Electronic Supplementary Information

Reactions of Coordinated Hydroxymethylphosphines with NH-Functional Amines: The Phosphorus Lone Pair is Crucial for the Phosphorus Mannich Reaction

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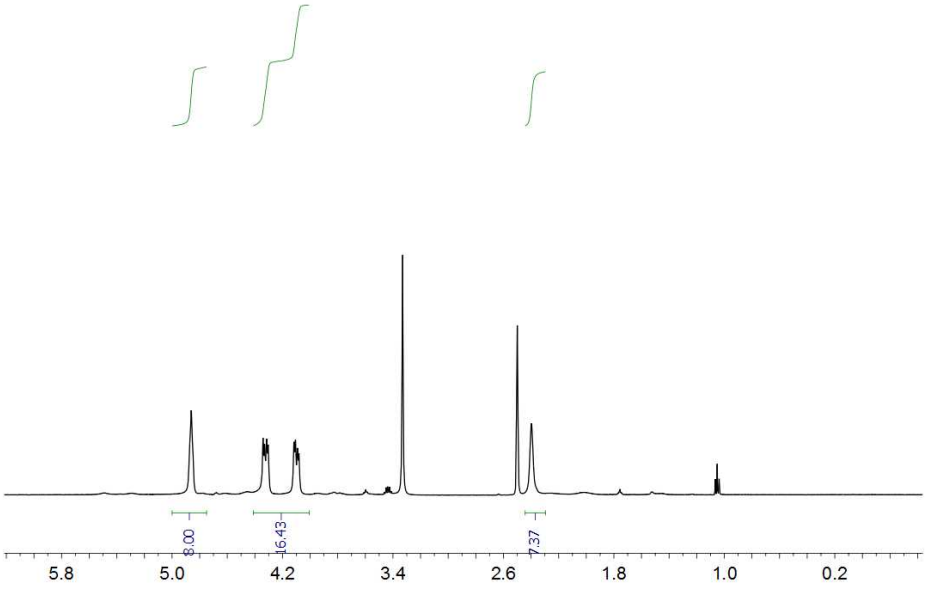
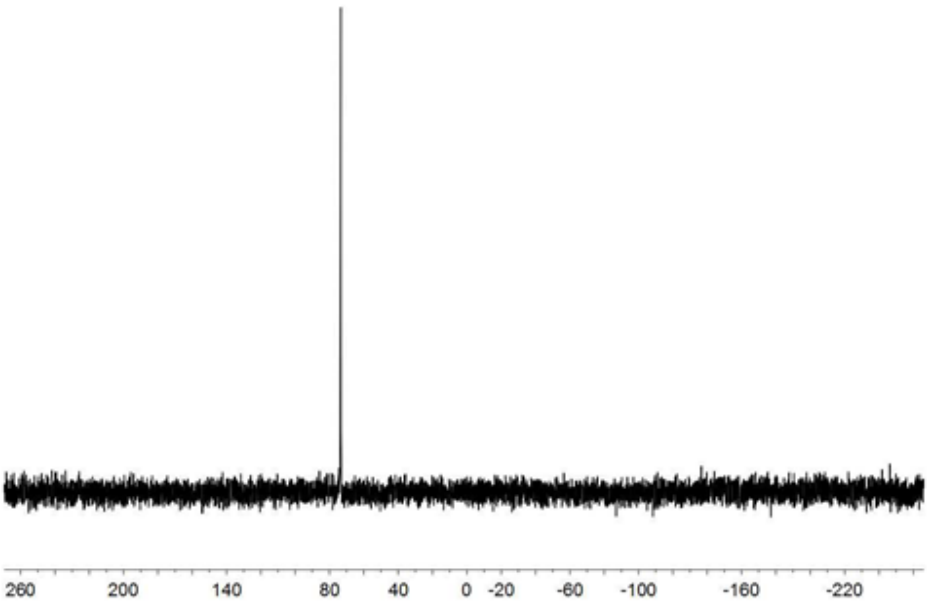
^{1}H NMR Spectrum of *trans*-Fe(DHMPE)$_2$Cl$_2$ (DMSO-d_6)



^{31}P{^{1}H} NMR Spectrum of *trans*-Fe(DHMPE)$_2$Cl$_2$ (DMSO-d_6)



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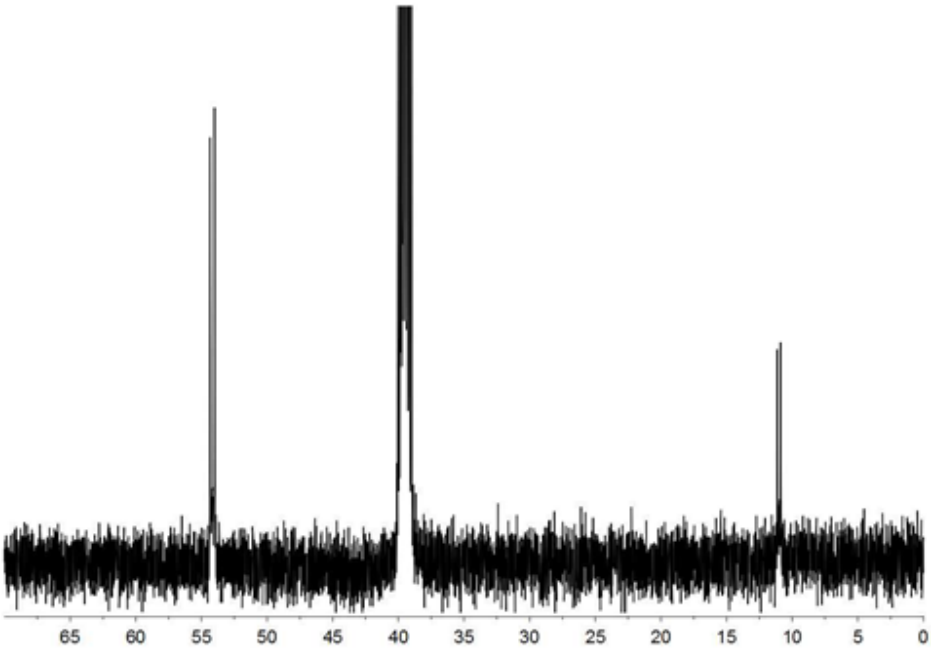
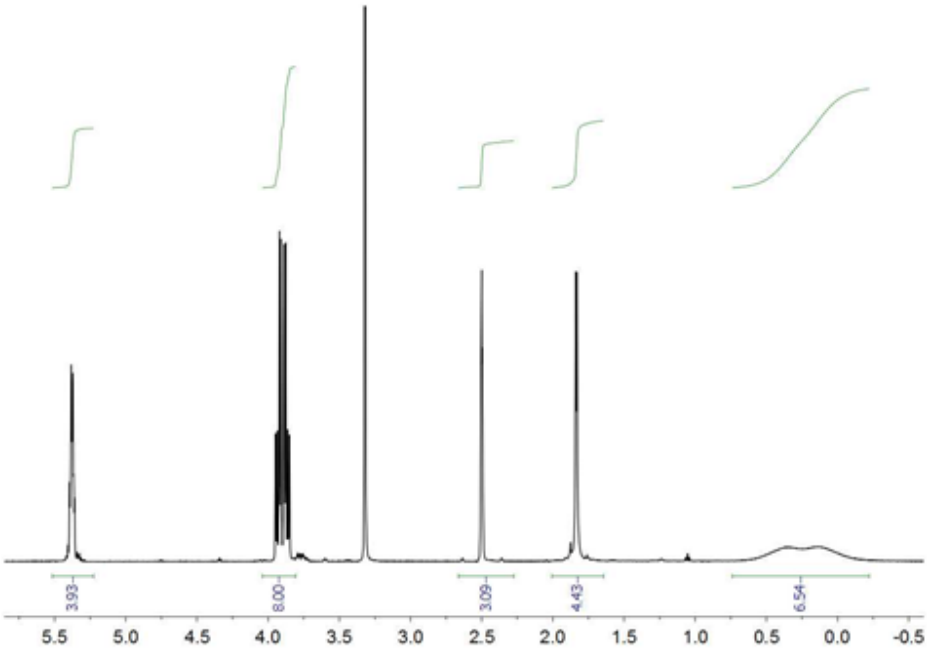
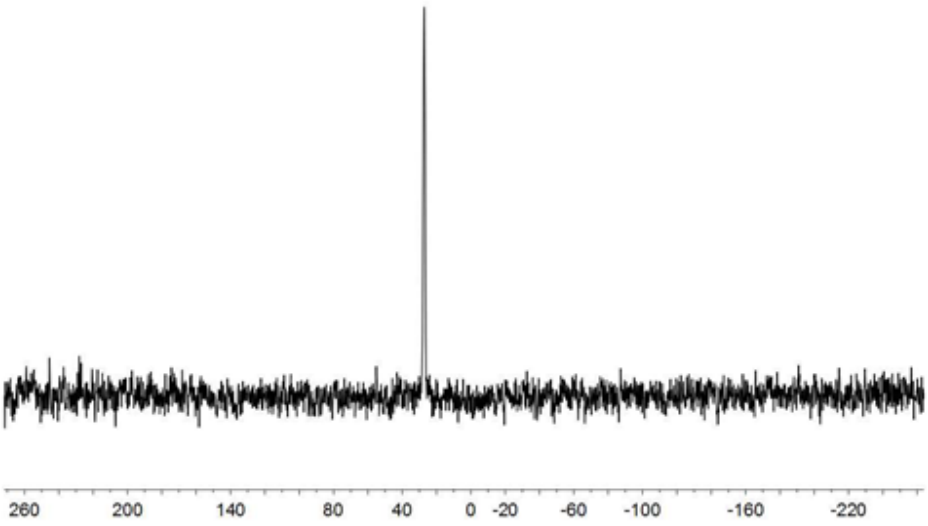


