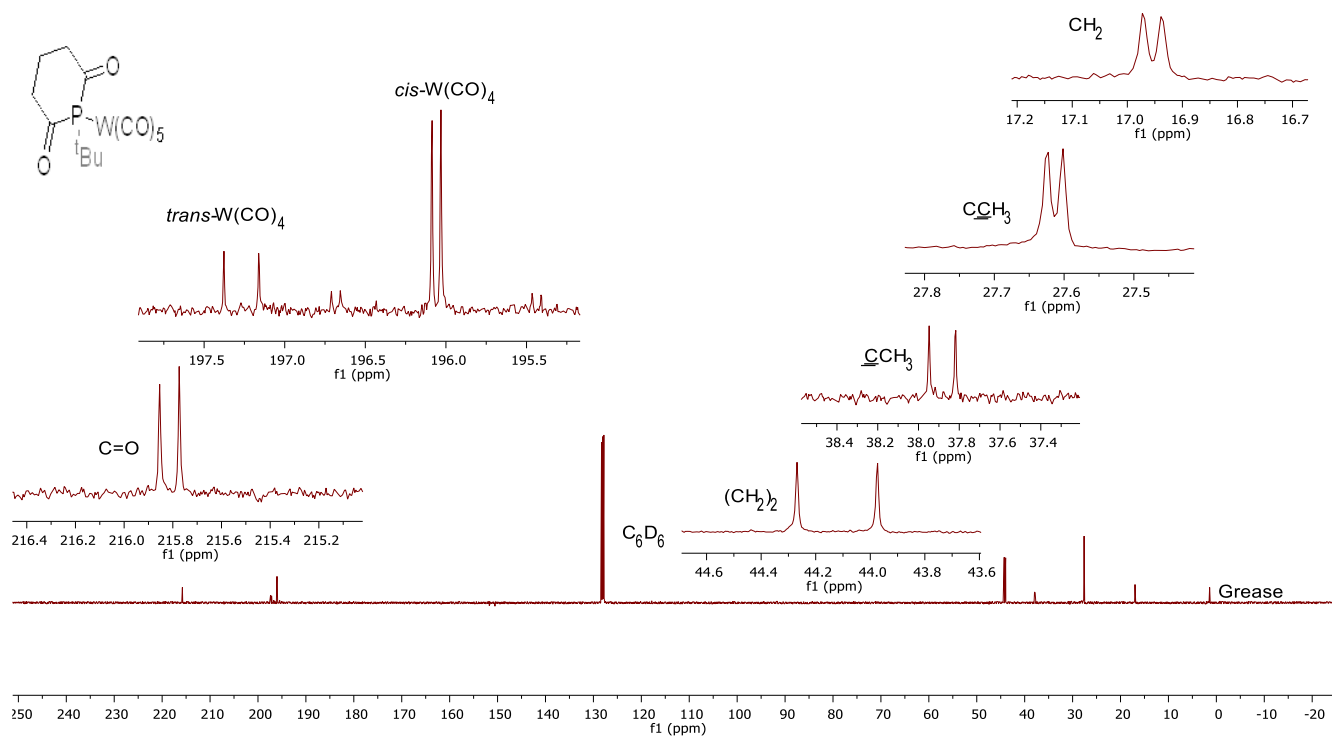


Figure S71.  $^1\text{H}$  NMR Spectrum ( $\text{C}_6\text{D}_6$ , 303 K, 399.49 MHz) for compound 5c.



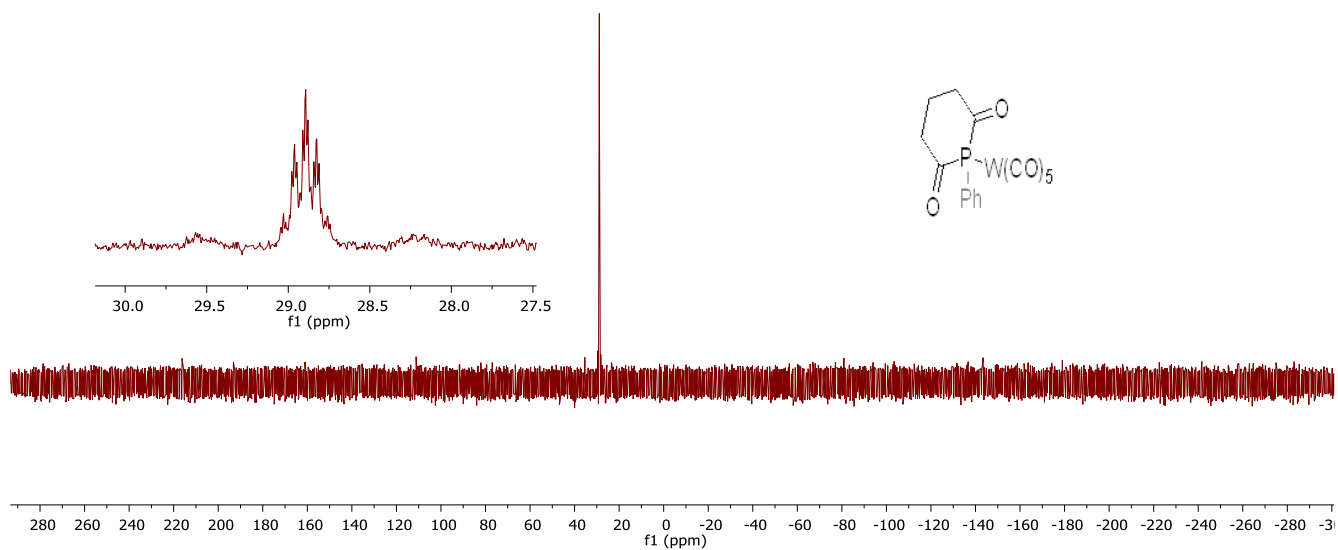


Figure S74.  $^{31}\text{P}$  NMR Spectrum ( $\text{C}_6\text{D}_6$ , 303 K, 161.72 MHz) for compound 5d.

