Coordination-Driven Self-Assembly of Truncated Tetrahedra Capable of Encapsulating 1,3,5-Triphenylbenzene

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Methods and Materials. 90° organoplatinum acceptor **1**[1] and hexapyridyl donor **2**[2] were prepared according to literature procedures. Deuterated acetone, nitromethane, and water were purchased from Cambridge Isotope Laboratory (Andover, MA). NMR spectra were recorded on a Varian Unity 300 spectrometer, and pulsed field gradient spin echo (PGSE) NMR data were obtained on an Inova 500 MHz spectrometer. The ^{1}H NMR chemical shifts are reported relative to residual solvent signals, and ^{31}P NMR resonances are referenced to an external unlocked sample of 85% H_3PO_4 (δ 0.0). Mass spectra were recorded on a Micromass Quattro II triple-quadrupole mass spectrometer using electrospray ionization (ESI) with a MassLynx operating system. Element analysis was performed by Atlantic Microlab (Norcross, GA).

Truncated Tetrahedron 3a. 90° organoplatinum acceptor **1a** (5.87 mg, 8.05 mmol) and hexapyridyl donor **2** (2.65 mg, 2.66 mmol) were placed in a 2-dram vial followed by addition of 1.0 mL CD_3NO_2/Acetone-d_6 (*v/v* 7/3) solvent. The mixture was stirred at 80 °C for 16 h, and the truncated tetrahedron **3a** was formed. The solvent was then removed by evaporation under N_2 flow and vacuum. 0.8 ml Acetone was added to the mixture to dissolve the self-assembly. The OTf–counterions were exchanged for PF_6^- using an aqueous solution of KPF_6 to precipitate the product, which was collected and washed with excess water and then dried in vacuum. Yield: 93 %. ^{1}H NMR (CD_3NO_2/Acetone-d_6: 7/3, 300 MHz): δ 8.91 (m, 48H, $H_{\alpha\text{-Py}}$), 7.58 (d, J_1 = 5.1 Hz, 24H, $H_{\beta\text{-Py}}$), 7.44 (d, J_1 = 5.1 Hz, 24H, $H_{\beta\text{-Py}}$), 7.14 (dd, J_1 = 7.8 Hz, J_2 = 33.9 Hz, 48H, PhH_{exterior}), 6.94 (dd, J_1 = 6.0 Hz, J_2 = 6.3 Hz, 48H, PhH_{interior}), 2.10 (m, 144H, PC*H*2CH3), 1.29 (m, 216H, PCH2C*H*3). ^{31}P{^{1}H} NMR (CD_3NO_2/Acetone-d_6: 7/3, 121.4 MHz): δ 0.92 (s, $^{1}J_{\text{Pt-P}}$ = 3121 Hz). Anal. Calcd for $C_{432}H_{552}F_{144}N_{24}P_{48}Pt_{12}$: C, 41.03; H, 4.40; N, 2.66. Found: C, 40.80; H, 4.50; N, 2.65.

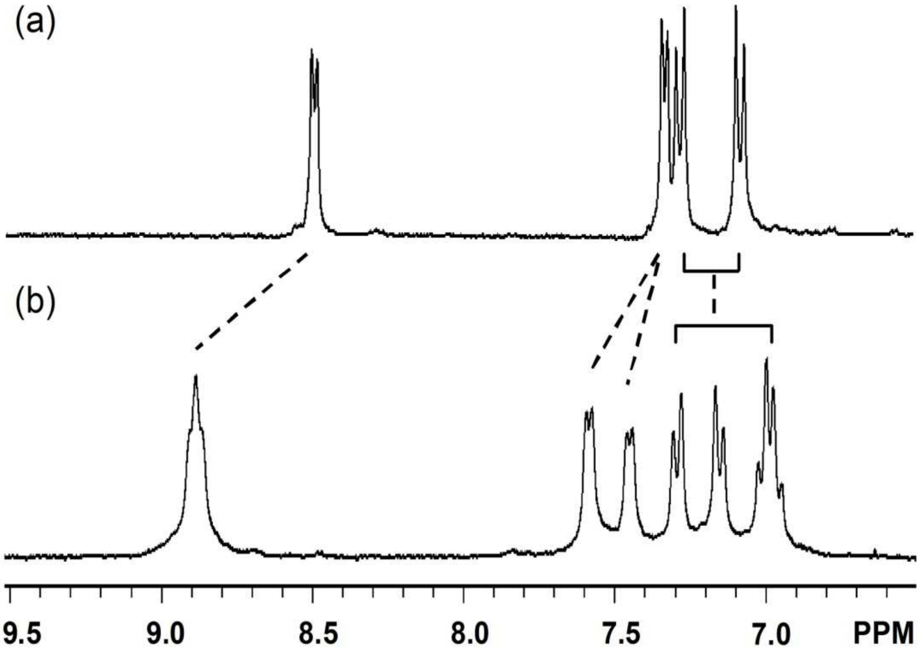

Figure S1. Partial ^{1}H NMR (300MHz) spectra of hexapyridyl ligand **2** (a) and truncated tetrahedron **3a** (b).

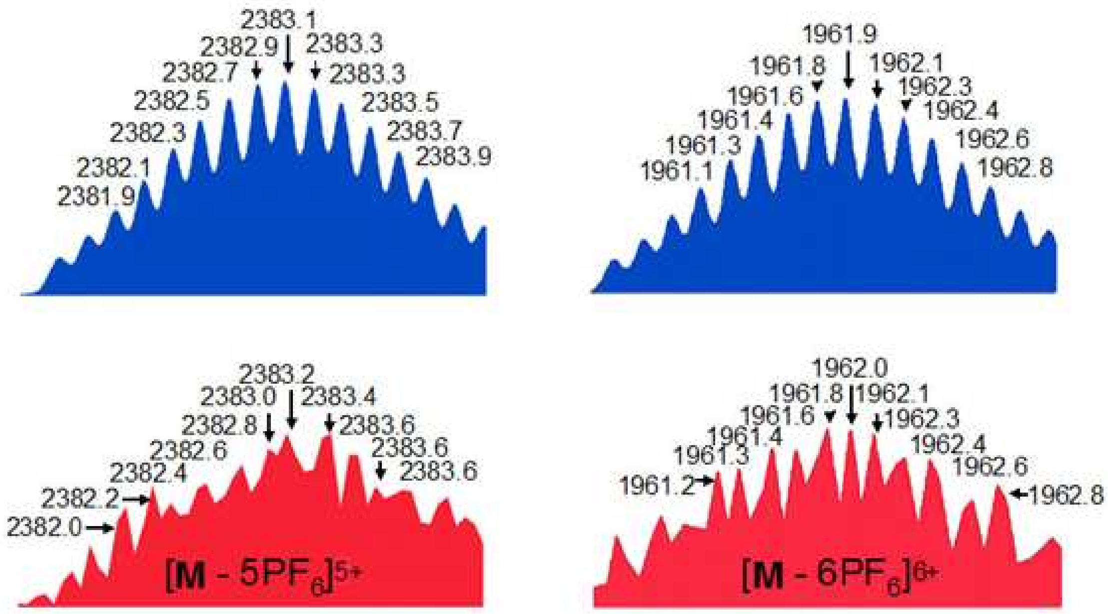

Figure S2. Calculated (top) and Experimental (bottom) ESI mass spectra of **3a**.

Truncated Tetrahedron 3b. 90° organoplatinum acceptor **1b** (3.57 mg, 5.53 mmol) and hexapyridyl donor **2** (1.83 mg, 1.84 mmol) were placed in a 2-dram vial followed by addition of 1.0 mL D_2O/Acetone-d_6 (v/v 1/1) solvent. The mixture was stirred at 80 °C for 16 h, and the truncated tetrahedron **3b** was formed. The OTf–counterions were exchanged for PF_6^- using an aqueous solution of KPF_6 to precipitate the product, which was collected and washed with excess water and then dried in vacuum. Yield: 91 %. ^{1}H NMR (D_2O/Acetone-d_6: 1/1, 300 MHz): δ 8.68 (m, 48H, $H_{\alpha\text{-Py}}$), 7.48 (m, 48H, $H_{\beta\text{-Py}}$), 7.03 (dd, J_1 = 7.8 Hz, J_2 = 37.2 Hz, 48H, PhH_{exterior}), 6.78 (m, 48H, PhH_{interior}), 1.81 (d, J = 10.8 Hz, 216H, PCH3). ^{31}P{^{1}H} NMR (D_2O/Acetone-d_6: 1/1, 121.4 MHz): δ -28.2 ppm (s, $^1J_{\text{Pt-P}}$ = 3110 Hz). Anal. Calcd for $C_{360}H_{408}F_{144}N_{24}P_{48}Pt_{12}$: C, 37.16; H, 3.53; N, 2.89. Found: C, 37.53; H, 3.80; N, 2.82.

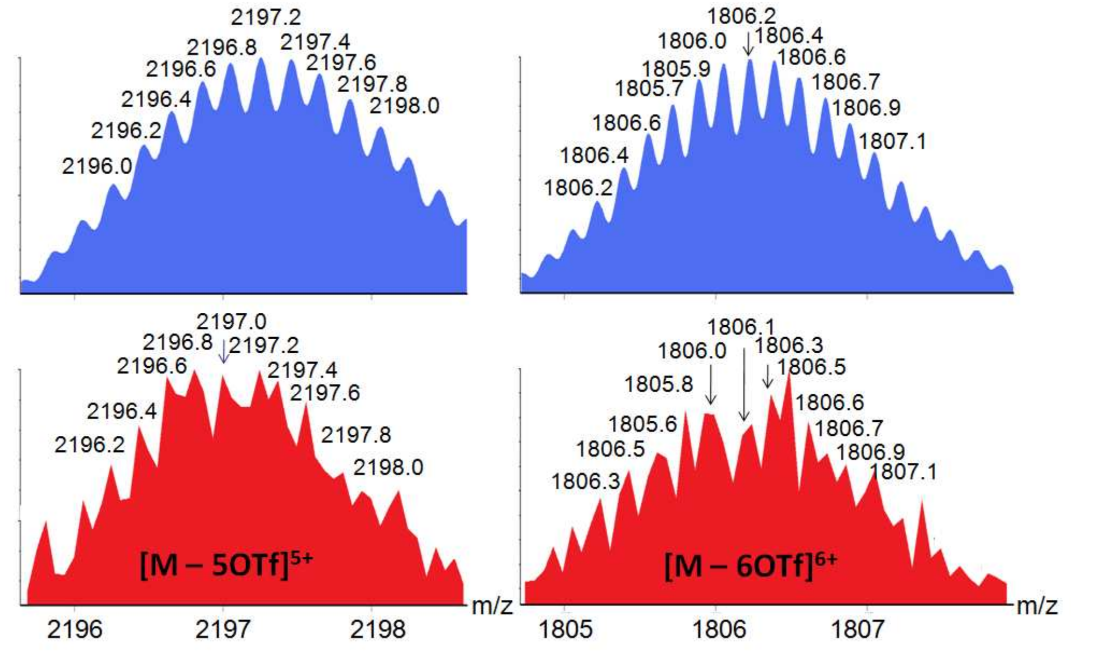

Figure S3. Calculated (top) and Experimental (bottom) ESI mass spectra of **3b**.