^{1}H NMR Spectrum of DHMPE·2BH$_3$ (DMSO-d_6)



^{31}P{^{1}H} NMR Spectrum of DHMPE·2BH$_3$ (DMSO-d_6)



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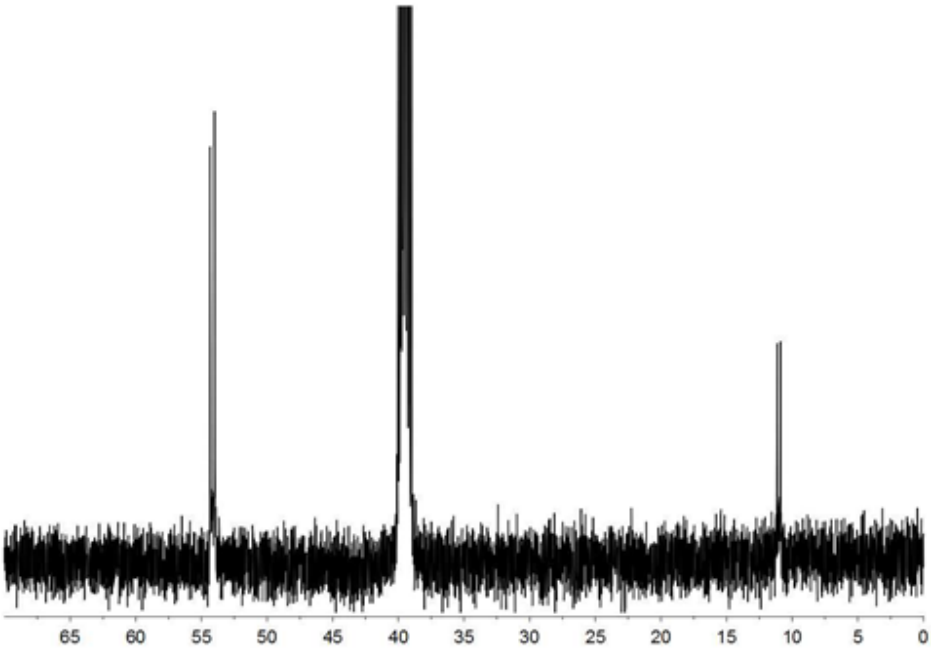
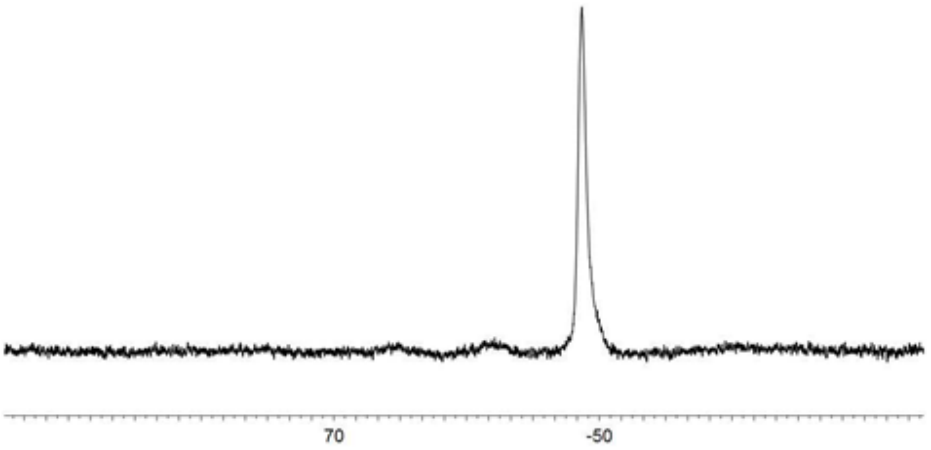
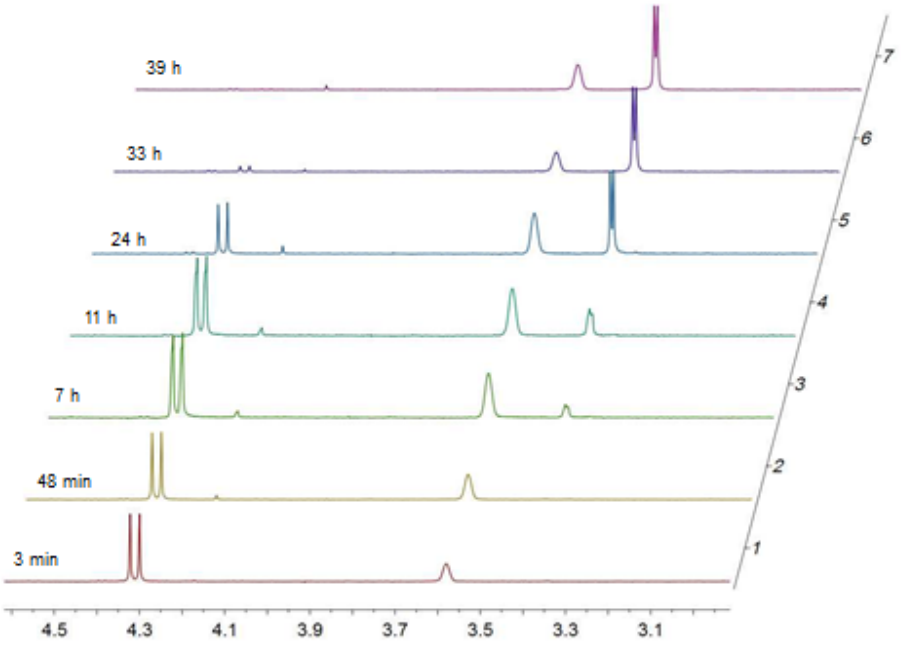