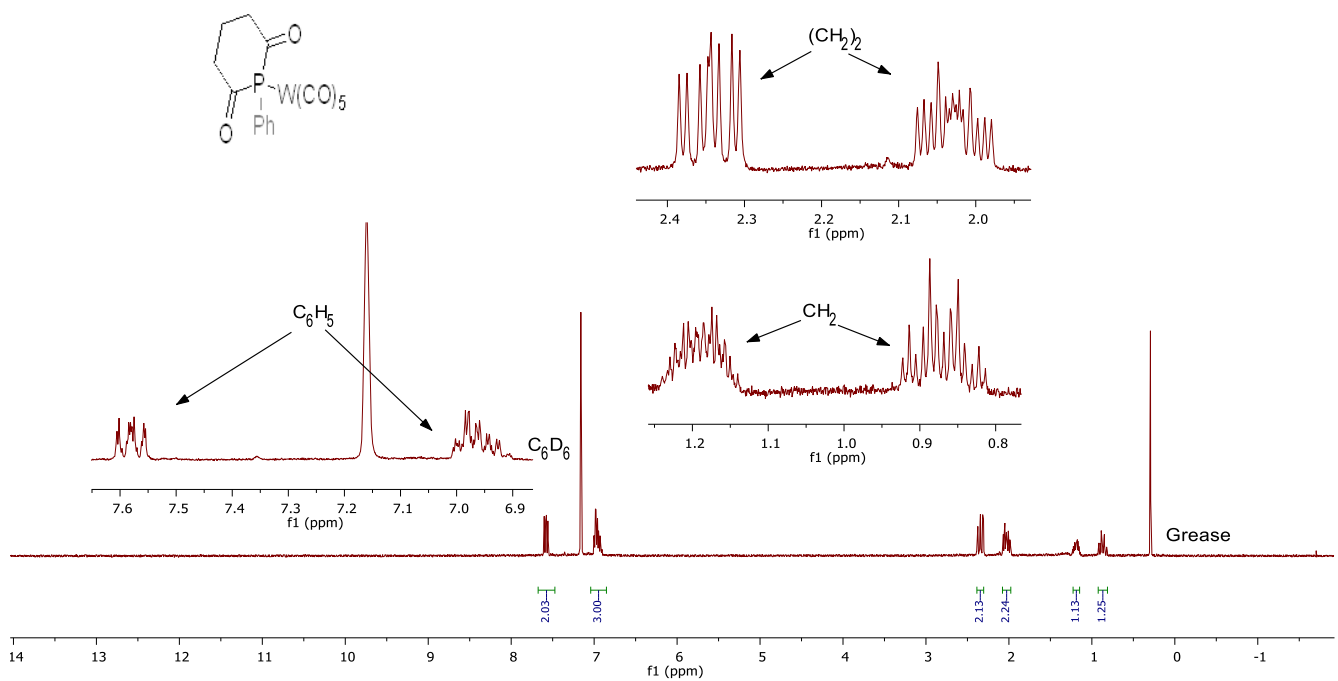
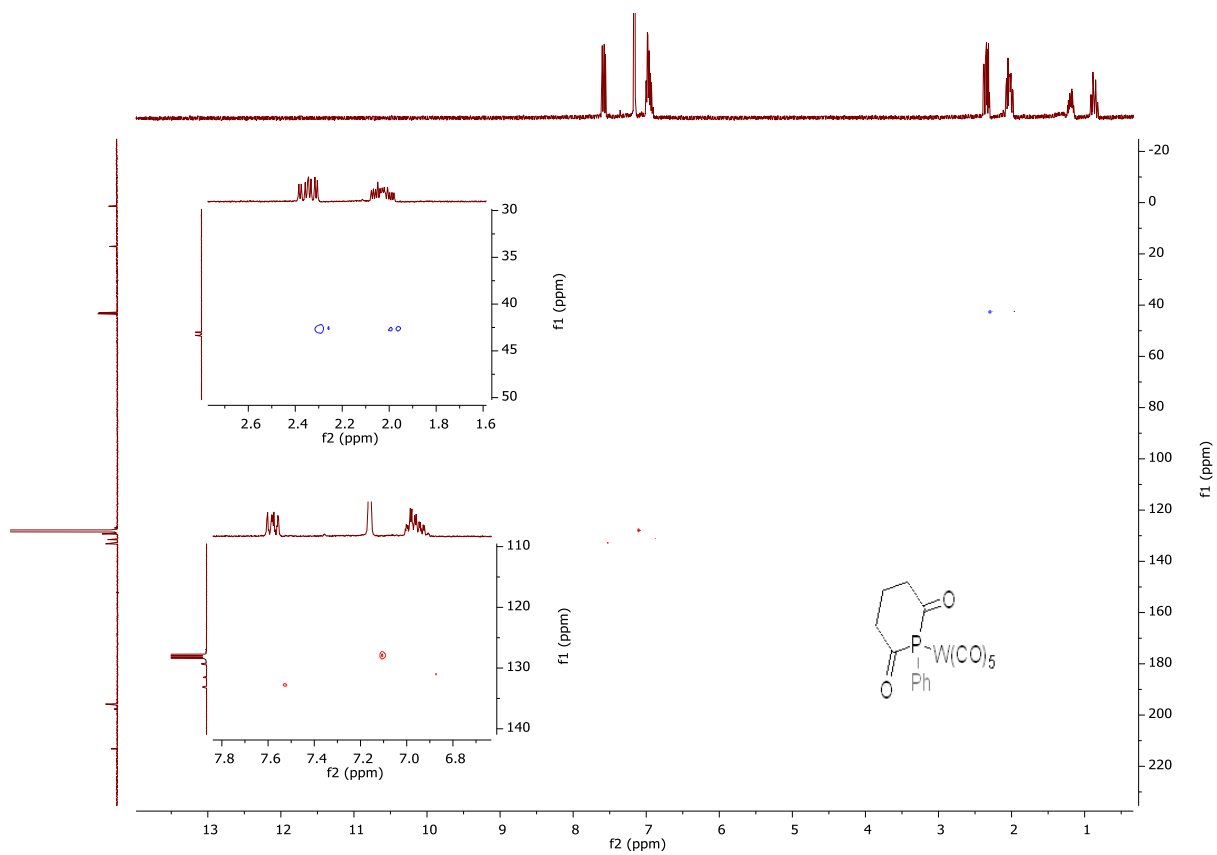
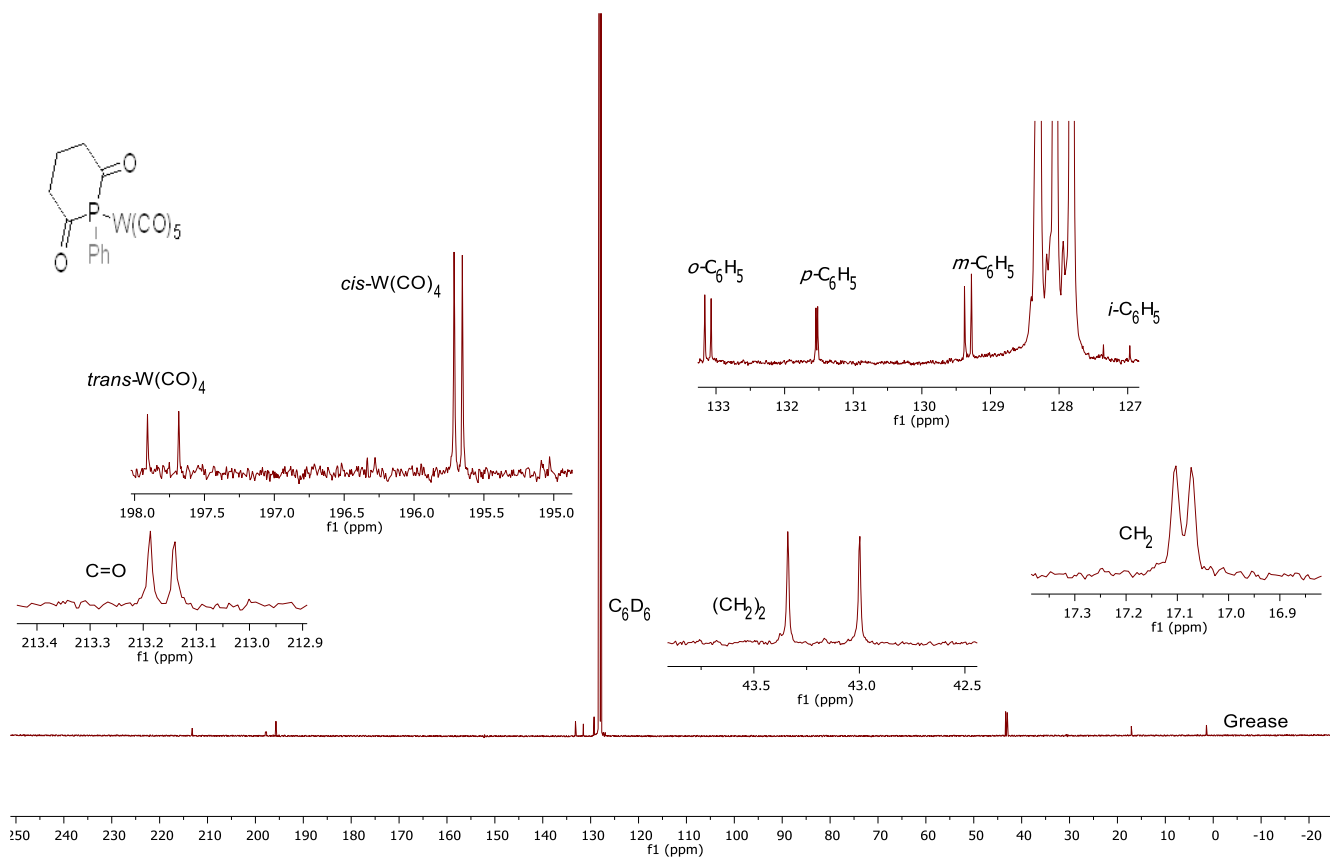


Figure S75.  $^1\text{H}$  NMR Spectrum ( $\text{C}_6\text{D}_6$ , 303 K, 399.49 MHz) for compound 5d.