X-Ray Crystallographic Data of Truncated Tetrahedron 3a

The diffraction data from single crystals of **3a** mounted on a loop were collected at 90 K on an ADSC Quantum 210 CCD diffractometer with a synchrotron radiation (λ = 0.90000 Å) at Macromolecular Crystallography Beamline 6B1, Pohang Accelerator Laboratory (PAL), Pohang, Korea. The raw data were processed and scaled using the program HKL2000. The structure was solved by direct methods, and the refinements were carried out with full-matrix least-squares on F^2 with appropriate softwares implemented in SHELXTL program package. X-ray data for **3a**: $C_{108}H_{138}F_{36}N_6P_{12}Pt_3$, M = 3161.15, tetragonal, P-42$_1c$ (No. 114), a = 32.987(5) Å, c = 31.918(6) Å, V =34730(10) Å^3, Z = 8, T = 90 K, μ(synchrotron) = 4.137 mm^{-1}, d_{calc} = 1.209 g·cm^{-3}, 154346 reflections measured, 23131 unique (R_{int} = 0.0674), R_1 = 0.0841, wR_2 = 0.2244 for 19314 reflections ($I > 2\sigma(I)$), R_1 = 0.0981, wR_2 = 0.2396 (all data), GoF = 1.060, Flack = 0.015(6), 1487 parameters and 3157 restraints. All the non-hydrogen atoms were refined anisotropically. Hydrogen atoms were added to their geometrically ideal positions.

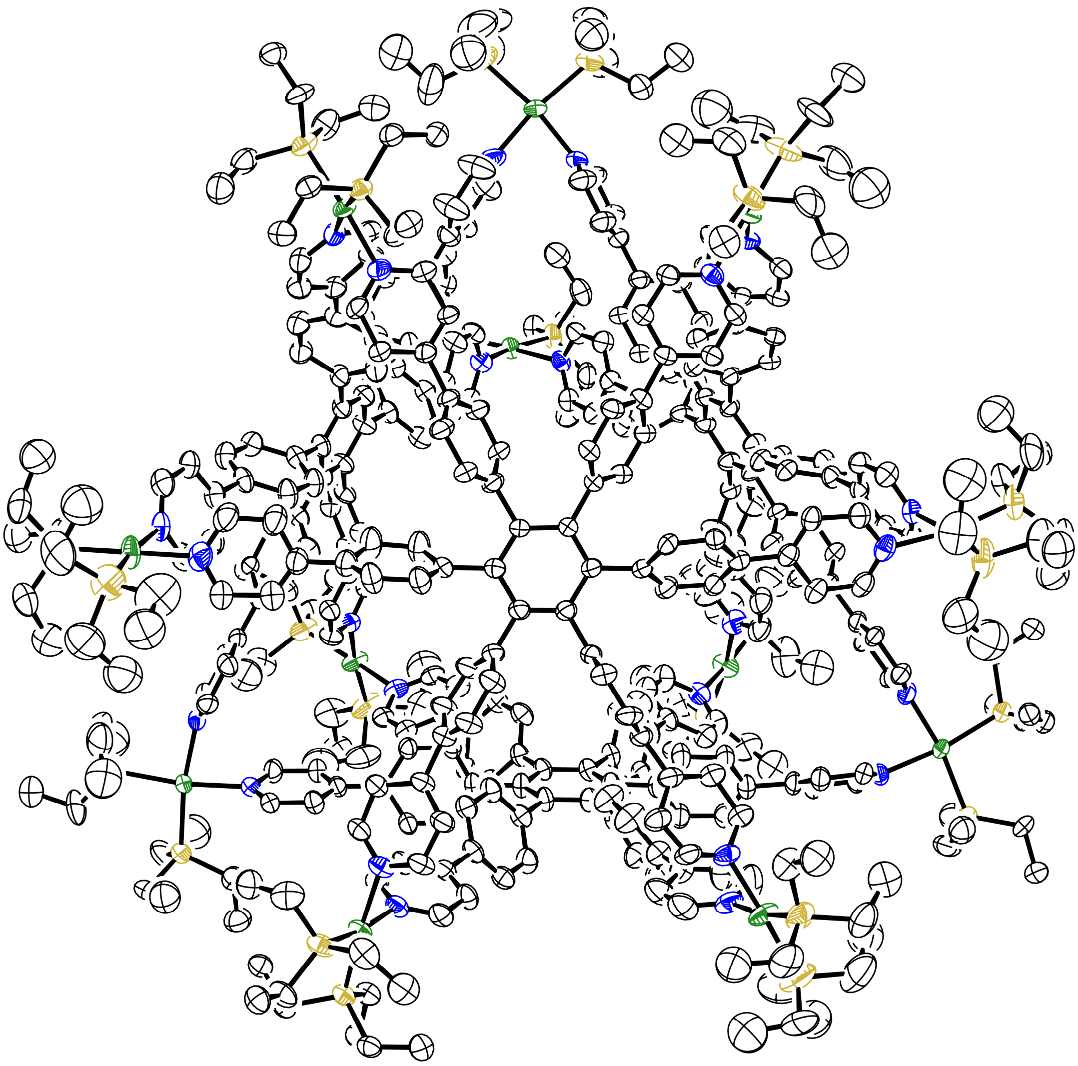

Figure S4. X-ray crystal structure of **3a** with 30 % probability.

Table S1. Crystal data and structure refinement for **3a**.

Identification code	**3a**	
Empirical formula	C108 H138 F36 N6 P12 Pt3	
Formula weight	3161.15	
Temperature	90(2) K	
Wavelength	0.90000 Å	
Crystal system	Tetragonal	
Space group	P-$42_1/c$	
Unit cell dimensions	a = 32.987(5) Å	α = 90°
	b = 32.987(5) Å	β = 90°
	c = 31.918(6) Å	γ = 90°
Volume	34730(10) Å^3	
Z	8	
Density (calculated)	1.209 g/cm^3	
Absorption coefficient	4.137 mm^{-1}	
F(000)	12528	
Crystal size	0.42 × 0.42 × 0.38 mm^3	
Theta range for data collection	1.56 to 30.37°	
Index ranges	$-36 \leq h \leq 36$, $-36 \leq k \leq 37$, $-32 \leq l \leq 32$	
Reflections collected	154346	
Independent reflections	23131 [R_{int} = 0.0674]	
Completeness to theta = 25.00°	99.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.3024 and 0.2754	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	23131 / 3157 / 1487	
Goodness-of-fit on F^2	1.050	
Final R indices [I>2sigma(I)]	R_1 = 0.0841, wR_2 = 0.2244	
R indices (all data)	R_1 = 0.0981, wR_2 = 0.2396	
Absolute structure parameter	0.015(6)	
Extinction coefficient	0.00123(6)	
Largest diff. peak and hole	2.491 and -1.069 e.Å^{-3}	

Table S2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for **3a**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Pt(1)	-1800(1)	-2495(1)	-3546(1)	71(1)
Pt(2)	-3351(1)	-4668(1)	-1095(1)	88(1)
Pt(3)	-8958(1)	-3661(1)	-3679(1)	101(1)
N(1)	-3713(4)	-4363(3)	-1538(3)	72(2)
N(2)	-8352(3)	-3483(4)	-3629(4)	86(3)
N(3)	-2418(3)	-2576(4)	-3559(3)	79(2)
N(4)	-7101(4)	-1836(3)	-5943(3)	76(3)
N(5)	-6538(3)	-4853(3)	-1504(4)	81(3)
N(6)	-3980(5)	-1290(4)	-5820(5)	103(4)
C(1)	-4663(4)	-4040(4)	-2745(4)	72(3)
C(2)	-4425(4)	-3814(4)	-2469(4)	72(3)
C(3)	-4170(4)	-4003(4)	-2146(4)	68(3)
C(4)	-4435(4)	-3390(4)	-2516(4)	73(3)
C(5)	-4893(4)	-3863(4)	-3059(4)	76(3)
C(6)	-4894(4)	-3451(4)	-3092(4)	70(3)
C(7)	-4674(4)	-3213(4)	-2826(4)	73(3)
C(8)	-5152(4)	-3244(4)	-3421(4)	73(3)
C(9)	-5705(4)	-3490(4)	-2700(4)	68(3)
C(10)	-5762(3)	-3570(5)	-3117(4)	72(3)
C(11)	-6070(4)	-4093(4)	-2485(4)	67(3)
C(12)	-6220(4)	-4338(4)	-2140(4)	69(3)
C(13)	-6135(4)	-4174(5)	-2887(4)	80(3)
C(14)	-5971(4)	-3926(5)	-3210(5)	86(3)
C(15)	-5560(3)	-3318(4)	-3440(4)	68(3)
C(16)	-4971(4)	-3001(4)	-3736(4)	74(3)
C(17)	-3766(4)	-3935(5)	-2123(4)	79(3)
C(18)	-4110(5)	-4439(5)	-1554(4)	84(3)
C(19)	-4349(4)	-4255(4)	-1860(4)	76(3)
C(20)	-3549(4)	-4116(4)	-1803(4)	72(3)
C(21)	-5859(4)	-2673(4)	-4379(4)	75(3)
C(22)	-5193(4)	-2813(4)	-4043(4)	73(3)