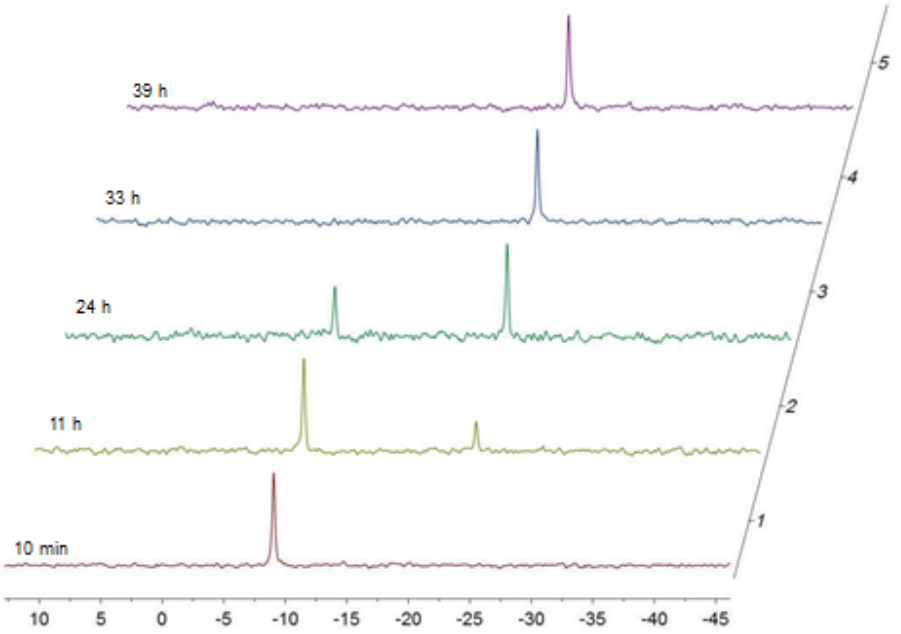
^{11}B NMR Spectrum of DHMPE·2BH$_3$ (DMSO-d_6)



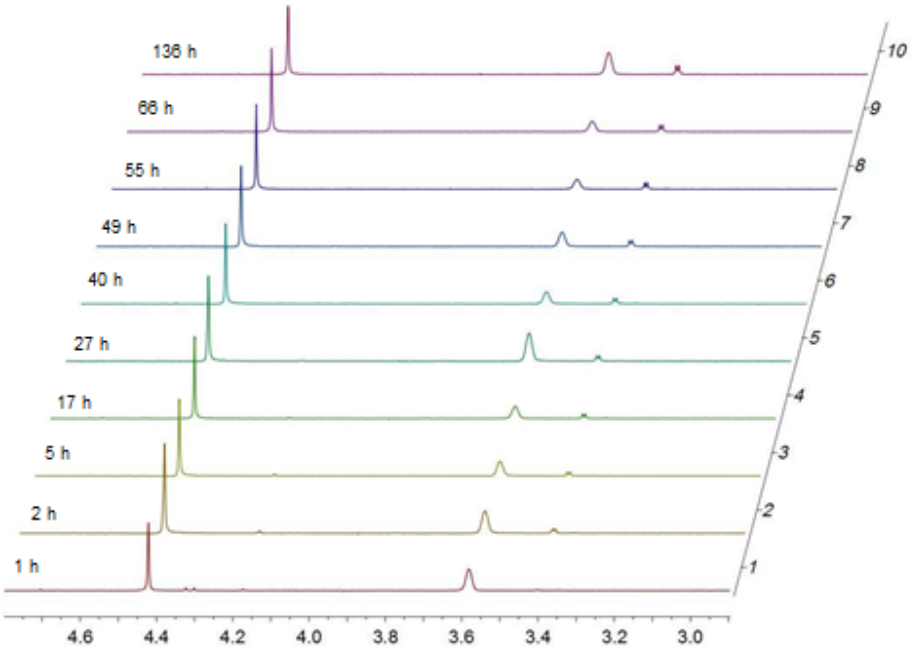
^{1}H NMR Spectra of Ph_2PCH_2OH + Et_2NH (ethanol-d_6)



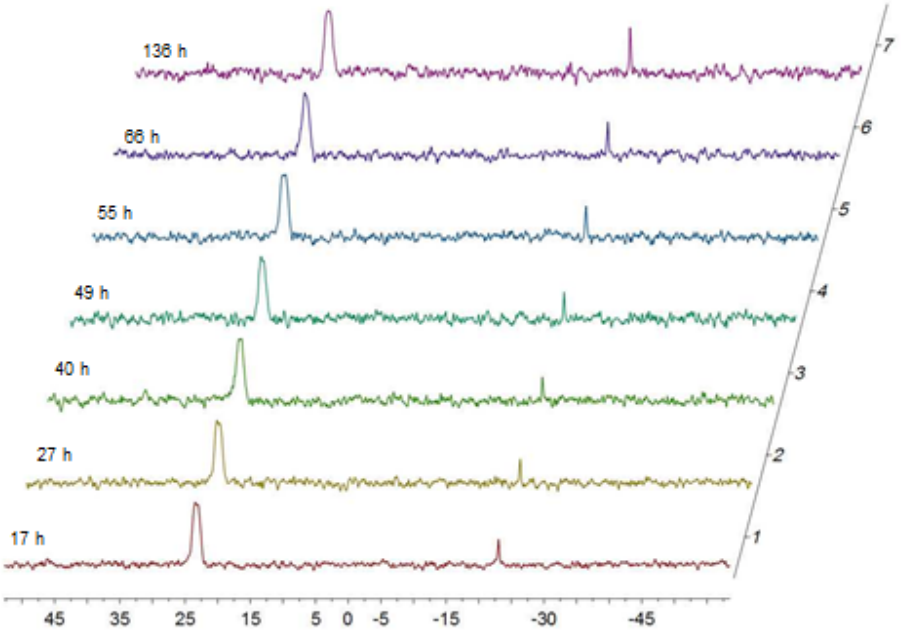
^{31}P{^{1}H} NMR Spectra of Ph_2PCH_2OH + Et_2NH (ethanol-d_6)