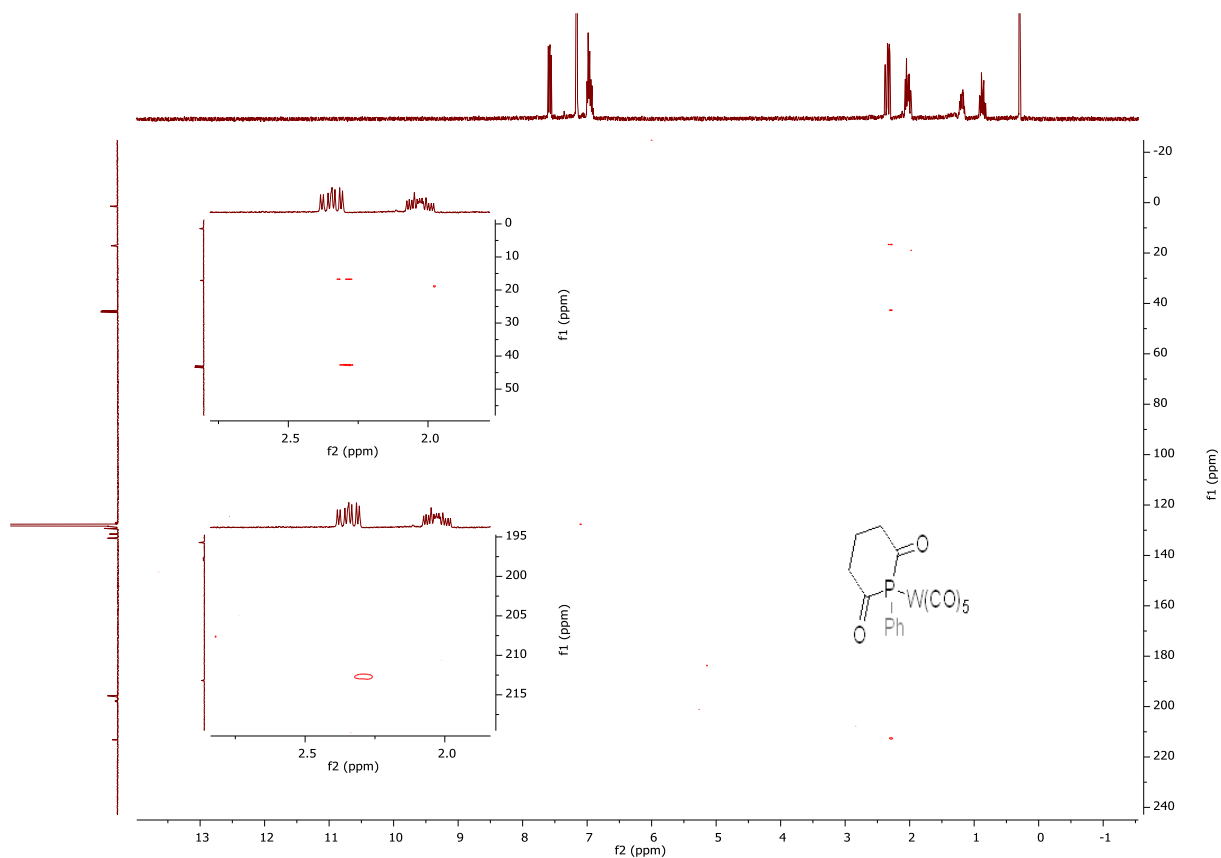


Figure S78.  $^1\text{H}$ - $^{13}\text{C}$  HMBC trace ( $\text{C}_6\text{D}_6$ , 303 K, 399.49, 100.46 MHz) for Compound 5d.