C(23)	-5799(4)	-3120(4)	-3762(4)	69(3)
C(24)	-4485(7)	-2423(6)	-4883(6)	122(6)
C(25)	-4590(5)	-2018(5)	-4926(5)	89(4)
C(26)	-4849(6)	-1873(5)	-4632(5)	108(5)
C(27)	-5047(6)	-2131(5)	-4353(5)	94(4)
C(28)	-4991(4)	-2531(4)	-4351(4)	71(3)
C(29)	-4697(6)	-2692(6)	-4615(6)	115(5)
C(30)	-4513(4)	-2929(5)	-3728(4)	74(3)
C(31)	-4245(4)	-3256(5)	-3743(4)	81(3)
C(32)	-4376(4)	-2527(5)	-3661(5)	88(3)
C(33)	-3949(4)	-2461(5)	-3639(4)	82(3)
C(34)	-3835(4)	-3187(5)	-3700(4)	79(3)
C(35)	-3690(4)	-2788(5)	-3656(4)	75(3)
C(36)	-5615(4)	-2881(4)	-4054(4)	70(3)
C(37)	-6242(4)	-3181(5)	-3772(4)	72(3)
C(38)	-6841(4)	-3548(6)	-3948(5)	93(4)
C(39)	-6427(4)	-3507(5)	-3964(5)	90(4)
C(40)	-6909(4)	-2942(5)	-3534(5)	85(3)
C(41)	-7086(4)	-3266(5)	-3738(4)	79(3)
C(42)	-7527(4)	-3333(5)	-3697(4)	82(3)
C(43)	-6037(4)	-4377(4)	-1771(4)	74(3)
C(44)	-6594(5)	-4569(5)	-2190(5)	88(3)
C(45)	-7658(4)	-3713(6)	-3536(5)	97(4)
C(46)	-8093(5)	-3770(6)	-3495(6)	105(4)
C(47)	-8210(4)	-3127(6)	-3755(5)	98(4)
C(48)	-2651(4)	-2334(5)	-3790(5)	84(3)
C(49)	-3240(4)	-2718(4)	-3608(4)	71(3)
C(50)	-3016(4)	-2952(5)	-3362(4)	73(3)
C(51)	-6712(4)	-1894(4)	-6030(4)	77(3)
C(52)	-7247(5)	-1899(5)	-5568(4)	86(4)
C(53)	-6435(5)	-2027(5)	-5734(5)	84(3)
C(54)	-6603(4)	-2123(4)	-5320(4)	78(3)
C(55)	-6324(4)	-2289(5)	-4995(5)	83(3)
C(56)	-6197(4)	-4626(4)	-1455(4)	78(3)
C(57)	-6487(4)	-2892(5)	-3545(5)	84(3)
C(58)	-7820(4)	-3033(5)	-3819(5)	93(4)
C(59)	-2586(4)	-2874(5)	-3338(5)	80(3)
C(60)	-4387(5)	-1777(6)	-5232(6)	99(4)

C(61)	-3990(7)	-1668(7)	-5170(6)	122(5)
C(62)	-5845(5)	-2803(6)	-4787(5)	96(4)
C(63)	-6113(5)	-2332(5)	-4278(5)	90(4)
C(64)	-7002(5)	-2032(5)	-5244(4)	85(3)
C(65)	-6062(5)	-2607(5)	-5096(5)	93(4)
C(66)	-5845(4)	-3733(4)	-2380(4)	63(2)
C(67)	-3078(4)	-2399(5)	-3829(5)	85(4)
C(68)	-6726(4)	-4810(4)	-1862(5)	82(3)
C(69)	-6351(5)	-2150(5)	-4582(5)	88(4)
C(70)	-4357(7)	-1370(9)	-5858(9)	158(8)
C(71)	-4560(8)	-1604(9)	-5523(8)	162(8)
C(72)	-3779(7)	-1442(7)	-5480(6)	123(5)
P(1)	-1115(1)	-2486(1)	-3628(1)	87(1)
C(1P)	-938(6)	-2995(4)	-3675(6)	119(5)
C(2P)	-996(7)	-3252(6)	-3286(6)	123(5)
C(3P)	-1001(5)	-2230(6)	-4124(5)	115(4)
C(4P)	-1196(7)	-1813(6)	-4191(7)	132(6)
C(5P)	-792(3)	-2244(6)	-3256(5)	97(4)
C(6P)	-343(4)	-2274(6)	-3345(5)	96(4)
P(2)	-1859(1)	-2061(1)	-2994(1)	73(1)
C(7P)	-2385(3)	-2005(5)	-2821(4)	85(3)
C(8P)	-2444(5)	-1712(5)	-2458(5)	97(4)
C(9P)	-1719(4)	-1544(3)	-3121(4)	80(3)
C(10P)	-1981(5)	-1336(5)	-3449(5)	101(4)
C(11P)	-1579(4)	-2207(4)	-2535(4)	82(3)
C(12P)	-1688(5)	-2640(4)	-2395(5)	83(3)
P(3)	-3355(2)	-4108(1)	-676(2)	110(2)
C(13P)	-3656(6)	-4220(9)	-213(5)	151(6)
C(14P)	-4100(7)	-4322(11)	-311(11)	199(9)
C(15P)	-3614(8)	-3680(5)	-915(5)	129(6)
C(16P)	-3726(7)	-3321(5)	-644(6)	124(5)
C(17P)	-2888(5)	-3907(6)	-486(6)	124(5)
P(4)	-2964(2)	-5072(2)	-682(2)	137(2)
C(19P)	-2601(8)	-5323(10)	-1018(8)	191(7)
C(18P)	-2660(8)	-3736(9)	-859(8)	164(8)
C(20P)	-2324(13)	-4950(12)	-1097(14)	244(11)
C(21P)	-3221(9)	-5535(6)	-536(11)	188(7)
C(22P)	-3602(13)	-5444(15)	-279(14)	252(12)

C(23P)	-2866(10)	-4936(7)	-143(5)	176(8)
C(24P)	-2694(8)	-5264(7)	141(8)	151(7)
P(5)	-9130(2)	-3311(2)	-3091(2)	117(2)
C(25P)	-9432(7)	-2865(6)	-3139(9)	157(7)
C(26P)	-9265(10)	-2545(9)	-3439(10)	185(8)
C(27P)	-8692(5)	-3112(7)	-2826(6)	128(5)
C(28P)	-8771(9)	-2922(9)	-2399(6)	156(7)
C(29P)	-9402(7)	-3593(7)	-2696(8)	154(7)
C(30P)	-9164(9)	-3967(8)	-2538(9)	173(8)
P(6)	-9588(2)	-3912(2)	-3822(3)	165(3)
C(31P)	-9583(8)	-4246(10)	-4261(9)	230(11)
C(32P)	-9971(10)	-4507(9)	-4287(9)	215(9)
C(33P)	-9956(5)	-3533(8)	-3760(10)	212(9)
C(34P)	-9826(8)	-3264(12)	-4133(11)	232(11)
C(35P)	-9785(10)	-4278(9)	-3450(11)	227(10)
C(36P)	-9513(10)	-4654(10)	-3447(9)	210(10)
P(1A)	-10379(2)	-2989(2)	-2112(2)	107(1)
F(1A)	-10815(4)	-3211(5)	-2185(5)	162(4)
F(2A)	-9951(5)	-2810(5)	-2086(5)	166(4)
F(3A)	-10472(6)	-2892(5)	-1671(5)	179(4)
F(4A)	-10220(5)	-3417(5)	-2010(6)	180(4)
F(5A)	-10573(5)	-2574(5)	-2258(5)	170(4)
F(6A)	-10266(4)	-3106(5)	-2584(4)	153(4)
P(2B)	-7944(2)	-2048(2)	-3028(2)	107(1)
F(1B)	-7553(4)	-2105(3)	-3297(3)	123(3)
F(2B)	-8213(4)	-2158(5)	-3409(4)	156(4)
F(3B)	-7928(4)	-2510(4)	-2910(4)	137(3)
F(4B)	-7672(3)	-1945(3)	-2617(4)	119(3)
F(5B)	-7953(3)	-1577(3)	-3135(4)	121(3)
F(6B)	-8339(4)	-1989(4)	-2727(5)	148(4)
P(3C)	-3843(2)	-4813(2)	1003(2)	130(2)
F(1C)	-4231(5)	-4866(5)	1285(5)	179(4)
F(2C)	-4046(4)	-4396(4)	828(4)	140(3)
F(3C)	-3663(4)	-4548(5)	1366(4)	157(4)
F(4C)	-4060(6)	-4991(6)	601(6)	202(5)
F(5C)	-3429(4)	-4732(4)	743(4)	145(4)
F(6C)	-3699(6)	-5248(5)	1126(6)	195(5)
P(4D)	-7793(4)	-4560(4)	-2516(4)	208(4)

F(1D)	-7579(6)	-4408(5)	-2090(6)	183(4)
F(2D)	-7522(9)	-4953(7)	-2534(8)	239(6)
F(3D)	-7409(6)	-4353(6)	-2802(6)	193(5)
F(4D)	-8121(8)	-4735(8)	-2246(9)	252(6)
F(5D)	-8006(8)	-4737(8)	-2880(9)	257(6)
F(6D)	-8044(6)	-4156(7)	-2532(6)	208(5)
P(5E)	-1676(2)	-4198(2)	4(2)	125(2)
F(1E)	-1578(7)	-3925(7)	391(6)	212(5)
F(2E)	-2086(6)	-3993(6)	38(6)	221(5)
F(3E)	-1532(6)	-3890(6)	-274(7)	228(6)
F(4E)	-1867(6)	-4511(7)	341(8)	246(6)
F(5E)	-1880(8)	-4436(8)	-292(8)	249(6)
F(6E)	-1324(6)	-4443(6)	-24(7)	218(5)
P(6F)	-2630(3)	-1576(3)	-4833(2)	162(3)
F(1F)	-2539(7)	-1510(7)	-4363(4)	217(5)
F(2F)	-3001(6)	-1818(7)	-4682(7)	226(5)
F(3F)	-2715(7)	-1683(7)	-5293(4)	205(5)
F(4F)	-2321(5)	-1264(5)	-4960(6)	192(5)
F(5F)	-2914(7)	-1218(6)	-4912(7)	231(5)
F(6F)	-2334(6)	-1925(6)	-4798(7)	228(6)

Table S3. Bond lengths [Å] and angles [°] for **3a**.

