Stabilization of Hexa Coordinated P(V) Corroles by Axial Silyloxy Groups

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Chemicals: All the solvents such as Chloroform, Toluene, Triethylamine, Dichloromethane, n-hexane, Benzene were dried by following available methods and distilled under inert atmosphere prior to use. Deuterated solvents such as CDCl₃ and C₆D₆ were obtained from Aldrich and used as received. All the chemicals used for the synthesis were reagent grade. Column chromatography was performed using 60-120 mesh silica gel and basic aluminium oxide obtained from Sisco Research Laboratories, India.

Instrumentation: ¹H, ¹³C and ³¹P NMR spectra were recorded in C₆D₆ using a Brukar 400 MHz and 500 MHz spectrometer and reported in δ ppm. Absorption spectra were obtained using Cary series UV-VIS NIR spectrophotometer. The fluorescence quantum yields (ϕ_i) were calculated from the emission and absorption spectra by a comparative method at a excitation wavelength of 430 nm using Tetratolyl porphyrin (H₂TTP) ($\phi_f = 0.11$) as the standard. The time resolved fluorescence decay measurement were carried out at magic angle (54.60) using a picoseconds diode laser based TCSPC fluorescence spectrometer from IBH, UK. All the decays were fitted to single exponential decay. Cyclic voltammetry (CV) studies are carried out with a BAS electrochemical system by utilizing the three-electrode configuration consisting of glassy carbon (working electrode), platinum wire (auxiliary electrode) and saturated calomel (reference electrode) electrodes. The experiments were carried out in dry dichloromethane using tetrabutylammonium perchlorate as the supporting electrolyte. The high resolution mass spectra (HRMS) were recorded on a Q-Tof micro mass spectrometer.



Compound 2 Mol. Wt. = 732.25



Figure S1: HR-MS mass spectrum of compound 2.



Compound 3 Mol. Wt. = 1104.34



Figure S2: HR-MS mass spectrum of compound 3.



Compound 4 Mol. Wt. = 1164.40



Figure S3: HR-MS mass spectrum of compound 4.



Compound 5 Mol. Wt. = 816.34



Figure S4: HR-MS mass spectrum of compound 5.



Figure S5. ³¹P NMR spectrum of compound 2 recorded in C_6D_6 at room temperature.



Figure S6. ³¹P NMR spectrum of compound **3** recorded in C_6D_6 at room temperature.

-20	-40 -4	60 -80	-100	-120	-140	-160	-180	-200	-220	ppm
PC	1.40									
GB	0									
LB	1.00	Hz								
aco a	EM									
S.F	202.4563350	MHZ								
51 0P	32768	Mile								
F2 - Pro	cessing paramet	ers								
LUNID	0.21505555									
PLWIN	0.34327999	W								
PLW2	13.00000000									
PCPD2	80.00	usec								
CPDPRG [2	waltz16	A STATE OF								
NUC2	18									
SF02	500.1320005	MHz								
	CHANNEL f2									
PLW1	64.00000000	W						T		
PI	11 90	1100-0								
SPUL	202.4462121	PILL								
0000	CHANNEL f1	Mile						1		
	The standards									
TDO	1									
D11	0.03000000	sec						5		
D1	2.0000000	sec						2		
TE	296.2	K						6		
DR	6 50	11000						-		
DOM:	6 133	11000								
102	197 27	Bec								
FILKES	1.243523	HZ								
SWH	81521.742	HZ								
DS	4									
NS	17									
SOLVENT	C6D6									
TD	65536									
PULPROG	zgpg30									
PROBHD	5 mm PABBO BB/									
INSTRUM	spect									
Time	2.25									
Date	20150914	the bank of								
F2 - Acr	uisition Parame	ters								
PROCNO	1									
EXPNO	6									
NAME	MR-TC-PH2CH3PC	OR-31P	MR-TC-F	H2CH3PCO	R-31P					
Current	Data Parameters									
-										

Figure S7. ³¹P NMR spectrum of compound 4 recorded in C_6D_6 at room temperature.



Figure S8. ³¹P NMR spectrum of compound 5 recorded in C_6D_6 at room temperature.



Figure S9. ¹³C NMR spectrum of compound 2 recorded in C_6D_6 at room temperature.



Figure S10. ¹³C NMR spectrum of compound 3 recorded in C_6D_6 at room temperature.



Figure S11. ¹³C NMR spectrum of compound 4 recorded in C_6D_6 at room temperature.



Figure S12. ¹³C NMR spectrum of compound 5 recorded in C_6D_6 at room temperature.



Figure S13. Unit cell packing diagram for 4 as determined from the single crystal structural analysis.