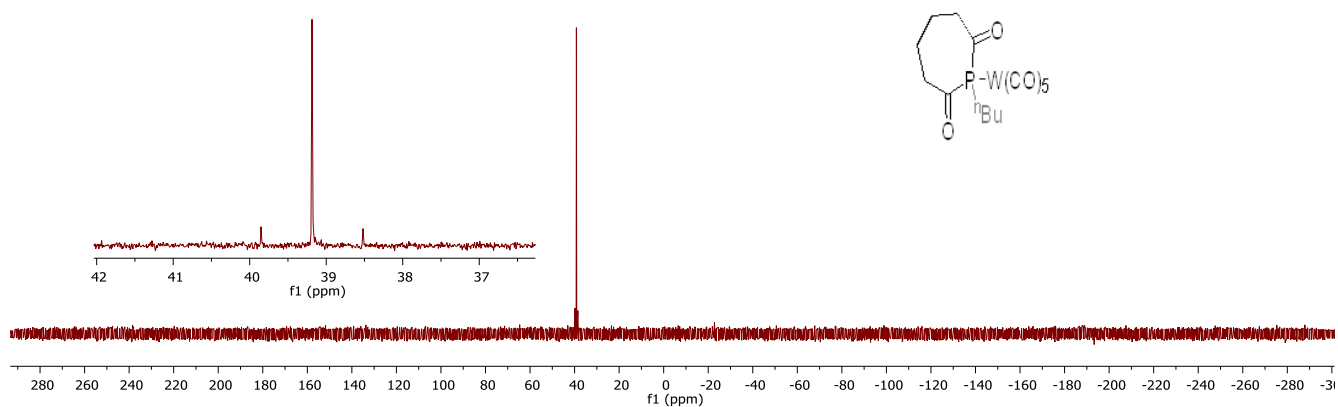
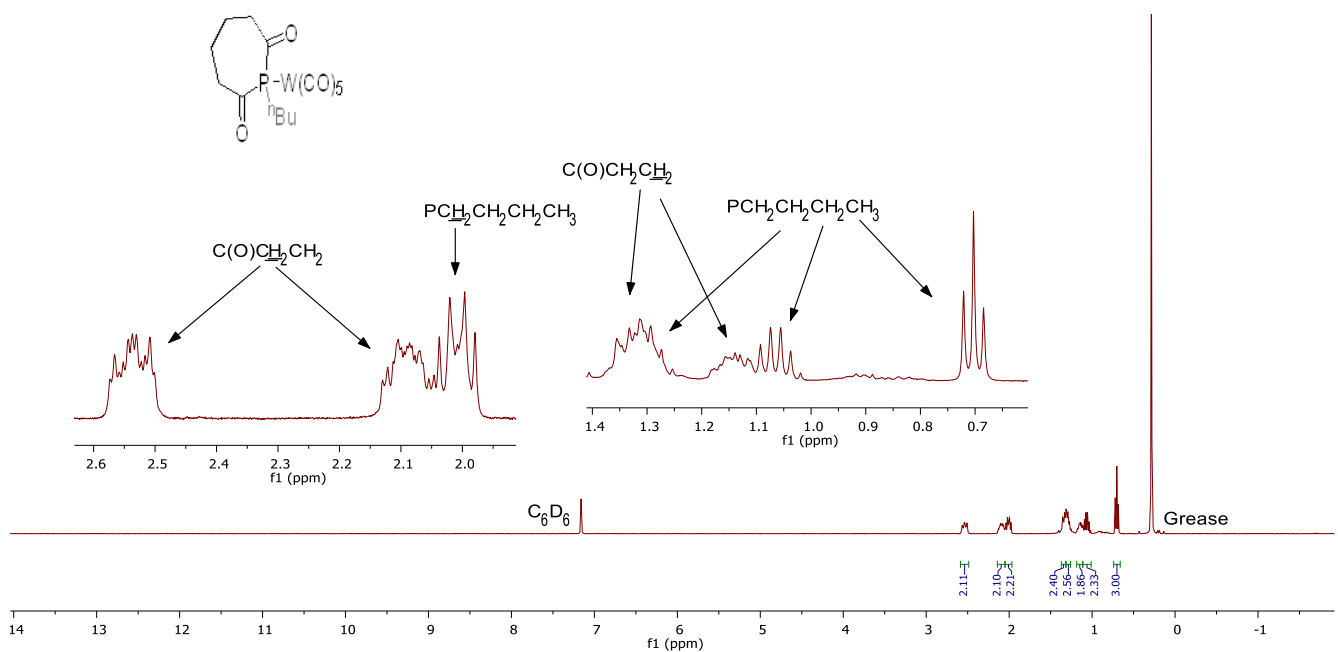
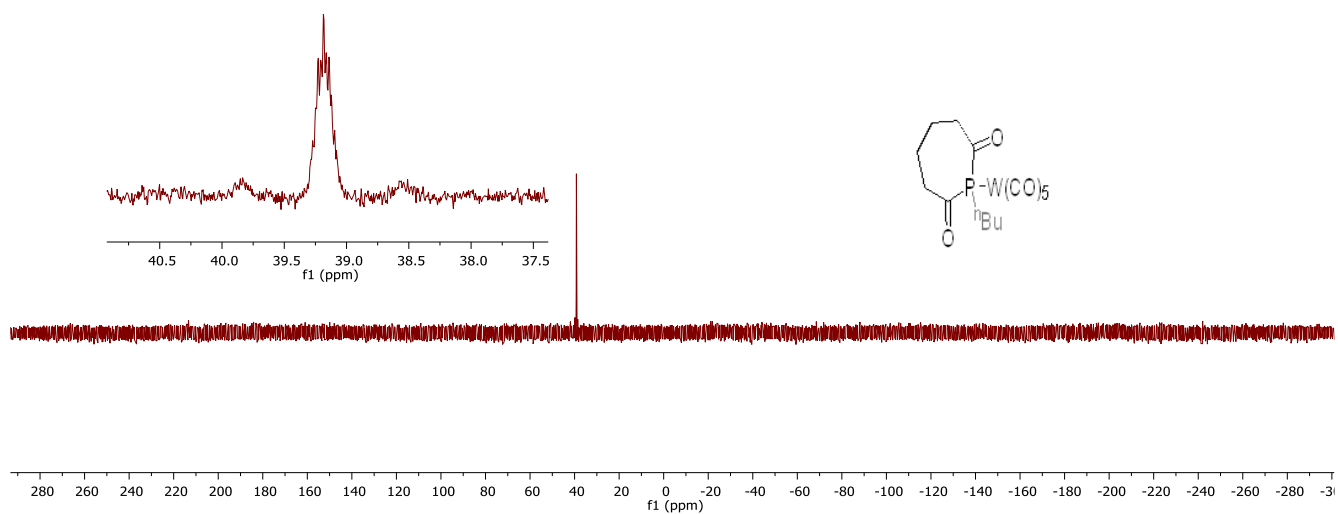
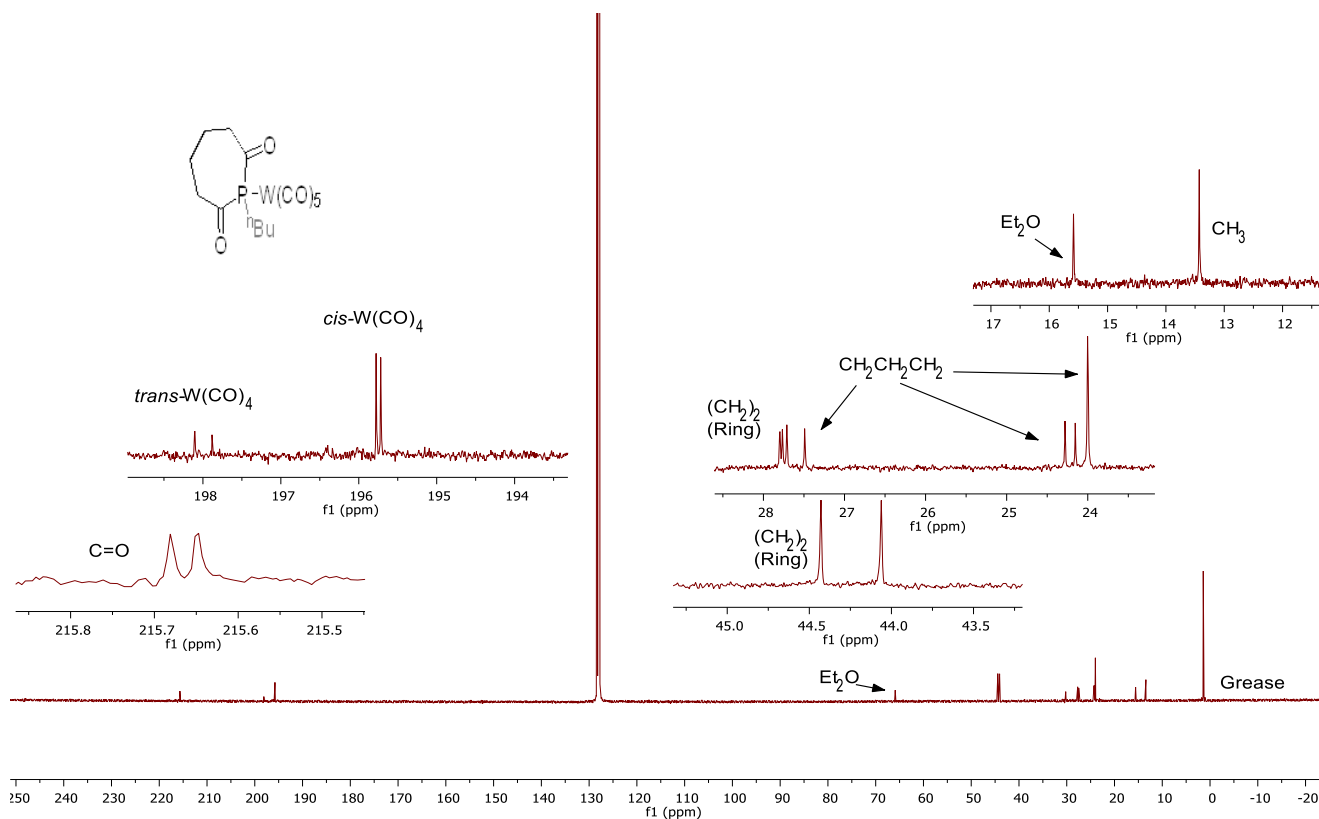
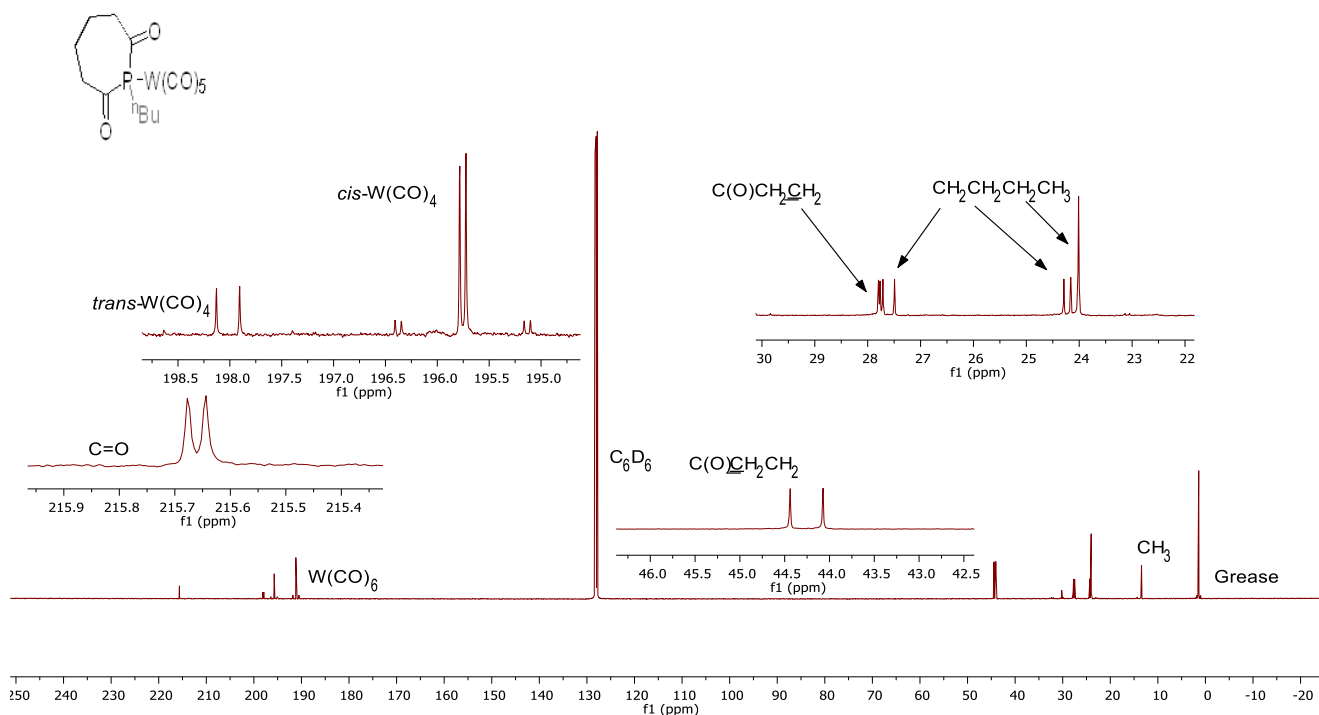
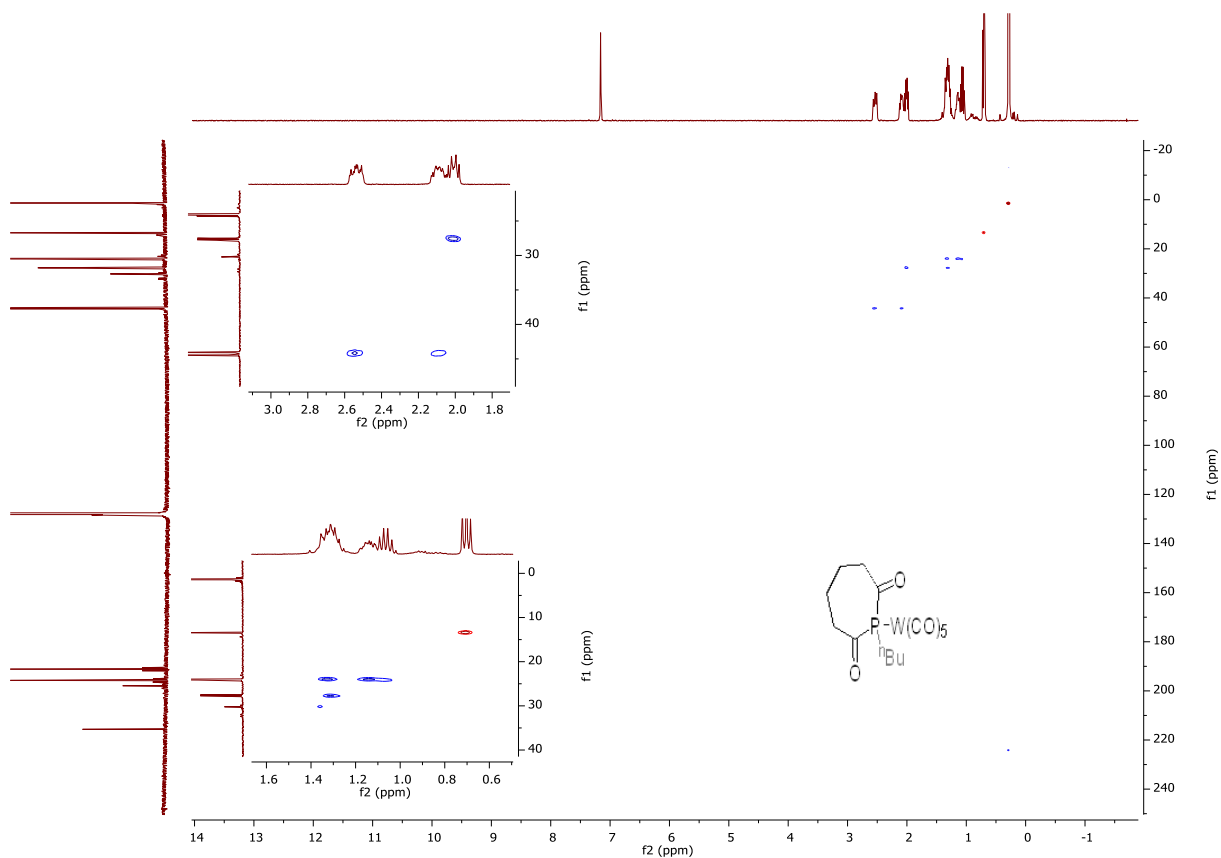