Pt(1)-N(3)	2.059(11)
Pt(1)-N(4)#1	2.109(10)
Pt(1)-P(1)	2.273(3)
Pt(1)-P(2)	2.278(3)
Pt(2)-N(5)#2	2.083(12)
Pt(2)-N(1)	2.106(10)
Pt(2)-P(4)	2.270(4)
Pt(2)-P(3)	2.279(4)
Pt(3)-N(2)	2.088(11)
Pt(3)-N(6)#3	2.082(15)
Pt(3)-P(5)	2.276(5)
Pt(3)-P(6)	2.285(6)
N(1)-C(20)	1.290(17)
N(1)-C(18)	1.337(19)
N(2)-C(47)	1.33(2)
N(2)-C(46)	1.35(2)
N(3)-C(48)	1.331(18)
N(3)-C(59)	1.332(19)
N(4)-C(52)	1.307(19)
N(4)-C(51)	1.329(18)
N(4)-Pt(1)#3	2.109(10)
N(5)-C(68)	1.31(2)
N(5)-C(56)	1.360(18)
N(5)-Pt(2)#2	2.083(12)
N(6)-C(70)	1.28(3)
N(6)-C(72)	1.37(2)
N(6)-Pt(3)#1	2.082(15)
C(1)-C(5)	1.389(18)
C(1)-C(2)	1.395(19)
C(2)-C(4)	1.40(2)
C(2)-C(3)	1.472(17)
C(3)-C(17)	1.352(19)
C(3)-C(19)	1.368(19)
C(4)-C(7)	1.392(18)
C(5)-C(6)	1.365(19)
C(6)-C(7)	1.365(19)

C(6)-C(8)	1.513(17)
C(8)-C(15)	1.368(17)
C(8)-C(16)	1.419(18)
C(9)-C(10)	1.370(19)
C(9)-C(66)	1.380(17)
C(10)-C(14)	1.39(2)
C(10)-C(15)	1.482(18)
C(11)-C(13)	1.327(19)
C(11)-C(66)	1.439(18)
C(11)-C(12)	1.453(19)
C(12)-C(43)	1.329(19)
C(12)-C(44)	1.46(2)
C(13)-C(14)	1.43(2)
C(15)-C(23)	1.451(17)
C(16)-C(22)	1.372(17)
C(16)-C(30)	1.532(17)
C(17)-C(20)	1.384(19)
C(18)-C(19)	1.397(19)
C(21)-C(62)	1.37(2)
C(21)-C(63)	1.44(2)
C(21)-C(36)	1.482(18)
C(22)-C(36)	1.410(18)
C(22)-C(28)	1.509(18)
C(23)-C(36)	1.362(18)
C(23)-C(37)	1.476(17)
C(24)-C(25)	1.39(2)
C(24)-C(29)	1.42(2)
C(25)-C(26)	1.35(2)
C(25)-C(60)	1.43(2)
C(26)-C(27)	1.40(2)
C(27)-C(28)	1.33(2)
C(28)-C(29)	1.39(2)
C(30)-C(32)	1.42(2)
C(30)-C(31)	1.40(2)
C(31)-C(34)	1.378(18)
C(32)-C(33)	1.426(18)
C(33)-C(35)	1.38(2)
C(34)-C(35)	1.41(2)

C(35)-C(49)	1.511(17)
C(37)-C(39)	1.38(2)
C(37)-C(57)	1.44(2)
C(38)-C(39)	1.373(19)
C(38)-C(41)	1.40(2)
C(40)-C(57)	1.401(19)
C(40)-C(41)	1.38(2)
C(41)-C(42)	1.475(19)
C(42)-C(45)	1.42(2)
C(42)-C(58)	1.44(2)
C(43)-C(56)	1.403(19)
C(44)-C(68)	1.38(2)
C(45)-C(46)	1.45(2)
C(47)-C(58)	1.34(2)
C(48)-C(67)	1.431(19)
C(49)-C(50)	1.326(19)
C(49)-C(67)	1.37(2)
C(50)-C(59)	1.441(18)
C(51)-C(53)	1.386(19)
C(52)-C(64)	1.385(19)
C(53)-C(54)	1.47(2)
C(54)-C(64)	1.37(2)
C(54)-C(55)	1.493(19)
C(55)-C(65)	1.40(2)
C(55)-C(69)	1.40(2)
C(60)-C(61)	1.37(3)
C(60)-C(71)	1.23(3)
C(61)-C(72)	1.42(3)
C(62)-C(65)	1.38(2)
C(63)-C(69)	1.38(2)
C(70)-C(71)	1.48(3)
P(1)-C(1P)	1.782(12)
P(1)-C(5P)	1.786(11)
P(1)-C(3P)	1.833(12)
C(1P)-C(2P)	1.515(14)
C(3P)-C(4P)	1.533(14)
C(5P)-C(6P)	1.510(13)
P(2)-C(11P)	1.797(11)

P(2)-C(9P)	1.814(10)
P(2)-C(7P)	1.832(10)
C(7P)-C(8P)	1.522(13)
C(9P)-C(10P)	1.521(13)
C(11P)-C(12P)	1.540(13)
P(3)-C(17P)	1.784(12)
P(3)-C(15P)	1.817(12)
P(3)-C(13P)	1.819(13)
C(13P)-C(14P)	1.538(15)
C(15P)-C(16P)	1.513(14)
C(17P)-C(18P)	1.514(15)
P(4)-C(19P)	1.806(14)
P(4)-C(23P)	1.807(13)
P(4)-C(21P)	1.806(14)
C(19P)-C(20P)	1.553(16)
C(21P)-C(22P)	1.532(16)
C(23P)-C(24P)	1.521(15)
P(5)-C(25P)	1.785(13)
P(5)-C(27P)	1.797(13)
P(5)-C(29P)	1.804(13)
C(25P)-C(26P)	1.527(15)
C(27P)-C(28P)	1.522(14)
C(29P)-C(30P)	1.547(15)
P(6)-C(33P)	1.753(14)
P(6)-C(31P)	1.783(14)
P(6)-C(35P)	1.814(14)
C(31P)-C(32P)	1.546(15)
C(33P)-C(34P)	1.543(15)
C(35P)-C(36P)	1.531(16)
P(1A)-F(3A)	1.474(16)
P(1A)-F(4A)	1.540(16)
P(1A)-F(2A)	1.531(15)
P(1A)-F(5A)	1.582(15)
P(1A)-F(6A)	1.601(15)
P(1A)-F(1A)	1.631(15)
P(2B)-F(2B)	1.547(14)
P(2B)-F(1B)	1.559(12)
P(2B)-F(3B)	1.569(13)

P(2B)-F(5B)	1.590(12)
P(2B)-F(4B)	1.624(12)
P(2B)-F(6B)	1.633(13)
P(3C)-F(6C)	1.560(17)
P(3C)-F(4C)	1.582(18)
P(3C)-F(1C)	1.575(15)
P(3C)-F(3C)	1.568(15)
P(3C)-F(5C)	1.619(13)
P(3C)-F(2C)	1.629(14)
P(4D)-F(5D)	1.48(2)
P(4D)-F(2D)	1.57(3)
P(4D)-F(6D)	1.57(2)
P(4D)-F(4D)	1.50(3)
P(4D)-F(1D)	1.61(2)
P(4D)-F(3D)	1.70(2)
P(5E)-F(6E)	1.42(2)
P(5E)-F(3E)	1.426(19)
P(5E)-F(5E)	1.40(2)
P(5E)-F(1E)	1.563(19)
P(5E)-F(2E)	1.51(2)
P(5E)-F(4E)	1.62(2)
P(6F)-F(4F)	1.505(12)
P(6F)-F(6F)	1.513(14)
P(6F)-F(5F)	1.529(14)
P(6F)-F(3F)	1.535(13)
P(6F)-F(2F)	1.535(13)
P(6F)-F(1F)	1.546(13)

N(3)-Pt(1)-N(4)#1	81.1(4)
N(3)-Pt(1)-P(1)	169.7(3)
N(4)#1-Pt(1)-P(1)	88.6(3)
N(3)-Pt(1)-P(2)	90.7(3)
N(4)#1-Pt(1)-P(2)	171.8(3)
P(1)-Pt(1)-P(2)	99.60(13)
N(5)#2-Pt(2)-N(1)	80.9(4)
N(5)#2-Pt(2)-P(4)	91.0(3)
N(1)-Pt(2)-P(4)	171.8(3)
N(5)#2-Pt(2)-P(3)	168.9(3)

N(1)-Pt(2)-P(3)	90.2(3)
P(4)-Pt(2)-P(3)	97.99(17)
N(2)-Pt(3)-N(6)#3	79.9(5)
N(2)-Pt(3)-P(5)	91.9(3)
N(6)#3-Pt(3)-P(5)	171.2(4)
N(2)-Pt(3)-P(6)	171.1(4)
N(6)#3-Pt(3)-P(6)	91.2(4)
P(5)-Pt(3)-P(6)	97.0(3)
C(20)-N(1)-C(18)	120.3(11)
C(20)-N(1)-Pt(2)	120.3(9)
C(18)-N(1)-Pt(2)	119.4(9)
C(47)-N(2)-C(46)	119.6(13)
C(47)-N(2)-Pt(3)	124.2(10)
C(46)-N(2)-Pt(3)	115.9(12)
C(48)-N(3)-C(59)	119.9(12)
C(48)-N(3)-Pt(1)	120.3(9)
C(59)-N(3)-Pt(1)	119.8(9)
C(52)-N(4)-C(51)	121.6(11)
C(52)-N(4)-Pt(1)#3	119.0(10)
C(51)-N(4)-Pt(1)#3	117.3(9)
C(68)-N(5)-C(56)	115.6(12)
C(68)-N(5)-Pt(2)#2	123.2(10)
C(56)-N(5)-Pt(2)#2	119.5(11)
C(70)-N(6)-C(72)	118.2(17)
C(70)-N(6)-Pt(3)#1	120.0(15)
C(72)-N(6)-Pt(3)#1	120.6(13)
C(5)-C(1)-C(2)	122.5(13)
C(1)-C(2)-C(4)	116.9(12)
C(1)-C(2)-C(3)	122.4(12)
C(4)-C(2)-C(3)	120.7(12)
C(17)-C(3)-C(19)	119.5(12)
C(17)-C(3)-C(2)	122.1(13)
C(19)-C(3)-C(2)	118.5(12)
C(7)-C(4)-C(2)	120.4(13)
C(1)-C(5)-C(6)	118.5(13)
C(5)-C(6)-C(7)	121.5(12)
C(5)-C(6)-C(8)	120.3(12)
C(7)-C(6)-C(8)	118.2(13)