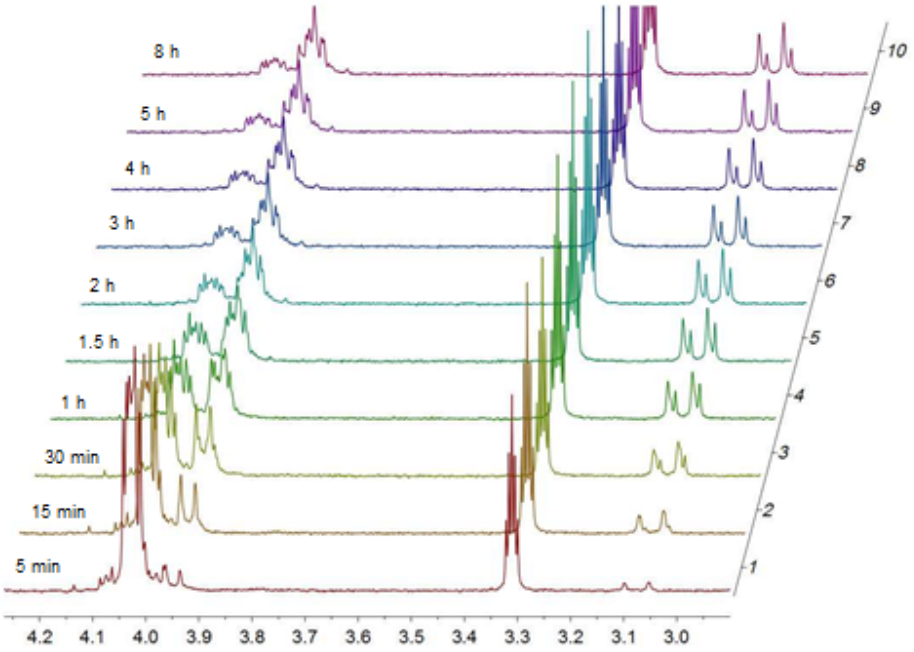
^{1}H NMR Spectra of $Ph_2PCH_2OH{\cdot}BH_3$ + Et_2NH (ethanol-d_6)



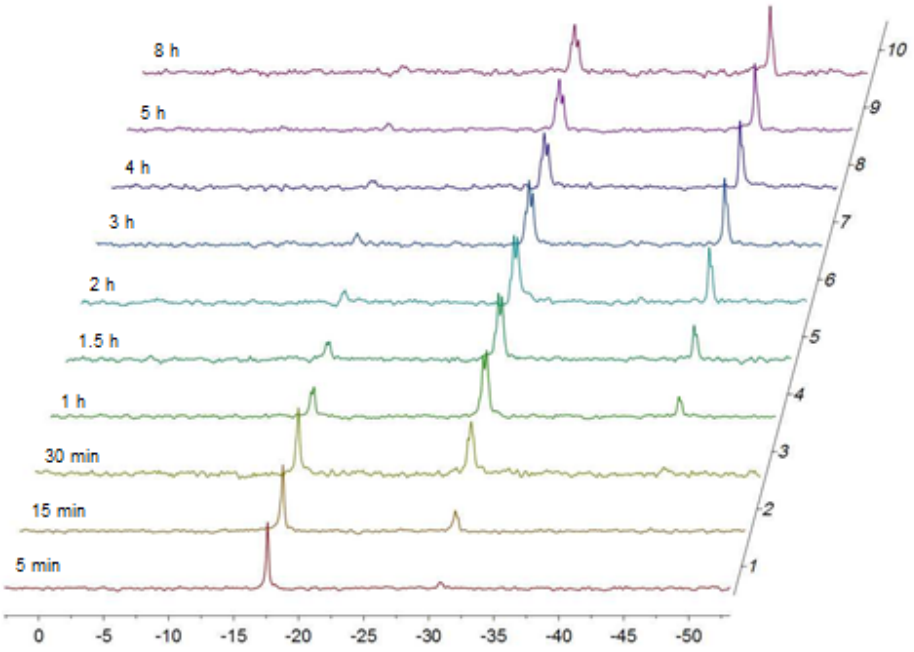
^{31}P{^{1}H} NMR Spectra of $Ph_2PCH_2OH{\cdot}BH_3$ + Et_2NH (ethanol-d_6)



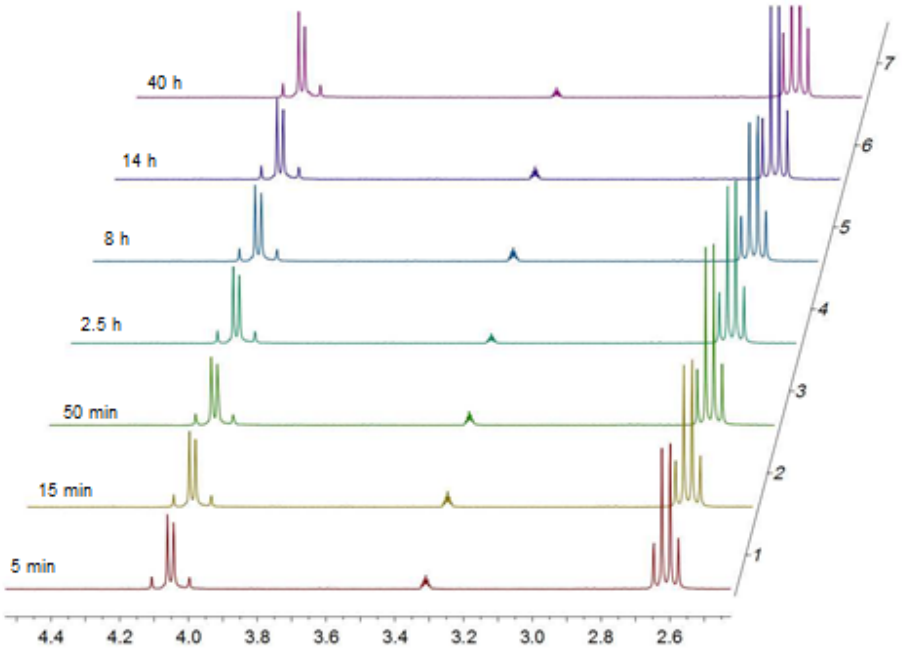
^{1}H NMR Spectra of DHMPE + Et_2NH (methanol-d_4)



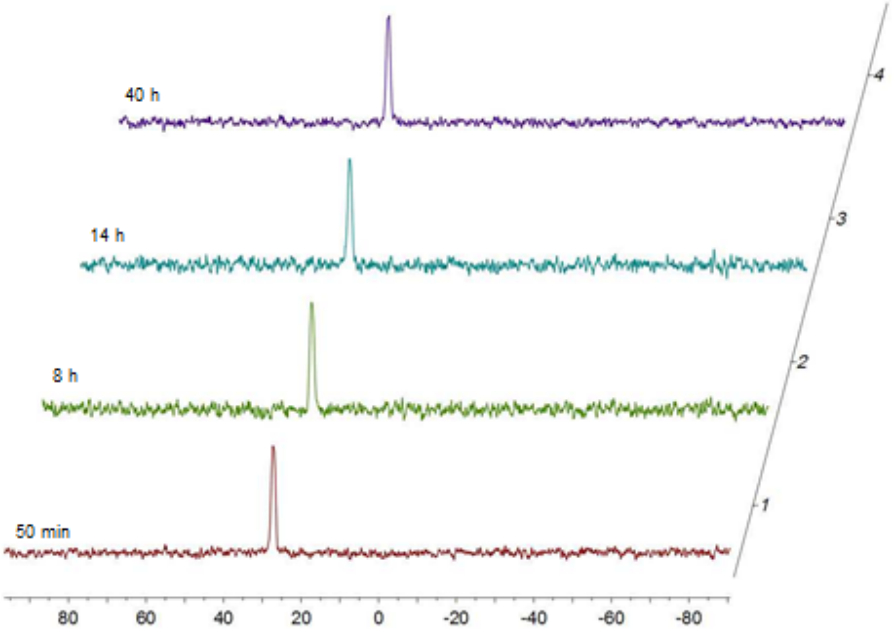
^{31}P{^{1}H} NMR Spectra of DHMPE + Et_2NH (methanol-d_4)