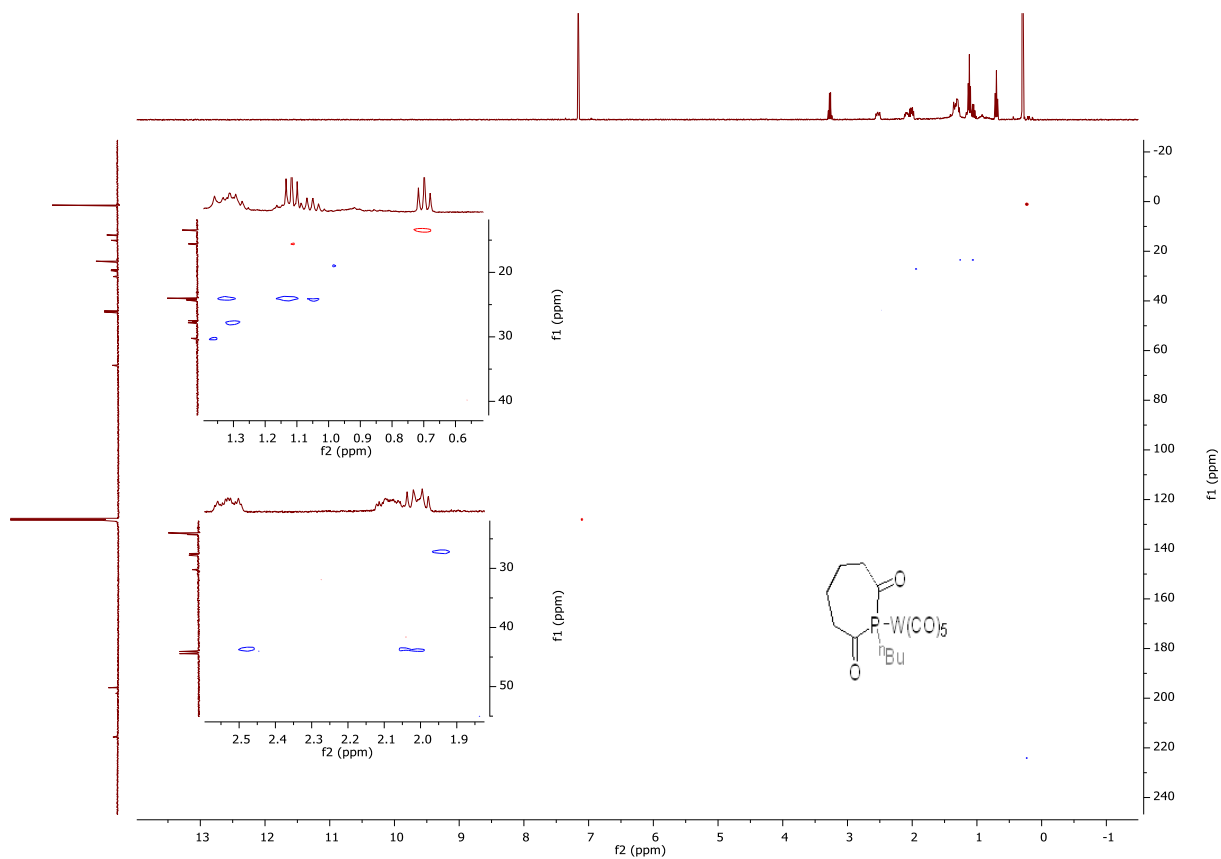
Figure S79.  $^{31}\text{P}\{^1\text{H}\}$  NMR Spectrum ( $\text{C}_6\text{D}_6$ , 303 K, 161.72 MHz) for compound 6.