C(4)-C(7)-C(6)	120.2(13)
C(15)-C(8)-C(16)	118.8(11)
C(15)-C(8)-C(6)	120.2(11)
C(16)-C(8)-C(6)	120.7(10)
C(10)-C(9)-C(66)	124.1(13)
C(9)-C(10)-C(14)	116.0(12)
C(9)-C(10)-C(15)	120.3(12)
C(14)-C(10)-C(15)	123.2(12)
C(13)-C(11)-C(66)	118.4(12)
C(13)-C(11)-C(12)	124.4(12)
C(66)-C(11)-C(12)	117.2(11)
C(43)-C(12)-C(44)	115.5(13)
C(43)-C(12)-C(11)	124.8(12)
C(44)-C(12)-C(11)	119.7(13)
C(11)-C(13)-C(14)	121.6(14)
C(10)-C(14)-C(13)	121.1(13)
C(8)-C(15)-C(23)	119.1(11)
C(8)-C(15)-C(10)	120.7(11)
C(23)-C(15)-C(10)	120.0(10)
C(22)-C(16)-C(8)	122.5(11)
C(22)-C(16)-C(30)	118.0(11)
C(8)-C(16)-C(30)	119.4(11)
C(3)-C(17)-C(20)	118.5(13)
N(1)-C(18)-C(19)	119.8(13)
C(3)-C(19)-C(18)	119.1(13)
N(1)-C(20)-C(17)	122.7(13)
C(62)-C(21)-C(63)	118.3(12)
C(62)-C(21)-C(36)	120.1(12)
C(63)-C(21)-C(36)	121.5(12)
C(16)-C(22)-C(36)	118.1(12)
C(16)-C(22)-C(28)	120.4(11)
C(36)-C(22)-C(28)	121.4(11)
C(36)-C(23)-C(15)	120.1(11)
C(36)-C(23)-C(37)	120.3(11)
C(15)-C(23)-C(37)	119.6(11)
C(25)-C(24)-C(29)	122.7(16)
C(26)-C(25)-C(24)	115.3(15)
C(26)-C(25)-C(60)	125.0(16)

C(24)-C(25)-C(60)	119.2(15)
C(25)-C(26)-C(27)	121.5(17)
C(28)-C(27)-C(26)	122.9(15)
C(27)-C(28)-C(29)	118.2(14)
C(27)-C(28)-C(22)	123.6(13)
C(29)-C(28)-C(22)	117.8(14)
C(28)-C(29)-C(24)	118.0(16)
C(32)-C(30)-C(31)	121.9(11)
C(32)-C(30)-C(16)	117.5(12)
C(31)-C(30)-C(16)	120.3(13)
C(34)-C(31)-C(30)	119.2(14)
C(30)-C(32)-C(33)	117.7(14)
C(35)-C(33)-C(32)	119.4(14)
C(31)-C(34)-C(35)	119.9(14)
C(33)-C(35)-C(34)	121.7(11)
C(33)-C(35)-C(49)	119.2(12)
C(34)-C(35)-C(49)	119.0(12)
C(23)-C(36)-C(22)	121.1(11)
C(23)-C(36)-C(21)	120.3(11)
C(22)-C(36)-C(21)	118.5(12)
C(39)-C(37)-C(57)	119.3(12)
C(39)-C(37)-C(23)	123.6(12)
C(57)-C(37)-C(23)	117.0(12)
C(39)-C(38)-C(41)	121.6(15)
C(37)-C(39)-C(38)	120.1(14)
C(57)-C(40)-C(41)	119.9(13)
C(38)-C(41)-C(40)	119.6(12)
C(38)-C(41)-C(42)	120.7(14)
C(40)-C(41)-C(42)	119.4(12)
C(45)-C(42)-C(41)	117.6(13)
C(45)-C(42)-C(58)	120.1(13)
C(41)-C(42)-C(58)	122.3(15)
C(12)-C(43)-C(56)	121.5(14)
C(68)-C(44)-C(12)	118.9(15)
C(42)-C(45)-C(46)	116.5(15)
N(2)-C(46)-C(45)	120.6(17)
N(2)-C(47)-C(58)	126.1(16)
N(3)-C(48)-C(67)	121.6(13)

C(50)-C(49)-C(67)	122.1(12)
C(50)-C(49)-C(35)	121.3(12)
C(67)-C(49)-C(35)	116.5(12)
C(49)-C(50)-C(59)	118.5(13)
N(4)-C(51)-C(53)	122.7(13)
N(4)-C(52)-C(64)	121.4(14)
C(51)-C(53)-C(54)	115.6(14)
C(64)-C(54)-C(53)	118.4(12)
C(64)-C(54)-C(55)	123.2(13)
C(53)-C(54)-C(55)	118.2(13)
C(65)-C(55)-C(69)	120.2(13)
C(65)-C(55)-C(54)	119.7(14)
C(69)-C(55)-C(54)	119.9(14)
N(5)-C(56)-C(43)	123.4(14)
C(40)-C(57)-C(37)	119.5(13)
C(47)-C(58)-C(42)	116.5(16)
N(3)-C(59)-C(50)	120.7(13)
C(61)-C(60)-C(71)	115.5(19)
C(61)-C(60)-C(25)	119.6(17)
C(71)-C(60)-C(25)	124.0(19)
C(60)-C(61)-C(72)	120.2(19)
C(65)-C(62)-C(21)	121.1(15)
C(69)-C(63)-C(21)	120.8(14)
C(54)-C(64)-C(52)	119.8(14)
C(62)-C(65)-C(55)	120.4(15)
C(9)-C(66)-C(11)	118.7(12)
C(49)-C(67)-C(48)	117.0(13)
N(5)-C(68)-C(44)	125.1(14)
C(63)-C(69)-C(55)	118.9(14)
N(6)-C(70)-C(71)	119(2)
C(60)-C(71)-C(70)	125(2)
N(6)-C(72)-C(61)	120.6(19)
C(1P)-P(1)-C(5P)	106.2(10)
C(1P)-P(1)-C(3P)	107.1(8)
C(5P)-P(1)-C(3P)	104.2(9)
C(1P)-P(1)-Pt(1)	108.9(7)
C(5P)-P(1)-Pt(1)	121.5(5)
C(3P)-P(1)-Pt(1)	108.1(6)

C(2P)-C(1P)-P(1)	114.7(12)
C(4P)-C(3P)-P(1)	116.6(13)
C(6P)-C(5P)-P(1)	115.5(11)
C(11P)-P(2)-C(9P)	107.7(6)
C(11P)-P(2)-C(7P)	105.6(7)
C(9P)-P(2)-C(7P)	102.3(7)
C(11P)-P(2)-Pt(1)	114.7(5)
C(9P)-P(2)-Pt(1)	113.3(5)
C(7P)-P(2)-Pt(1)	112.1(4)
C(8P)-C(7P)-P(2)	114.5(10)
C(10P)-C(9P)-P(2)	115.6(10)
C(12P)-C(11P)-P(2)	111.5(9)
C(17P)-P(3)-C(15P)	105.1(11)
C(17P)-P(3)-C(13P)	105.6(9)
C(15P)-P(3)-C(13P)	103.9(12)
C(17P)-P(3)-Pt(2)	119.8(7)
C(15P)-P(3)-Pt(2)	112.7(5)
C(13P)-P(3)-Pt(2)	108.4(9)
C(14P)-C(13P)-P(3)	113.5(17)
C(16P)-C(15P)-P(3)	119.0(12)
C(18P)-C(17P)-P(3)	107.4(14)
C(19P)-P(4)-C(23P)	124.1(14)
C(19P)-P(4)-C(21P)	94.5(16)
C(23P)-P(4)-C(21P)	92.7(15)
C(19P)-P(4)-Pt(2)	107.3(10)
C(23P)-P(4)-Pt(2)	120.5(7)
C(21P)-P(4)-Pt(2)	112.4(10)
C(20P)-C(19P)-P(4)	97(2)
C(22P)-C(21P)-P(4)	111(2)
C(24P)-C(23P)-P(4)	117.3(16)
C(25P)-P(5)-C(27P)	100.9(13)
C(25P)-P(5)-C(29P)	102.0(11)
C(27P)-P(5)-C(29P)	105.0(12)
C(25P)-P(5)-Pt(3)	119.1(9)
C(27P)-P(5)-Pt(3)	111.8(6)
C(29P)-P(5)-Pt(3)	116.1(9)
C(26P)-C(25P)-P(5)	115.0(17)
C(28P)-C(27P)-P(5)	115.7(16)