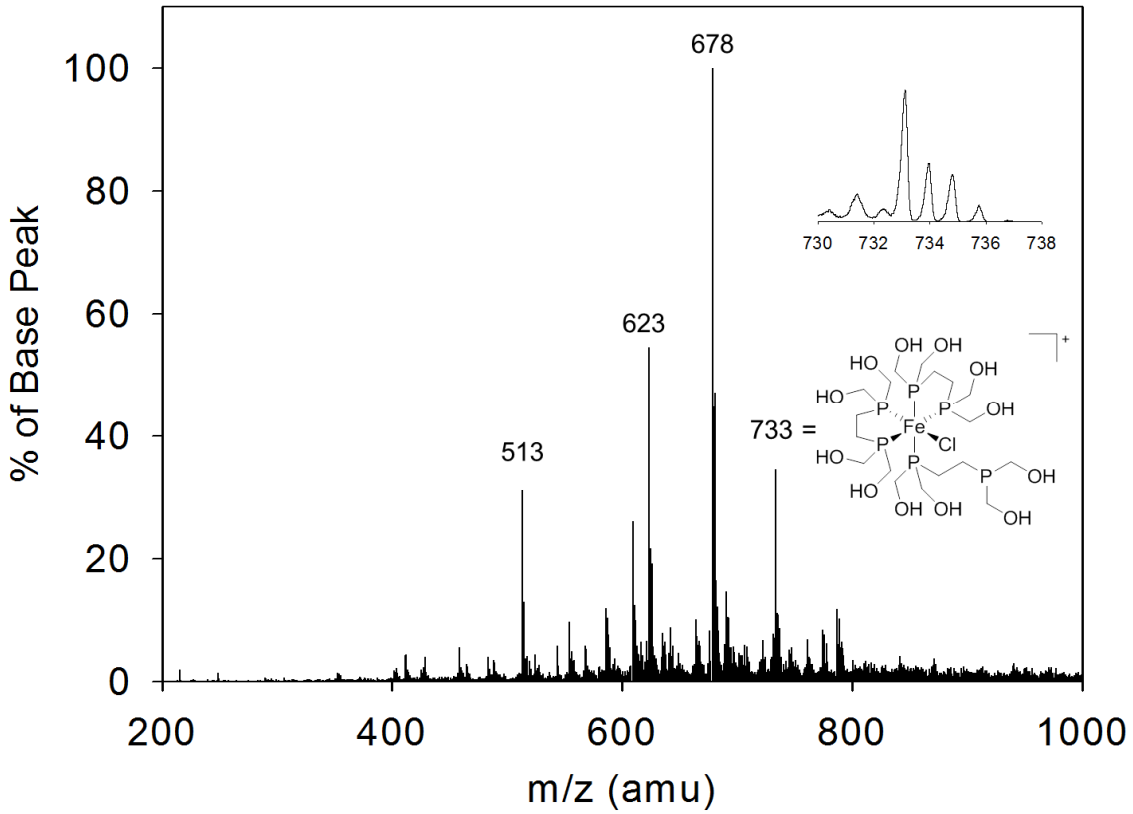
^{1}H NMR Spectra of DHMPE·2BH_3 + Et_2NH (methanol-d_4)



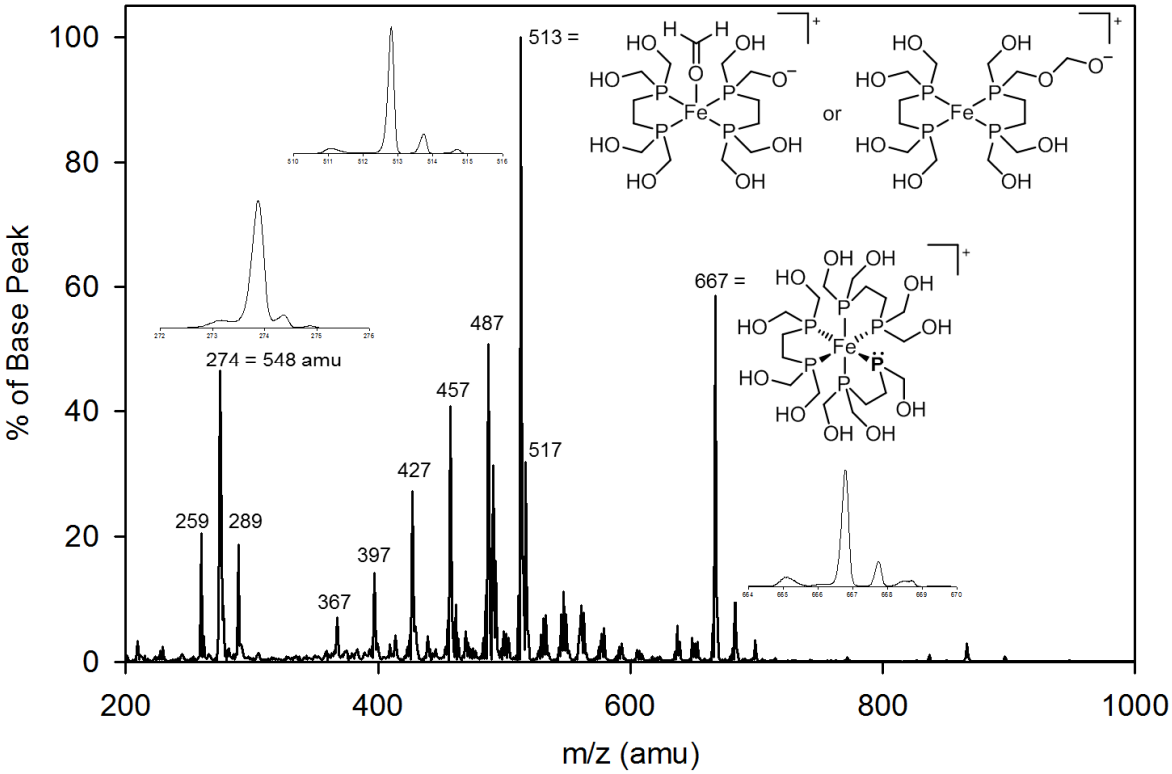
^{31}P{^{1}H} NMR Spectra of DHMPE·2BH_3 + Et_2NH (methanol-d_4)



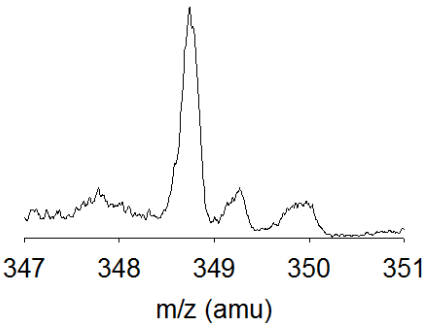
ESI-mass spectrum of $Fe(DHMPE)_2Cl_2 + BuNH_2$



ESI-mass spectrum of $[Fe(DHMPE)_3]^{2+}$



ESI-MS zoom scan of $[Fe(DHMPE)_3]^{2+}$



Crystallographic Data for *trans*-Fe(DHMPE)$_2$Cl$_2$

Table 1. Crystal data and structure refinement for *trans*-Fe(DHMPE)$_2$Cl$_2$.