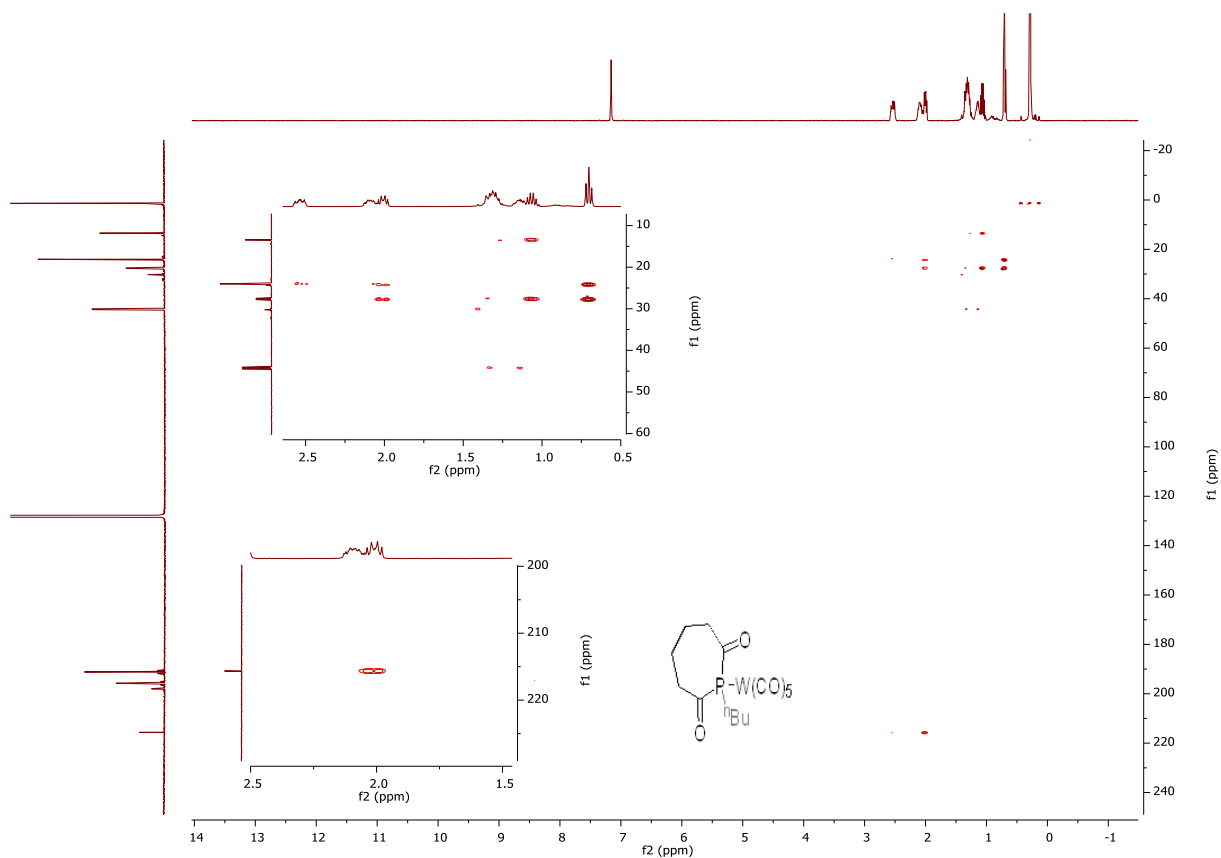




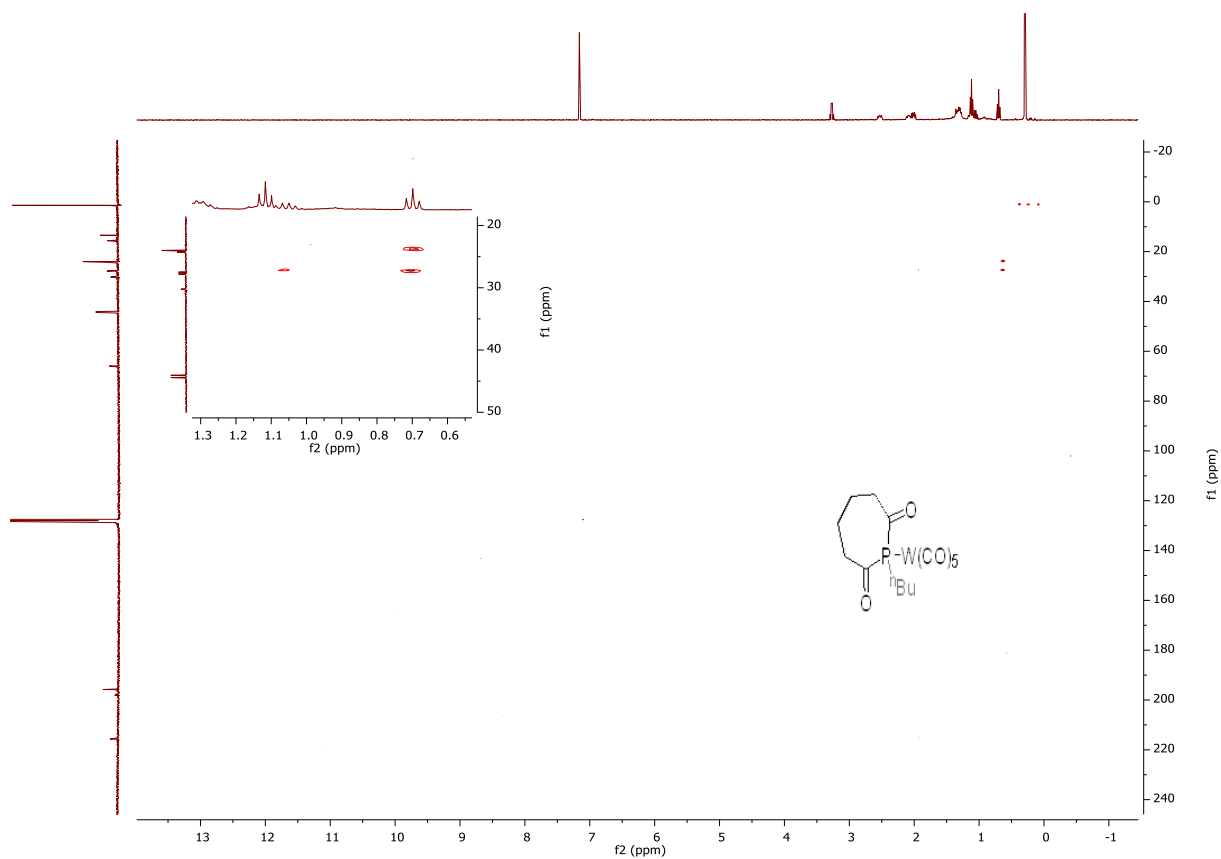
**Figure S84.**  $^1\text{H}$ - $^{13}\text{C}$  HSQC trace ( $\text{C}_6\text{D}_6$ , 303 K, 399.49, 100.46 MHz) for Compound **6**,  $\text{W}(\text{CO})_6$  present.



**Figure S85.**  $^1\text{H}$ - $^{13}\text{C}$  HSQC trace ( $\text{C}_6\text{D}_6$ , 303 K, 399.49, 100.46 MHz) for Compound **6**,  $\text{W}(\text{CO})_6$  removed.



**Figure S86.**  $^1\text{H}$ - $^{13}\text{C}$  HMBC trace ( $\text{C}_6\text{D}_6$ , 303 K, 399.49, 100.46 MHz) for Compound **6**,  $\text{W}(\text{CO})_6$  present.



**Figure S87.**  $^1\text{H}$ - $^{13}\text{C}$  HMBC trace ( $\text{C}_6\text{D}_6$ , 303 K, 399.49, 100.46 MHz) for Compound **6**,  $\text{W}(\text{CO})_6$  removed.

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Single Mass Analysis

Tolerance = 70.0 PPM / DBE: min = -1.5, max = 50.0

Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions

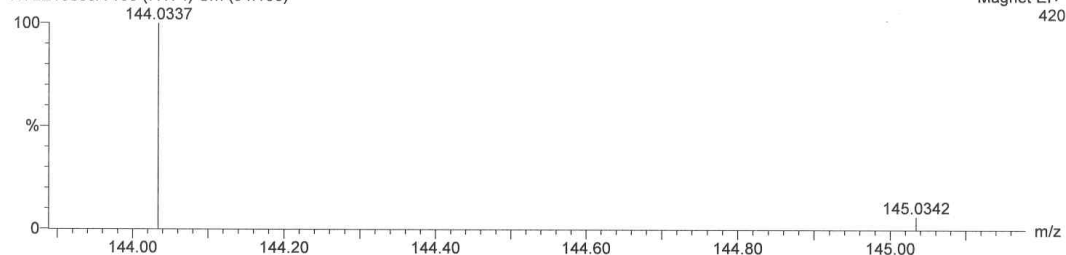
12 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 0-10 H: 0-1000 O: 0-2 P: 0-1

KGP-160

KYLE10885A 100 (7.174) Cm (94:106)



Minimum: -1.5  
Maximum: 5.0 70.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
144.0337	144.0340	-0.3	-2.1	3.0	2773013.8	C6 H9 O2 P

Figure S88. HRMS-EI for Compound 2a.

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Single Mass Analysis

Tolerance = 50.0 PPM / DBE: min = -1.5, max = 50.0

Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions

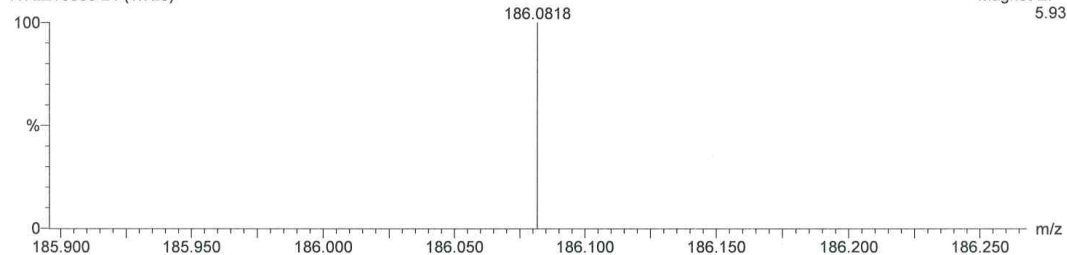
7 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 0-9 H: 0-1000 O: 0-2 P: 0-1

KGP-199

KYLE10883 24 (1.723)



Minimum: -1.5  
Maximum: 5.0 50.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
186.0818	186.0810	0.8	4.3	3.0	5546025.5	C9 H15 O2 P

Figure S89. HRMS-EI for Compound 2b.