C(30P)-C(29P)-P(5)	112.7(16)
C(33P)-P(6)-C(31P)	122.4(17)
C(33P)-P(6)-C(35P)	98.8(15)
C(31P)-P(6)-C(35P)	96.2(18)
C(33P)-P(6)-Pt(3)	110.4(9)
C(31P)-P(6)-Pt(3)	111.8(9)
C(35P)-P(6)-Pt(3)	115.8(13)
C(32P)-C(31P)-P(6)	112.2(15)
C(34P)-C(33P)-P(6)	97.5(15)
C(36P)-C(35P)-P(6)	109(2)
F(3A)-P(1A)-F(4A)	94.0(10)
F(3A)-P(1A)-F(2A)	93.3(10)
F(4A)-P(1A)-F(2A)	91.5(10)
F(3A)-P(1A)-F(5A)	90.5(10)
F(4A)-P(1A)-F(5A)	173.2(10)
F(2A)-P(1A)-F(5A)	93.3(9)
F(3A)-P(1A)-F(6A)	177.9(11)
F(4A)-P(1A)-F(6A)	84.1(9)
F(2A)-P(1A)-F(6A)	86.0(9)
F(5A)-P(1A)-F(6A)	91.5(9)
F(3A)-P(1A)-F(1A)	92.9(10)
F(4A)-P(1A)-F(1A)	85.2(9)
F(2A)-P(1A)-F(1A)	173.2(9)
F(5A)-P(1A)-F(1A)	89.4(9)
F(6A)-P(1A)-F(1A)	87.7(8)
F(2B)-P(2B)-F(1B)	90.9(8)
F(2B)-P(2B)-F(3B)	89.0(8)
F(1B)-P(2B)-F(3B)	89.4(7)
F(2B)-P(2B)-F(5B)	92.8(7)
F(1B)-P(2B)-F(5B)	90.8(7)
F(3B)-P(2B)-F(5B)	178.3(8)
F(2B)-P(2B)-F(4B)	177.7(9)
F(1B)-P(2B)-F(4B)	90.7(6)
F(3B)-P(2B)-F(4B)	89.4(6)
F(5B)-P(2B)-F(4B)	88.8(6)
F(2B)-P(2B)-F(6B)	91.8(8)
F(1B)-P(2B)-F(6B)	177.2(8)
F(3B)-P(2B)-F(6B)	90.1(8)

F(5B)-P(2B)-F(6B)	89.7(7)
F(4B)-P(2B)-F(6B)	86.5(7)
F(6C)-P(3C)-F(4C)	90.0(12)
F(6C)-P(3C)-F(1C)	90.1(9)
F(4C)-P(3C)-F(1C)	93.1(11)
F(6C)-P(3C)-F(3C)	102.3(10)
F(4C)-P(3C)-F(3C)	167.7(11)
F(1C)-P(3C)-F(3C)	87.1(9)
F(6C)-P(3C)-F(5C)	91.5(9)
F(4C)-P(3C)-F(5C)	91.5(9)
F(1C)-P(3C)-F(5C)	175.2(10)
F(3C)-P(3C)-F(5C)	88.1(8)
F(6C)-P(3C)-F(2C)	170.8(11)
F(4C)-P(3C)-F(2C)	81.2(10)
F(1C)-P(3C)-F(2C)	87.4(8)
F(3C)-P(3C)-F(2C)	86.5(8)
F(5C)-P(3C)-F(2C)	91.7(8)
F(5D)-P(4D)-F(2D)	85.2(16)
F(5D)-P(4D)-F(6D)	93.2(14)
F(2D)-P(4D)-F(6D)	175.1(16)
F(5D)-P(4D)-F(4D)	87.4(15)
F(2D)-P(4D)-F(4D)	96.6(15)
F(6D)-P(4D)-F(4D)	87.9(15)
F(5D)-P(4D)-F(1D)	173.9(15)
F(2D)-P(4D)-F(1D)	92.4(12)
F(6D)-P(4D)-F(1D)	89.6(12)
F(4D)-P(4D)-F(1D)	87.3(15)
F(5D)-P(4D)-F(3D)	95.1(15)
F(2D)-P(4D)-F(3D)	83.6(14)
F(6D)-P(4D)-F(3D)	92.0(11)
F(4D)-P(4D)-F(3D)	177.5(15)
F(1D)-P(4D)-F(3D)	90.2(10)
F(6E)-P(5E)-F(3E)	95.4(9)
F(6E)-P(5E)-F(5E)	91.8(15)
F(3E)-P(5E)-F(5E)	98.0(16)
F(6E)-P(5E)-F(1E)	102.0(13)
F(3E)-P(5E)-F(1E)	90.8(13)
F(5E)-P(5E)-F(1E)	162.9(16)

F(6E)-P(5E)-F(2E)	171.7(12)
F(3E)-P(5E)-F(2E)	91.4(8)
F(5E)-P(5E)-F(2E)	82.5(14)
F(1E)-P(5E)-F(2E)	82.6(12)
F(6E)-P(5E)-F(4E)	89.8(8)
F(3E)-P(5E)-F(4E)	174.2(12)
F(5E)-P(5E)-F(4E)	84.4(15)
F(1E)-P(5E)-F(4E)	85.4(13)
F(2E)-P(5E)-F(4E)	83.7(8)
F(4F)-P(6F)-F(6F)	95.9(13)
F(4F)-P(6F)-F(5F)	80.8(12)
F(6F)-P(6F)-F(5F)	174.4(14)
F(4F)-P(6F)-F(3F)	91.3(11)
F(6F)-P(6F)-F(3F)	90.8(14)
F(5F)-P(6F)-F(3F)	84.7(13)
F(4F)-P(6F)-F(2F)	168.0(15)
F(6F)-P(6F)-F(2F)	95.5(13)
F(5F)-P(6F)-F(2F)	88.1(14)
F(3F)-P(6F)-F(2F)	92.1(12)
F(4F)-P(6F)-F(1F)	91.8(12)
F(6F)-P(6F)-F(1F)	84.9(13)
F(5F)-P(6F)-F(1F)	99.7(14)
F(3F)-P(6F)-F(1F)	174.9(14)
F(2F)-P(6F)-F(1F)	85.6(12)

Symmetry transformations used to generate equivalent atoms:

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

Table S4. Anisotropic displacement parameters ($Å^2$x 10^3) for **3a**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Pt(1)	56(1)	85(1)	71(1)	-14(1)	-4(1)	-18(1)
Pt(2)	111(1)	62(1)	90(1)	0(1)	-52(1)	2(1)
Pt(3)	50(1)	139(1)	115(1)	-38(1)	15(1)	7(1)
N(1)	86(6)	68(6)	62(5)	-4(4)	-12(4)	0(5)
N(2)	58(4)	117(8)	83(7)	-25(6)	8(5)	18(4)
N(3)	72(5)	88(7)	78(6)	-6(5)	-13(5)	-18(4)
N(4)	91(6)	78(6)	60(5)	-6(5)	-14(5)	23(6)
N(5)	75(6)	70(6)	98(7)	-8(5)	32(5)	5(5)
N(6)	113(8)	56(6)	141(10)	8(6)	4(7)	-19(6)
C(1)	61(7)	76(6)	80(8)	11(5)	-10(5)	-3(6)
C(2)	80(7)	81(6)	56(6)	4(5)	-10(5)	3(6)
C(3)	70(6)	77(7)	58(6)	-3(5)	-13(5)	6(5)
C(4)	69(7)	85(6)	65(7)	-1(5)	-13(5)	4(6)
C(5)	62(7)	86(6)	79(8)	9(6)	-14(5)	-3(6)
C(6)	57(6)	92(6)	61(6)	21(5)	-5(4)	-4(6)
C(7)	75(7)	79(6)	64(7)	14(5)	-9(5)	2(6)
C(8)	56(5)	94(8)	69(7)	25(5)	-3(4)	-1(5)
C(9)	51(6)	79(7)	73(5)	2(5)	13(5)	1(5)
C(10)	48(6)	101(8)	66(5)	9(5)	-2(5)	-3(5)
C(11)	57(6)	72(6)	73(5)	-3(5)	1(5)	4(5)
C(12)	63(6)	66(6)	78(6)	-7(5)	14(5)	3(5)
C(13)	68(7)	95(8)	78(6)	-1(6)	6(6)	-12(6)
C(14)	70(8)	111(9)	76(7)	-3(6)	0(6)	-19(7)
C(15)	52(5)	89(7)	62(6)	5(5)	-4(4)	-6(5)
C(16)	54(5)	98(8)	71(7)	23(5)	-12(5)	-8(5)
C(17)	75(6)	93(9)	68(7)	12(6)	-8(5)	-8(6)
C(18)	89(6)	88(9)	75(8)	23(6)	-12(6)	-4(7)
C(19)	68(6)	90(9)	68(7)	11(5)	-4(5)	8(6)
C(20)	74(7)	71(7)	72(7)	1(5)	-15(5)	-1(6)
C(21)	54(6)	93(8)	77(6)	10(5)	2(5)	15(5)
C(22)	59(5)	89(7)	71(7)	15(5)	-1(4)	8(5)
C(23)	56(5)	92(8)	60(6)	1(5)	1(4)	2(5)
C(24)	135(13)	107(8)	123(12)	23(9)	60(10)	14(9)