Identification code	char2	
Empirical formula	C24 H64 Cl4 Fe2 O16 P8	
Formula weight	1110.01	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/n	
Unit cell dimensions	a = 7.7855(4) Å	a= 90°.
	b = 13.0809(7) Å	b= 92.3930(10)°.
	c = 10.4136(6) Å	g = 90°.
Volume	1059.61(10) Å^3	
Z	1	
Density (calculated)	1.740 Mg/m^3	
Absorption coefficient	1.303 mm^{-1}	
F(000)	576	
Crystal size	0.09 x 0.07 x 0.04 mm^3	
Theta range for data collection	2.50 to 27.50°.	
Index ranges	-10<=h<=9, -16<=k<=16, -13<=l<=13	
Reflections collected	11961	
Independent reflections	2428 [R(int) = 0.0494]	
Completeness to theta = 27.50°	99.6 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9497 and 0.8917	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2428 / 4 / 188	
Goodness-of-fit on F^2	1.115	
Final R indices [I>2sigma(I)]	R1 = 0.0364, wR2 = 0.0761	
R indices (all data)	R1 = 0.0509, wR2 = 0.0831	
Largest diff. peak and hole	0.482 and -0.328 e.Å^{-3}	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for *trans*-Fe(DHMPE)$_2$Cl$_2$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Fe(1)	0	5000	5000	11(1)
Cl(1)	2868(1)	5396(1)	5646(1)	17(1)
P(1)	535(1)	3321(1)	5249(1)	13(1)
P(2)	-551(1)	4954(1)	7085(1)	13(1)
O(1)	-584(3)	1348(1)	4917(2)	21(1)
O(2)	3966(3)	3175(2)	5826(2)	28(1)
O(3)	-1116(3)	6760(2)	8188(2)	24(1)
O(4)	-2957(3)	4722(2)	8904(2)	21(1)
C(1)	326(4)	2929(2)	6945(3)	18(1)
C(2)	568(4)	3855(2)	7824(3)	17(1)
C(3)	-864(4)	2355(2)	4436(3)	18(1)
C(4)	2695(4)	2843(2)	4892(3)	20(1)
C(5)	126(4)	5960(2)	8228(3)	18(1)
C(6)	-2790(4)	4759(2)	7543(3)	18(1)

Table 3. Bond lengths [Å] and angles [°] for *trans*-Fe(DHMPE)$_2$Cl$_2$.

Fe(1)-P(2)#1	2.2312(7)
Fe(1)-P(2)	2.2312(7)
Fe(1)-P(1)#1	2.2484(7)
Fe(1)-P(1)	2.2484(7)
Fe(1)-Cl(1)	2.3626(6)
Fe(1)-Cl(1)#1	2.3626(6)
P(1)-C(4)	1.846(3)
P(1)-C(3)	1.850(3)
P(1)-C(1)	1.853(3)
P(2)-C(2)	1.834(3)
P(2)-C(5)	1.837(3)
P(2)-C(6)	1.844(3)
O(1)-C(3)	1.422(3)
O(1)-H(1O)	0.879(18)
O(2)-C(4)	1.426(3)
O(2)-H(2O)	0.906(18)
O(3)-C(5)	1.424(3)
O(3)-H(3O)	0.935(19)
O(4)-C(6)	1.430(3)
O(4)-H(4O)	0.899(18)
C(1)-C(2)	1.525(4)
C(1)-H(1A)	0.97(3)
C(1)-H(1B)	0.91(3)
C(2)-H(2A)	1.03(3)
C(2)-H(2B)	0.98(3)
C(3)-H(3A)	0.95(3)
C(3)-H(3B)	0.94(3)
C(4)-H(4A)	0.94(3)
C(4)-H(4B)	0.95(3)
C(5)-H(5A)	0.96(3)
C(5)-H(5B)	0.98(3)
C(6)-H(6A)	0.91(3)
C(6)-H(6B)	0.96(3)

P(2)#1-Fe(1)-P(2)	180.0
P(2)#1-Fe(1)-P(1)#1	84.48(2)
P(2)-Fe(1)-P(1)#1	95.52(2)
P(2)#1-Fe(1)-P(1)	95.52(2)
P(2)-Fe(1)-P(1)	84.48(2)
P(1)#1-Fe(1)-P(1)	180.00(3)
P(2)#1-Fe(1)-Cl(1)	93.07(2)
P(2)-Fe(1)-Cl(1)	86.93(2)
P(1)#1-Fe(1)-Cl(1)	89.27(2)
P(1)-Fe(1)-Cl(1)	90.73(2)
P(2)#1-Fe(1)-Cl(1)#1	86.93(2)
P(2)-Fe(1)-Cl(1)#1	93.07(2)
P(1)#1-Fe(1)-Cl(1)#1	90.73(2)
P(1)-Fe(1)-Cl(1)#1	89.27(2)
Cl(1)-Fe(1)-Cl(1)#1	180.0
C(4)-P(1)-C(3)	101.55(14)
C(4)-P(1)-C(1)	102.43(14)
C(3)-P(1)-C(1)	99.99(14)
C(4)-P(1)-Fe(1)	118.24(10)
C(3)-P(1)-Fe(1)	120.80(9)
C(1)-P(1)-Fe(1)	110.92(9)
C(2)-P(2)-C(5)	99.86(13)
C(2)-P(2)-C(6)	102.74(13)
C(5)-P(2)-C(6)	100.39(13)
C(2)-P(2)-Fe(1)	108.80(9)
C(5)-P(2)-Fe(1)	123.35(10)
C(6)-P(2)-Fe(1)	118.52(10)
C(3)-O(1)-H(1O)	107(2)
C(4)-O(2)-H(2O)	107(2)
C(5)-O(3)-H(3O)	104(2)
C(6)-O(4)-H(4O)	109(2)
C(2)-C(1)-P(1)	109.82(19)
C(2)-C(1)-H(1A)	112.0(16)
P(1)-C(1)-H(1A)	108.2(16)
C(2)-C(1)-H(1B)	110.9(18)
P(1)-C(1)-H(1B)	101.9(17)

H(1A)-C(1)-H(1B)	113(2)
C(1)-C(2)-P(2)	109.0(2)
C(1)-C(2)-H(2A)	111.7(16)
P(2)-C(2)-H(2A)	104.9(16)
C(1)-C(2)-H(2B)	111.3(17)
P(2)-C(2)-H(2B)	110.2(16)
H(2A)-C(2)-H(2B)	110(2)
O(1)-C(3)-P(1)	113.16(19)
O(1)-C(3)-H(3A)	106(2)
P(1)-C(3)-H(3A)	106(2)
O(1)-C(3)-H(3B)	109.9(17)
P(1)-C(3)-H(3B)	109.7(17)
H(3A)-C(3)-H(3B)	112(3)
O(2)-C(4)-P(1)	111.8(2)
O(2)-C(4)-H(4A)	109.8(16)
P(1)-C(4)-H(4A)	111.1(16)
O(2)-C(4)-H(4B)	106.6(16)
P(1)-C(4)-H(4B)	110.0(16)
H(4A)-C(4)-H(4B)	107(2)
O(3)-C(5)-P(2)	109.32(19)
O(3)-C(5)-H(5A)	110.7(18)
P(2)-C(5)-H(5A)	106.8(18)
O(3)-C(5)-H(5B)	111.2(18)
P(2)-C(5)-H(5B)	109.6(17)
H(5A)-C(5)-H(5B)	109(2)
O(4)-C(6)-P(2)	112.8(2)
O(4)-C(6)-H(6A)	111.9(18)
P(2)-C(6)-H(6A)	107.4(18)
O(4)-C(6)-H(6B)	108.2(18)
P(2)-C(6)-H(6B)	106.8(18)
H(6A)-C(6)-H(6B)	110(3)