## Elemental Composition Report

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### Single Mass Analysis

Tolerance = 50.0 PPM / DBE: min = -1.5, max = 50.0

Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions

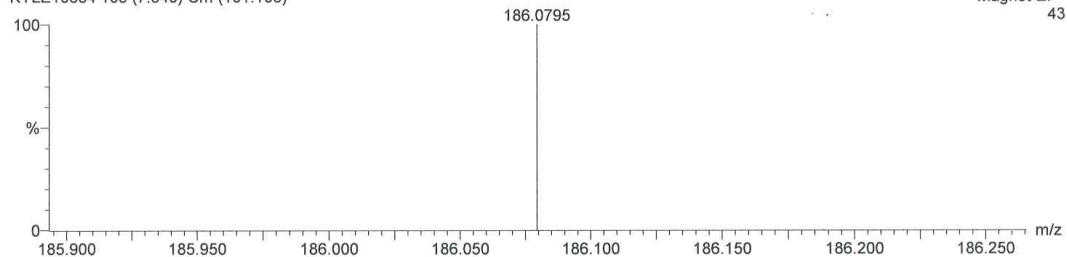
7 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 0-9 H: 0-1000 O: 0-2 P: 0-1

KGP-181

KYLE10884 105 (7.540) Cm (101:105)



Minimum:				-1.5		
Maximum:	5.0	50.0	50.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
186.0795	186.0810	-1.5	-8.1	3.0	5546029.5	C9 H15 O2 P

Figure S90. HRMS-El for Compound 2c.

## Elemental Composition Report

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### Single Mass Analysis

Tolerance = 50.0 PPM / DBE: min = -1.5, max = 50.0

Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions

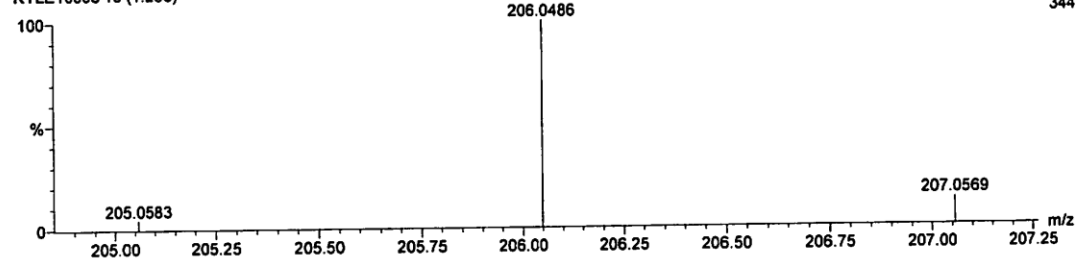
9 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 0-12 H: 0-1000 O: 0-2 P: 0-1

KGP-198

KYLE10368 16 (1.253)



Minimum:				-1.5		
Maximum:	5.0	50.0	50.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
206.0486	206.0497	-1.1	-5.3	7.0	2773016.5	C11 H11 O2 P

Figure S91. HRMS-El for Compound 2d.

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Single Mass Analysis

Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0

Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions

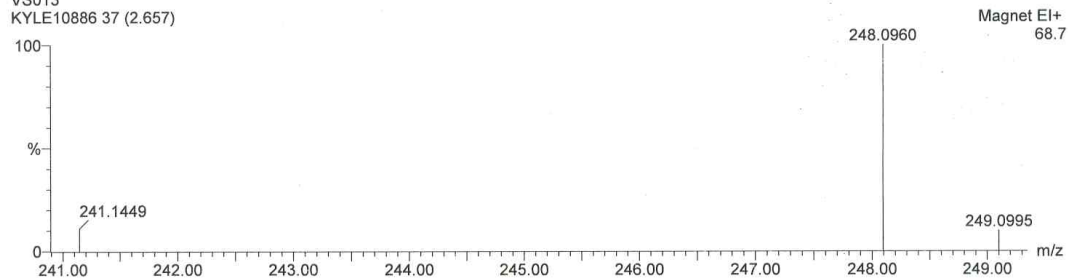
7 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 0-14 H: 0-1000 O: 0-2 P: 0-1

VS013

KYLE10886 37 (2.657)



Minimum: -1.5  
Maximum: 5.0 10.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
248.0960	248.0966	-0.6	-2.4	7.0	2773013.5	C14 H17 O2 P

Figure S92. HRMS-EI for Compound 2e.

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Single Mass Analysis

Tolerance = 50.0 PPM / DBE: min = -1.5, max = 50.0

Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions

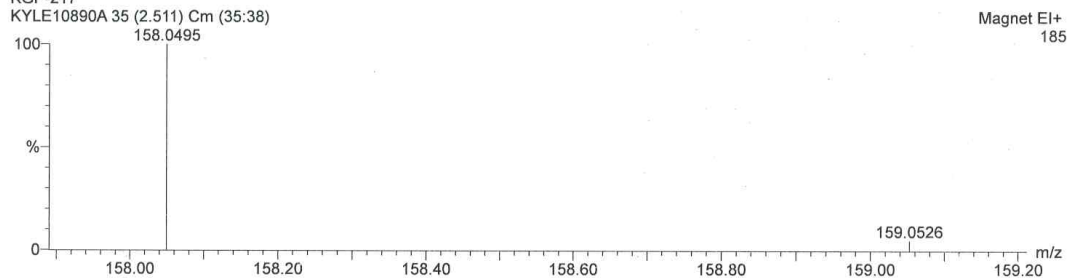
12 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 0-10 H: 0-1000 O: 0-2 P: 0-1

KGP-217

KYLE10890A 35 (2.511) Cm (35:38)



Minimum: -1.5  
Maximum: 5.0 50.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
158.0495	158.0497	-0.2	-1.3	3.0	2773014.3	C7 H11 O2 P

Figure S93. HRMS-EI for Compound 3a.



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### Single Mass Analysis

Tolerance = 10000.0 PPM / DBE: min = -1.5, max = 50.0

Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions

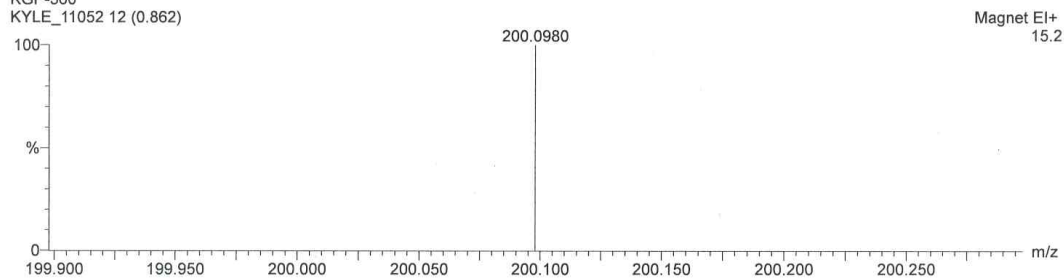
7 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 0-10 H: 0-1000 O: 0-2 P: 0-1

KGP-360

KYLE\_11052 12 (0.862)



Minimum:				-1.5			
Maximum:		5.0	10000.0	50.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula	
200.0980	200.0966	1.4	7.0	3.0	5310.1	C10 H17 O2 P	

Figure S94. HRMS-El for Compound 3b.

## Elemental Composition Report

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### Single Mass Analysis

Tolerance = 10000.0 PPM / DBE: min = -1.5, max = 50.0

Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions

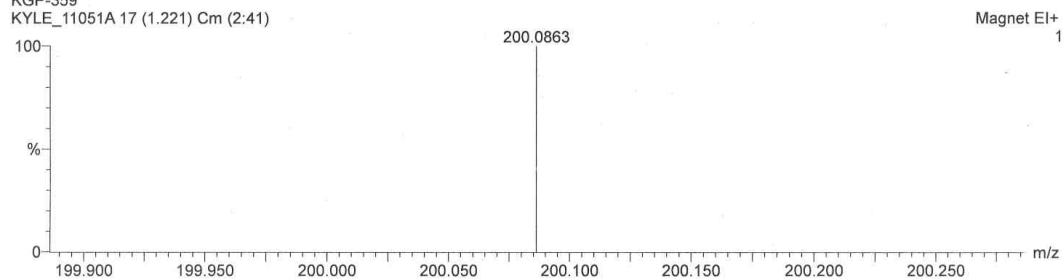
7 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 0-10 H: 0-1000 O: 0-2 P: 0-1

KGP-359

KYLE\_11051A 17 (1.221) Cm (2.41)



Minimum:				-1.5			
Maximum:		5.0	10000.0	50.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula	
200.0863	200.0966	-10.3	-51.5	3.0	353.6	C10 H17 O2 P	

Figure S95. HRMS-El for Compound 3c (very volatile, gave only one scan out of 3 samples, leading to higher error).