C(25)	92(9)	92(7)	85(8)	6(6)	18(6)	0(7)
C(26)	139(13)	95(8)	90(10)	25(7)	31(8)	16(8)
C(27)	110(11)	98(7)	76(8)	15(7)	17(7)	8(8)
C(28)	66(6)	92(6)	55(6)	17(5)	-3(4)	0(5)
C(29)	132(13)	97(8)	116(12)	16(8)	54(9)	12(9)
C(30)	48(5)	99(7)	74(7)	-2(6)	3(5)	-9(4)
C(31)	51(5)	105(7)	85(8)	12(7)	-5(6)	-16(5)
C(32)	56(5)	108(7)	102(9)	18(8)	-7(6)	-9(5)
C(33)	58(5)	97(7)	90(8)	-7(7)	-6(6)	-13(5)
C(34)	51(5)	99(7)	87(8)	1(7)	-5(6)	-4(5)
C(35)	51(5)	106(7)	68(7)	-2(6)	-6(5)	-15(4)
C(36)	57(5)	86(8)	68(6)	7(5)	2(4)	13(5)
C(37)	56(5)	105(8)	57(7)	7(5)	-3(4)	3(5)
C(38)	56(5)	131(11)	91(9)	-26(8)	10(6)	-3(6)
C(39)	58(5)	119(10)	92(9)	-28(7)	8(6)	1(6)
C(40)	56(5)	109(9)	89(9)	-9(7)	-9(6)	22(5)
C(41)	57(5)	121(9)	59(7)	3(6)	1(5)	9(5)
C(42)	59(5)	111(8)	76(8)	-12(6)	-5(5)	10(5)
C(43)	75(7)	73(7)	74(6)	-1(5)	22(5)	3(6)
C(44)	73(7)	97(9)	94(8)	-3(6)	20(6)	-4(6)
C(45)	68(6)	115(9)	108(10)	5(8)	6(7)	12(6)
C(46)	68(6)	136(10)	111(11)	14(9)	10(7)	13(6)
C(47)	55(5)	129(9)	109(11)	0(9)	0(7)	14(6)
C(48)	67(5)	96(9)	89(9)	4(6)	-1(6)	-29(6)
C(49)	66(5)	79(7)	67(7)	-4(5)	-7(5)	-13(4)
C(50)	53(5)	100(8)	66(7)	2(6)	-8(5)	-15(5)
C(51)	91(7)	66(7)	75(7)	12(6)	-14(5)	8(6)
C(52)	92(8)	102(10)	65(7)	-3(7)	-12(5)	35(8)
C(53)	83(7)	87(9)	82(7)	18(7)	-12(5)	-3(7)
C(54)	77(6)	82(8)	75(6)	10(6)	-16(5)	18(6)
C(55)	75(7)	94(8)	79(6)	16(6)	-15(6)	6(6)
C(56)	89(7)	81(8)	65(6)	-9(5)	14(6)	3(6)
C(57)	67(5)	91(8)	94(9)	-8(7)	-8(6)	3(6)
C(58)	67(6)	108(9)	103(10)	-12(7)	11(7)	17(5)
C(59)	63(5)	85(8)	93(9)	-9(6)	-9(6)	-8(6)
C(60)	89(8)	102(9)	105(9)	25(7)	23(6)	8(7)
C(61)	130(10)	139(14)	97(10)	2(8)	3(8)	-56(11)
C(62)	90(9)	125(11)	72(6)	5(7)	-9(7)	39(8)

C(63)	98(10)	97(9)	76(7)	5(6)	-10(6)	24(7)
C(64)	83(7)	98(9)	74(7)	12(7)	-14(5)	12(7)
C(65)	88(9)	113(10)	77(7)	7(6)	-14(6)	30(7)
C(66)	63(6)	60(6)	66(6)	7(4)	9(5)	13(5)
C(67)	63(6)	108(9)	84(9)	15(6)	-13(6)	-23(6)
C(68)	73(8)	61(7)	114(9)	0(6)	18(6)	-8(6)
C(69)	88(9)	94(9)	83(7)	2(6)	-21(7)	24(7)
C(70)	118(10)	181(16)	175(15)	96(13)	-7(10)	-34(12)
C(71)	122(11)	213(18)	153(14)	91(12)	3(9)	-22(12)
C(72)	114(11)	134(14)	120(12)	3(9)	-2(8)	-51(10)
P(1)	63(2)	107(3)	92(2)	-31(2)	8(2)	-17(2)
C(1P)	104(10)	123(8)	130(11)	-26(7)	40(10)	5(8)
C(2P)	115(9)	122(8)	130(9)	-4(7)	-17(8)	12(8)
C(3P)	79(9)	159(11)	107(8)	-7(9)	19(7)	-12(9)
C(4P)	133(10)	142(8)	120(9)	6(7)	11(8)	-16(8)
C(5P)	49(5)	129(11)	114(9)	-21(9)	-10(6)	-20(7)
C(6P)	71(5)	118(8)	101(8)	-5(7)	0(6)	4(6)
P(2)	63(2)	83(2)	73(2)	-11(2)	-5(2)	-10(2)
C(7P)	73(6)	99(10)	83(9)	-18(6)	7(6)	-6(6)
C(8P)	89(7)	106(8)	94(7)	-20(6)	10(6)	1(6)
C(9P)	69(7)	79(6)	92(8)	-15(5)	-6(6)	-10(6)
C(10P)	95(8)	103(7)	105(8)	-2(6)	-14(6)	2(7)
C(11P)	73(7)	91(8)	83(7)	-5(6)	-7(6)	-13(7)
C(12P)	85(7)	89(6)	76(6)	-6(5)	3(6)	-8(6)
P(3)	162(4)	79(2)	90(3)	-9(2)	-51(3)	8(3)
C(13P)	196(13)	181(17)	75(8)	-5(8)	-33(8)	-35(14)
C(14P)	187(9)	210(13)	201(13)	6(9)	-7(8)	-15(9)
C(15P)	198(17)	93(9)	97(10)	-15(7)	-45(11)	37(11)
C(16P)	147(10)	102(7)	123(9)	-11(7)	5(8)	12(7)
C(17P)	168(11)	92(11)	111(10)	-24(8)	-50(8)	-17(9)
P(4)	178(5)	95(3)	136(4)	-9(3)	-96(4)	31(3)
C(19P)	155(15)	209(17)	210(17)	-10(15)	-57(11)	66(9)
C(18P)	164(10)	170(11)	159(11)	8(9)	-7(8)	-3(8)
C(20P)	240(13)	245(15)	245(14)	12(10)	-12(9)	16(9)
C(21P)	242(19)	118(12)	200(20)	55(11)	-49(13)	23(11)
C(22P)	257(13)	250(15)	251(15)	10(10)	-6(9)	0(9)
C(23P)	220(20)	204(16)	106(9)	45(10)	-85(12)	15(15)
C(24P)	166(11)	153(11)	136(9)	21(8)	-37(8)	18(8)

P(5)	86(3)	147(4)	119(4)	-19(3)	35(2)	20(3)
C(25P)	131(13)	149(12)	189(18)	-24(9)	52(12)	41(11)
C(26P)	180(12)	179(10)	195(12)	15(8)	7(9)	12(9)
C(27P)	133(11)	121(13)	129(12)	-37(10)	17(9)	-8(9)
C(28P)	167(11)	168(11)	133(8)	-27(8)	20(8)	2(9)
C(29P)	124(13)	173(15)	165(14)	-5(11)	64(12)	4(10)
C(30P)	177(12)	175(10)	167(11)	11(8)	2(9)	-3(8)
P(6)	72(3)	167(5)	255(8)	-14(5)	1(4)	-27(3)
C(31P)	210(20)	240(20)	243(17)	-56(15)	-96(15)	-33(14)
C(32P)	210(11)	215(11)	221(11)	-15(7)	-11(7)	-23(7)
C(33P)	85(9)	315(19)	230(20)	42(14)	10(14)	61(14)
C(34P)	237(14)	233(12)	228(14)	12(9)	-5(9)	6(9)
C(35P)	152(17)	211(17)	320(20)	10(17)	50(20)	-57(10)
C(36P)	216(13)	205(11)	208(13)	5(9)	5(9)	-16(9)
P(1A)	108(3)	109(3)	104(3)	1(2)	-12(2)	-5(3)
F(1A)	120(5)	184(8)	183(8)	-10(7)	19(5)	-24(6)
F(2A)	145(6)	175(8)	179(8)	3(7)	-32(6)	-30(6)
F(3A)	215(9)	185(8)	137(5)	-8(6)	2(6)	-2(7)

Table S5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **3a**.

	x	y	z	U(eq)
H(1A)	-4667	-4327	-2715	87
H(4B)	-4277	-3224	-2336	88
H(5B)	-5046	-4025	-3248	91
H(7A)	-4684	-2927	-2851	87
H(9A)	-5560	-3251	-2627	81
H(13A)	-6296	-4402	-2960	96
H(14A)	-6004	-4005	-3495	103
H(17A)	-3634	-3767	-2323	95
H(18A)	-4230	-4618	-1356	101
H(19A)	-4633	-4303	-1871	91
H(20A)	-3269	-4056	-1776	87
H(24A)	-4262	-2524	-5039	146
H(26A)	-4897	-1590	-4616	130
H(27A)	-5230	-2017	-4156	113
H(29A)	-4639	-2974	-4615	138
H(31A)	-4344	-3524	-3783	97
H(32A)	-4562	-2310	-3632	106
H(33A)	-3845	-2194	-3613	98
H(34A)	-3651	-3408	-3700	95
H(38A)	-6965	-3773	-4084	111
H(39A)	-6268	-3703	-4106	107
H(40A)	-7073	-2753	-3386	102
H(43A)	-5793	-4232	-1720	89
H(44A)	-6744	-4555	-2443	106
H(45A)	-7471	-3919	-3458	116
H(46A)	-8195	-4012	-3372	126
H(47A)	-8403	-2919	-3805	117
H(48A)	-2530	-2111	-3933	101
H(50A)	-3135	-3165	-3205	88
H(51A)	-6619	-1843	-6307	93
H(52A)	-7527	-1853	-5517	104

H(53)	-6154	-2053	-5795	101
H(56A)	-6060	-4637	-1194	94
H(57A)	-6364	-2671	-3405	101
H(58A)	-7741	-2781	-3938	111
H(59A)	-2421	-3038	-3162	96
H(61)	-3856	-1744	-4918	147
H(62A)	-5684	-3031	-4858	115
H(63A)	-6118	-2230	-4000	108
H(64A)	-7109	-2059	-4969	102
H(65A)	-6034	-2690	-5380	111
H(66A)	-5793	-3665	-2096	76
H(67A)	-3242	-2230	-4000	102
H(68A)	-6972	-4955	-1900	99
H(69A)	-6529	-1934	-4511	106
H(70A)	-4504	-1282	-6097	190
H(71A)	-4847	-1622	-5533	195
H(72A)	-3496	-1396	-5451	147
H(1PA)	-1081	-3126	-3911	143
H(1PB)	-646	-2988	-3744	143
H(2PA)	-892	-3526	-3339	184
H(2PB)	-1285	-3267	-3218	184
H(2PC)	-849	-3131	-3051	184
H(3PA)	-703	-2198	-4144	138
H(3PB)	-1086	-2409	-4356	138
H(4PA)	-1123	-1711	-4469	198
H(4PB)	-1098	-1624	-3977	198
H(4PC)	-1491	-1838	-4170	198
H(5PA)	-866	-1954	-3241	117
H(5PB)	-844	-2364	-2977	117
H(6PA)	-191	-2133	-3125	145
H(6PB)	-284	-2149	-3617	145
H(6PC)	-262	-2560	-3352	145
H(7PA)	-2550	-1911	-3062	102
H(7PB)	-2489	-2275	-2737	102
H(8PA)	-2731	-1709	-2376	145
H(8PB)	-2362	-1439	-2545	145
H(8PC)	-2278	-1799	-2220	145
H(9PA)	-1728	-1381	-2860	96