Symmetry transformations used to generate equivalent atoms:

#1 -x,-y+1,-z+1

Table 4. Anisotropic displacement parameters ($Å^2$x 10^3)for *trans*-$Fe(DHMPE)_2Cl_2$. The anisotropic displacement factor exponent takes the form: $-2p^2[h^2a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Fe(1)	12(1)	10(1)	11(1)	0(1)	1(1)	0(1)
Cl(1)	13(1)	16(1)	22(1)	-1(1)	0(1)	-1(1)
P(1)	14(1)	11(1)	13(1)	0(1)	2(1)	1(1)
P(2)	15(1)	12(1)	12(1)	0(1)	2(1)	0(1)
O(1)	23(1)	11(1)	29(1)	0(1)	10(1)	2(1)
O(2)	20(1)	18(1)	45(1)	4(1)	-10(1)	2(1)
O(3)	22(1)	18(1)	33(1)	-9(1)	0(1)	1(1)
O(4)	28(1)	16(1)	20(1)	-1(1)	12(1)	-2(1)
C(1)	21(2)	16(1)	17(1)	3(1)	3(1)	1(1)
C(2)	23(2)	16(1)	13(1)	2(1)	3(1)	2(1)
C(3)	21(2)	12(1)	20(2)	-2(1)	1(1)	-2(1)
C(4)	20(2)	17(2)	22(2)	4(1)	4(1)	4(1)
C(5)	23(2)	16(1)	14(1)	-2(1)	1(1)	-1(1)
C(6)	21(2)	14(1)	19(2)	2(1)	5(1)	-1(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for *trans*-Fe(DHMPE)$_2$Cl$_2$.

	x	y	z	U(eq)
H(1O)	330(30)	1110(30)	4550(30)	45(11)
H(2O)	4010(40)	3867(14)	5780(30)	39(10)
H(3O)	-540(50)	7310(20)	8580(40)	68(13)
H(4O)	-3080(50)	5360(17)	9210(30)	51(12)
H(1A)	1170(30)	2400(20)	7140(30)	15(7)
H(1B)	-780(40)	2700(20)	6940(30)	16(8)
H(2A)	1830(40)	4070(20)	7910(30)	27(8)
H(2B)	130(30)	3720(20)	8670(30)	15(7)
H(3A)	-2000(40)	2530(30)	4630(30)	39(10)
H(3B)	-700(30)	2370(20)	3550(30)	18(8)
H(4A)	3010(30)	3050(20)	4070(30)	13(7)
H(4B)	2710(30)	2120(20)	4910(20)	11(7)
H(5A)	200(40)	5650(20)	9060(30)	21(8)
H(5B)	1270(40)	6220(20)	8020(30)	25(8)
H(6A)	-3430(40)	5270(20)	7170(30)	17(8)
H(6B)	-3150(40)	4110(20)	7190(30)	25(8)

Table 6. Torsion angles [°] for *trans*-Fe(DHMPE)$_2$Cl$_2$.

P(2)#1-Fe(1)-P(1)-C(4)	59.41(11)
P(2)-Fe(1)-P(1)-C(4)	-120.59(11)
P(1)#1-Fe(1)-P(1)-C(4)	-31(100)
Cl(1)-Fe(1)-P(1)-C(4)	-33.74(11)
Cl(1)#1-Fe(1)-P(1)-C(4)	146.26(11)
P(2)#1-Fe(1)-P(1)-C(3)	-66.38(12)
P(2)-Fe(1)-P(1)-C(3)	113.62(12)
P(1)#1-Fe(1)-P(1)-C(3)	-157(100)
Cl(1)-Fe(1)-P(1)-C(3)	-159.53(12)
Cl(1)#1-Fe(1)-P(1)-C(3)	20.47(12)
P(2)#1-Fe(1)-P(1)-C(1)	177.25(11)
P(2)-Fe(1)-P(1)-C(1)	-2.75(11)
P(1)#1-Fe(1)-P(1)-C(1)	87(100)
Cl(1)-Fe(1)-P(1)-C(1)	84.10(11)
Cl(1)#1-Fe(1)-P(1)-C(1)	-95.90(11)
P(2)#1-Fe(1)-P(2)-C(2)	-159(100)
P(1)#1-Fe(1)-P(2)-C(2)	-156.76(10)
P(1)-Fe(1)-P(2)-C(2)	23.24(10)
Cl(1)-Fe(1)-P(2)-C(2)	-67.79(10)
Cl(1)#1-Fe(1)-P(2)-C(2)	112.21(10)
P(2)#1-Fe(1)-P(2)-C(5)	-43(100)
P(1)#1-Fe(1)-P(2)-C(5)	-40.62(12)
P(1)-Fe(1)-P(2)-C(5)	139.38(12)
Cl(1)-Fe(1)-P(2)-C(5)	48.35(11)
Cl(1)#1-Fe(1)-P(2)-C(5)	-131.65(11)
P(2)#1-Fe(1)-P(2)-C(6)	85(100)
P(1)#1-Fe(1)-P(2)-C(6)	86.49(11)
P(1)-Fe(1)-P(2)-C(6)	-93.51(11)
Cl(1)-Fe(1)-P(2)-C(6)	175.46(11)
Cl(1)#1-Fe(1)-P(2)-C(6)	-4.54(11)
C(4)-P(1)-C(1)-C(2)	104.2(2)
C(3)-P(1)-C(1)-C(2)	-151.5(2)
Fe(1)-P(1)-C(1)-C(2)	-22.8(2)
P(1)-C(1)-C(2)-P(2)	41.2(2)

C(5)-P(2)-C(2)-C(1)	-174.4(2)
C(6)-P(2)-C(2)-C(1)	82.5(2)
Fe(1)-P(2)-C(2)-C(1)	-44.0(2)
C(4)-P(1)-C(3)-O(1)	60.1(2)
C(1)-P(1)-C(3)-O(1)	-44.9(2)
Fe(1)-P(1)-C(3)-O(1)	-166.69(15)
C(3)-P(1)-C(4)-O(2)	-154.9(2)
C(1)-P(1)-C(4)-O(2)	-51.9(2)
Fe(1)-P(1)-C(4)-O(2)	70.4(2)
C(2)-P(2)-C(5)-O(3)	-154.9(2)
C(6)-P(2)-C(5)-O(3)	-49.8(2)
Fe(1)-P(2)-C(5)-O(3)	84.7(2)
C(2)-P(2)-C(6)-O(4)	58.7(2)
C(5)-P(2)-C(6)-O(4)	-44.0(2)
Fe(1)-P(2)-C(6)-O(4)	178.62(15)

Symmetry transformations used to generate equivalent atoms:

#1 -x,-y+1,-z+1

Table 7. Hydrogen bonds for *trans*-Fe(DHMPE)$_2$Cl$_2$ [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(3)-H(3O)...O(2)#2	0.935(19)	1.76(2)	2.673(3)	164(4)
O(2)-H(2O)...Cl(1)	0.906(18)	2.19(2)	3.031(2)	154(3)
O(1)-H(1O)...O(4)#3	0.879(18)	1.87(2)	2.726(3)	165(3)
O(4)-H(4O)...O(1)#4	0.899(18)	1.91(2)	2.728(3)	149(3)
O(4)-H(4O)...O(3)	0.899(18)	2.64(3)	3.132(3)	116(3)

Symmetry transformations used to generate equivalent atoms:

#1 -x,-y+1,-z+1 #2 -x+1/2,y+1/2,-z+3/2 #3 x+1/2,-y+1/2,z-1/2

#4 -x-1/2,y+1/2,-z+3/2

Crystallographic Data for DHMPE·2BH$_3$

Table 8. Crystal data and structure refinement for DHMPE·2BH$_3$.