## Elemental Composition Report

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### Single Mass Analysis

Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0

Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions

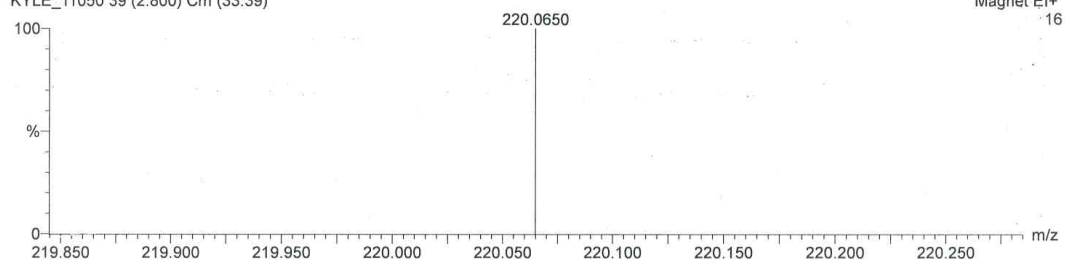
7 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 0-12 H: 0-1000 O: 0-2 P: 0-1

KGP-358

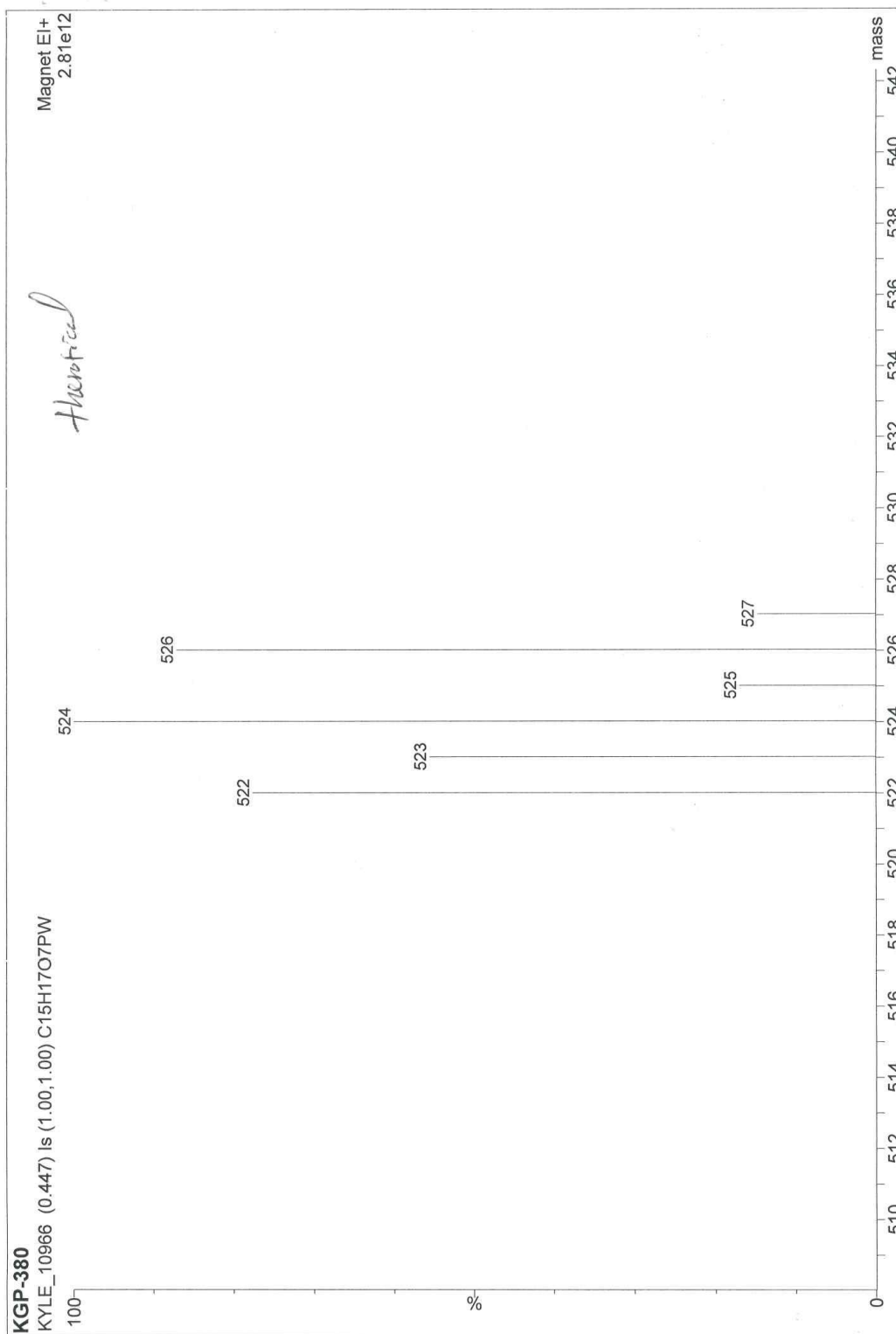
KYLE\_11050 39 (2.800) Cm (33:39)



Minimum: -1.5  
Maximum: 5.0 10.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
220.0650	220.0653	-0.3	-1.4	7.0	5546026.0	C12 H13 O2 P

**Figure S96. HRMS-EI for Compound 3d.**



**Figure S97.** LRMS-EI Theoretical Isotope Distribution for Compound 6.

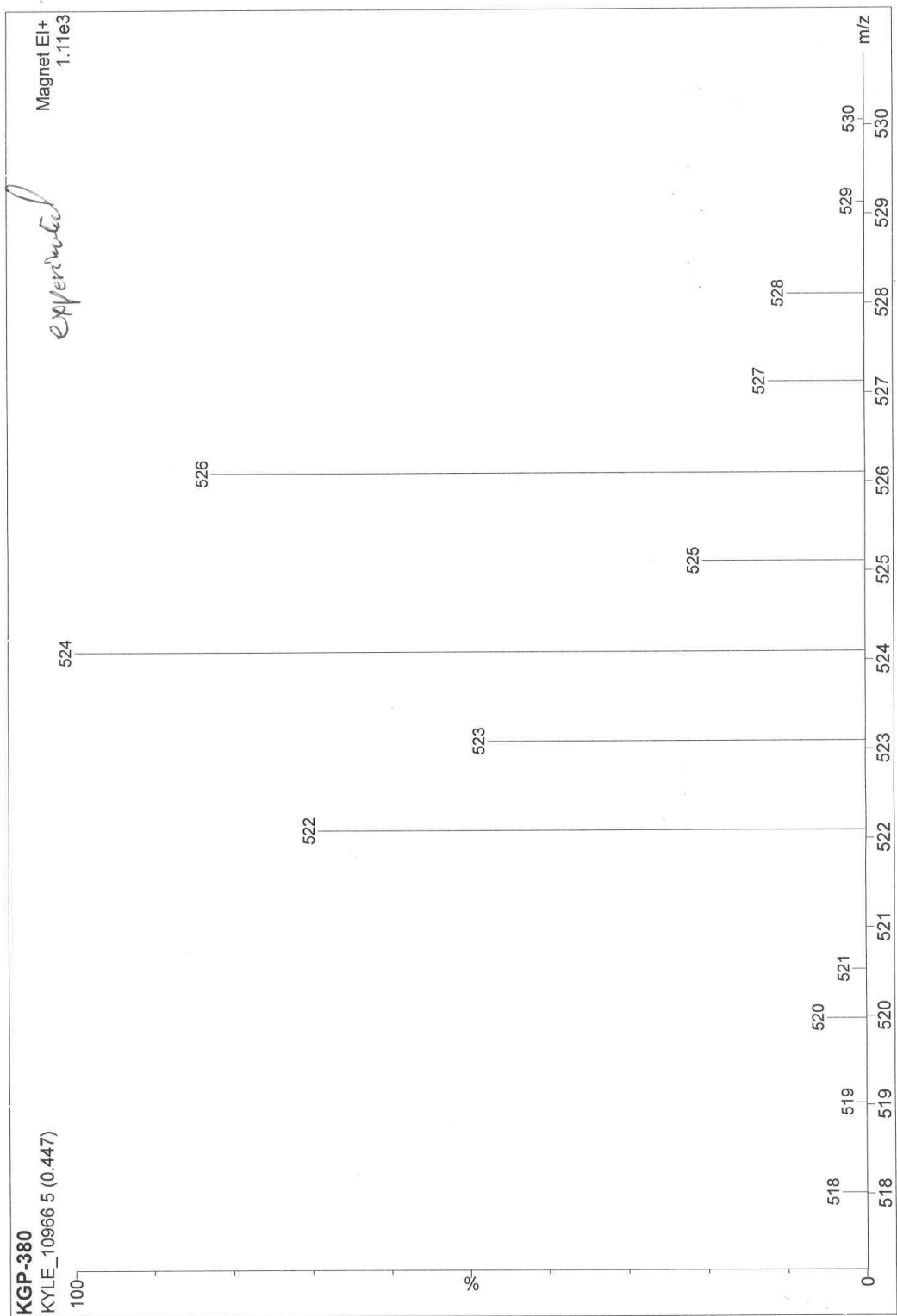


Figure S98. LRMS-EI for Compound 6.