H(9PB)	-1435	-1543	-3221	96
H(10A)	-1883	-1059	-3494	151
H(10B)	-2263	-1328	-3351	151
H(10C)	-1967	-1488	-3712	151
H(11A)	-1285	-2192	-2595	99
H(11B)	-1639	-2015	-2304	99
H(12A)	-1528	-2712	-2146	125
H(12B)	-1626	-2831	-2621	125
H(12C)	-1977	-2654	-2327	125
H(13B)	-3531	-4451	-63	181
H(13C)	-3648	-3982	-23	181
H(14B)	-4246	-4375	-49	299
H(14C)	-4112	-4564	-490	299
H(14D)	-4227	-4094	-457	299
H(15A)	-3441	-3579	-1146	155
H(15B)	-3867	-3784	-1043	155
H(16A)	-3874	-3121	-813	186
H(16B)	-3479	-3196	-533	186
H(16C)	-3897	-3411	-412	186
H(17B)	-2940	-3691	-278	148
H(17C)	-2727	-4123	-350	148
H(19B)	-2457	-5545	-873	230
H(19C)	-2726	-5425	-1279	230
H(18B)	-2404	-3618	-764	247
H(18C)	-2825	-3526	-994	247
H(18D)	-2605	-3954	-1060	247
H(20B)	-2095	-5030	-1275	365
H(20C)	-2221	-4848	-829	365
H(20D)	-2480	-4736	-1237	365
H(21A)	-3297	-5686	-792	226
H(21B)	-3035	-5707	-370	226
H(22A)	-3734	-5700	-201	379
H(22B)	-3789	-5280	-446	379
H(22C)	-3527	-5296	-24	379
H(23A)	-2675	-4704	-142	211
H(23B)	-3124	-4840	-19	211
H(24B)	-2675	-5160	428	227
H(24C)	-2424	-5341	43	227

H(24D)	-2873	-5501	136	227
H(25A)	-9706	-2944	-3237	188
H(25B)	-9463	-2741	-2859	188
H(26B)	-9456	-2319	-3459	277
H(26C)	-9226	-2665	-3717	277
H(26D)	-9004	-2445	-3333	277
H(27B)	-8494	-3335	-2790	154
H(27C)	-8565	-2905	-3009	154
H(28A)	-8514	-2830	-2278	234
H(28B)	-8895	-3123	-2213	234
H(28C)	-8955	-2690	-2431	234
H(29B)	-9460	-3412	-2456	185
H(29C)	-9665	-3683	-2813	185
H(30A)	-9331	-4118	-2338	260
H(30B)	-8914	-3877	-2400	260
H(30C)	-9096	-4141	-2776	260
H(31B)	-9556	-4084	-4521	276
H(31C)	-9344	-4426	-4241	276
H(32B)	-9971	-4660	-4550	323
H(32C)	-10210	-4331	-4277	323
H(32D)	-9978	-4696	-4050	323
H(33B)	-10235	-3638	-3793	254
H(33C)	-9930	-3390	-3489	254
H(34B)	-9944	-2993	-4102	348
H(34C)	-9921	-3386	-4395	348
H(34D)	-9529	-3243	-4139	348
H(35A)	-9793	-4156	-3166	273
H(35B)	-10065	-4355	-3528	273
H(36A)	-9621	-4853	-3248	314
H(36B)	-9238	-4576	-3363	314
H(36C)	-9506	-4773	-3728	314

Pulsed Field Gradient Spin Echo NMR Measurements of Truncated Tetrahedra 3a and 3b:

Pulsed gradient spin-echo (PGSE) NMR diffusion measurements were done by pulse-sequence developed by Stejskal and Tanner.[3]

$$\ln(I/I_0) = -\gamma_X{}^2\delta^2G^2(\Delta - \delta/3)D \qquad (1)$$

$$\gamma_X = \text{gyromagnetic ratio of the X nucleus;}$$
$$\delta = \text{length of the gradient pulse;} \quad G = \text{gradient strength;}$$
$$\Delta = \text{delay between the midpoints of the gradients;}$$
$$D = \text{diffusion coefficient}$$

Temp: 298K

Instrument: Inova 500 MHz

Stokes-Einstein Equation: The molecular size is obtained from the diffusion coefficient via the Stokes-Einstein equation where k_B is the Boltzmann constant, T is the absolute temperature and r_H is the hydrodynamic radius of the species under investigation. $D = \dfrac{k_B T}{6\pi\,\eta\,r}$

Gradient Calibration: The gradient strengths need to be carefully calibrated to obtain accurate D values to fit equation 1. Gradient strengths were calibrated using the width (in Hz) of a sample of known length along the NMR-tube (Z) axis, back-calculation of the coil constant from a diffusion experiment on D_2O using D = 1.9 × 10^{-5} cm^2/s for D_2O at 298K[4] was used to calculate the gradient strengths of both the probes.

Issue of Viscosity: The effect of variable viscosity in different batch of same solvents was examined using the D values observed for the residual protons of the solvent resonance.

Pulse sequence: Stejskal-Tanner pulse sequence

Diffusion Coefficients for **3a** and **3b** in Acetone-d_6:

D(**3a**) = 4.27 (± 0.095) X 10^{-6} cm^2/s
D(**3b**) = 4.41 (± 0.068) X 10^{-6} cm^2/s

Encapsulated Complex 3b·4$_3$. 90° organoplatinum acceptor **1b** (4.36 mg, 5.53 mmol), hexapyridyl donor **2** (1.83 mg, 1.84 mmol), and 1,3,5-triphenylbenzene **4** (3.31 mg, 10.8 mmol) were placed in a 2-dram vial followed by addition of 1.0 mL D_2O/Acetone-d_6 (*v/v* 1/1) solvent. The mixture was stirred at 80 °C for 16 h, and the encapsulated complex **3b·4$_3$** was formed. Excess **4** was filtered. The OTf–counterions were exchanged for PF_6^- using an aqueous solution of KPF_6 to precipitate the product, which was collected and washed with excess water and then dried in vacuum. Yield: 95 %. ^{1}H NMR (D_2O/Acetone-d_6: 1/1, 300 MHz): δ 8.76 (s, 48H, $H_{\alpha\text{-Py}}$), 7.36 (s, 48H, $H_{\beta\text{-Py}}$), 6.94 (m, 105H, Ph$H_{\mathbf{3b}\text{ and }\mathbf{4}}$), 6.35 (m, 45H, Ph$H_{\mathbf{4}}$), 1.63 (d, J = 4.2 Hz, 216H, PC*H*3). ^{31}P{^{1}H} NMR (D_2O/Acetone-d_6: 1/1, 121.4 MHz): δ -28.2 (s, $^1J_{Pt\text{-}P}$ = 3111 Hz). Anal. Calcd for $C_{432}H_{462}F_{144}N_{24}P_{48}Pt_{12}$: C, 41.33; H, 3.71; N, 2.68. Found: C, 40.95; H, 3.63; N, 2.83.

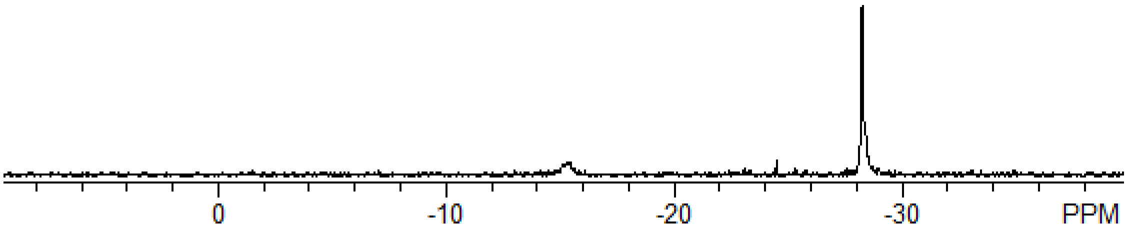

Figure S5. ^{31}P{^{1}H} NMR (300MHz) spectra of the encapsulated complex **3b•4**$_3$.

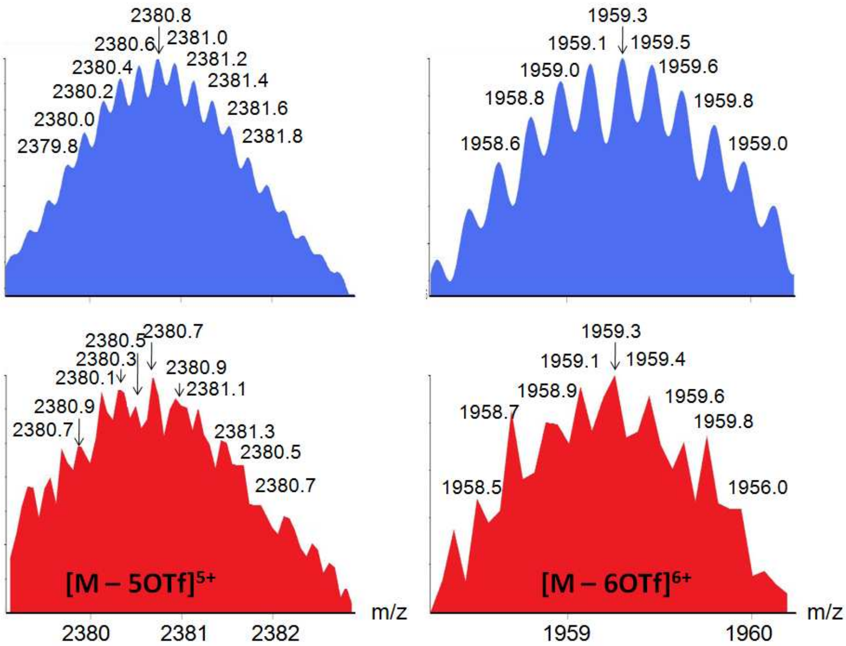

Figure S6. Calculated (top) and Experimental (bottom) ESI mass spectra of the encapsulated complex **3b•4**$_3$.

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