Identification code	char5	
Empirical formula	C6 H22 B2 O4 P2	
Formula weight	241.80	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C2/c	
Unit cell dimensions	a = 18.092(3) Å	a= 90°.
	b = 6.2042(11) Å	b= 100.688(3)°.
	c = 11.757(2) Å	g = 90°.
Volume	1296.8(4) Å^3	
Z	4	
Density (calculated)	1.238 Mg/m^3	
Absorption coefficient	0.323 mm^{-1}	
F(000)	520	
Crystal size	0.27 x 0.12 x 0.02 mm^3	
Theta range for data collection	2.29 to 27.00°.	
Index ranges	-22<=h<=22, -7<=k<=7, -14<=l<=15	
Reflections collected	6768	
Independent reflections	1408 [R(int) = 0.0549]	
Completeness to theta = 27.00°	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9936 and 0.9179	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	1408 / 2 / 108	
Goodness-of-fit on F^2	1.077	
Final R indices [I>2sigma(I)]	R1 = 0.0495, wR2 = 0.1148	
R indices (all data)	R1 = 0.0642, wR2 = 0.1245	
Largest diff. peak and hole	1.155 and -0.301 e.Å^{-3}	

Table 9. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for DHMPE·$2BH_3$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
P(1)	3940(1)	1883(1)	9624(1)	19(1)
O(1)	2934(1)	4245(3)	8185(1)	27(1)
O(2)	3126(1)	-1572(3)	8685(2)	32(1)
B(1)	4048(2)	3741(5)	10936(3)	29(1)
C(1)	3635(2)	3192(4)	8220(2)	26(1)
C(2)	3235(2)	-200(4)	9667(2)	25(1)
C(3)	4789(1)	479(4)	9433(2)	23(1)

Table 10. Bond lengths [Å] and angles [°] for DHMPE·2BH_3.

P(1)-C(3)	1.816(2)
P(1)-C(2)	1.823(3)
P(1)-C(1)	1.830(3)
P(1)-B(1)	1.907(3)
O(1)-C(1)	1.420(3)
O(1)-H(1O)	0.908(17)
O(2)-C(2)	1.419(3)
O(2)-H(2O)	0.913(18)
B(1)-H(1B)	1.13(4)
B(1)-H(2B)	1.16(3)
B(1)-H(3B)	1.08(3)
C(1)-H(1A)	0.92(3)
C(1)-H(1C)	0.99(3)
C(2)-H(2A)	0.96(3)
C(2)-H(2C)	0.94(3)
C(3)-C(3)#1	1.529(5)
C(3)-H(3A)	0.96(3)
C(3)-H(3C)	0.89(3)
C(3)-P(1)-C(2)	105.99(13)
C(3)-P(1)-C(1)	102.83(12)
C(2)-P(1)-C(1)	104.13(13)
C(3)-P(1)-B(1)	115.33(13)
C(2)-P(1)-B(1)	111.93(13)
C(1)-P(1)-B(1)	115.46(14)
C(1)-O(1)-H(1O)	109.7(15)
C(2)-O(2)-H(2O)	111(2)
P(1)-B(1)-H(1B)	101.2(19)
P(1)-B(1)-H(2B)	106.9(16)
H(1B)-B(1)-H(2B)	116(3)
P(1)-B(1)-H(3B)	107.2(17)
H(1B)-B(1)-H(3B)	116(3)
H(2B)-B(1)-H(3B)	109(2)
O(1)-C(1)-P(1)	110.42(18)

O(1)-C(1)-H(1A)	109.5(17)
P(1)-C(1)-H(1A)	108.2(17)
O(1)-C(1)-H(1C)	109.2(17)
P(1)-C(1)-H(1C)	104.0(16)
H(1A)-C(1)-H(1C)	115(2)
O(2)-C(2)-P(1)	113.25(18)
O(2)-C(2)-H(2A)	107.4(17)
P(1)-C(2)-H(2A)	107.9(17)
O(2)-C(2)-H(2C)	111.2(18)
P(1)-C(2)-H(2C)	108.8(18)
H(2A)-C(2)-H(2C)	108(2)
C(3)#1-C(3)-P(1)	112.1(2)
C(3)#1-C(3)-H(3A)	110.3(18)
P(1)-C(3)-H(3A)	105.0(17)
C(3)#1-C(3)-H(3C)	108.9(16)
P(1)-C(3)-H(3C)	109.4(15)
H(3A)-C(3)-H(3C)	111(2)

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,-y,-z+2

Table 11. Anisotropic displacement parameters ($Å^2 x\ 10^3$)for DHMPE·2BH_3. The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
P(1)	20(1)	17(1)	19(1)	0(1)	0(1)	2(1)
O(1)	28(1)	19(1)	30(1)	-1(1)	-7(1)	4(1)
O(2)	36(1)	20(1)	33(1)	-4(1)	-12(1)	0(1)
B(1)	32(2)	26(2)	27(2)	-6(1)	1(1)	3(1)
C(1)	30(2)	24(1)	23(1)	2(1)	0(1)	2(1)
C(2)	24(1)	21(1)	28(1)	1(1)	1(1)	0(1)
C(3)	23(1)	24(1)	21(1)	-1(1)	2(1)	4(1)

Table 12. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2 x\ 10^3$) for DHMPE·2BH_3.

	x	y	z	U(eq)
H(1O)	3013(13)	5640(30)	8400(20)	23(7)
H(2O)	2741(15)	-1080(60)	8130(20)	67(11)
H(1B)	4080(20)	2530(60)	11670(30)	72(11)
H(2B)	3527(18)	4860(50)	10800(30)	59(10)
H(3B)	4544(19)	4720(60)	10930(30)	60(10)
H(1A)	3992(16)	4190(50)	8120(20)	35(8)
H(1C)	3567(16)	1990(50)	7660(20)	41(8)
H(2A)	3404(15)	-1080(50)	10330(20)	33(7)
H(2C)	2782(17)	460(50)	9760(20)	40(8)
H(3A)	5092(17)	1560(50)	9150(20)	39(8)
H(3C)	4669(13)	-580(40)	8920(20)	15(6)

Table 13. Torsion angles [°] for DHMPE·2BH_3.

C(3)-P(1)-C(1)-O(1)	175.43(18)
C(2)-P(1)-C(1)-O(1)	65.0(2)
B(1)-P(1)-C(1)-O(1)	-58.1(2)
C(3)-P(1)-C(2)-O(2)	-55.2(2)
C(1)-P(1)-C(2)-O(2)	52.9(2)
B(1)-P(1)-C(2)-O(2)	178.29(18)
C(2)-P(1)-C(3)-C(3)#1	-79.3(3)
C(1)-P(1)-C(3)-C(3)#1	171.6(3)
B(1)-P(1)-C(3)-C(3)#1	45.1(3)

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,-y,-z+2

Table 14. Hydrogen bonds for DHMPE·2BH_3 [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(1)-H(1O)...O(2)#2	0.908(17)	1.764(17)	2.670(2)	174(2)
O(2)-H(2O)...O(1)#3	0.913(18)	1.80(2)	2.684(2)	163(3)

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,-y,-z+2 #2 x,y+1,z #3 -x+1/2,y-1/2,-z+3/2