Synthesis and Crystal Structure of Unprecedented Phosphine Adducts of d^1-Aryl Imido-Vanadium(IV) Complexes.**

Christian Lorber,* Robert Choukroun and Bruno Donnadieu

Laboratoire de Chimie de Coordination, CNRS UPR 8241,

205 route de Narbonne, 31077 Toulouse Cedex 04, France

Supplementary Material

1. Full experimental Section

2. Full X-ray Data

1. Experimental Section

General Methods and Instrumentation

All manipulations were carried out using standard Schlenk line or dry box techniques under an atmosphere of argon. Solvents were refluxed and dried over appropriate drying agents under an atmosphere of argon, and collected by distillation. EPR spectra were recorded on a Bruker ESP300E spectrometer. Elemental analyses were performed at the Laboratoire de Chimie de Coordination (Toulouse, France) (C,H,N) or by the Service Central de Microanalyses du CNRS at Vernaison (France) (C,H,N,Cl). Magnetic susceptibility data were collected on powdered samples of the different compounds with use of a home-made Faraday-type automatic magnetometer [for **2-3**] using mercury tetra(thiocyanato)cobaltate (susceptibility at 20°, 16.44 x 10^{-6} cm^3mol^{-1}) or with a Quantum Design MPMS SQUID susceptometer [for **4**] (magnetic susceptibility measurements were performed in the 2-300 K temperature range in a 1 T applied magnetic field). Diamagnetic corrections were applied by using Pascal's constants.[Pascal, P. *Ann. Chim. Phys.* **1910**, *19*, 5]

Compound [V(NAr)Cl_2] (**1**) (Ar = 2,6-*i*-Pr_2-C_6H_3) was prepared according to our known procedure.[Lorber, C.; Choukroun, R.; Donnadieu, B., *Inorg. Chem.*, **2002**, *41*, 4216-4226]

Synthesis of [V(N=Ar)Cl_2(PMe_2Ph)$_2$] (2). To a dichloromethane solution (2 mL) of 150 mg of **1** (0.5048 mM) was added 209 mg of PMe_2Ph (1.515 mM) at RT. After 3 days, the resulting dark yellow solution was evaporated to dryness under vacuum giving red-orange crystals that were washed with pentane (2 x 2 mL) (yield: 250 mg, 86%). EPR (CH_2Cl_2, 20°C) g = 1.993, A(^{51}V) = 83 G, A(^{31}P) = 30 G, A(^{14}N) = 5 G. μ_{eff} = 1.77 μ_B (300K). Anal. Calcd for $C_{28}H_{39}Cl_2NP_2V$: C, 58.65; H, 6.86; N, 2.44. Found: C, 58.37; H, 6.85; N, 2.53.

Synthesis of [V(N=Ar)Cl_2(dmpe)$_{1.5}$]$_2$ (3). dmpe (228 mg, 1.515 mM) was added to a dichloromethane solution (2 mL) of **1** (300 mg, 1.01 mM) at RT with stirring. The resulting dark yellow solution was layered with 4 mL of pentane to give a first crop of crystals (150 mg) that appeared to be contaminated by small amount of unreacted **1**. The solution was placed at –20°C to give a second crop of dark crystals of pure **3**.CH_2Cl_2 (yield: 200 mg, 35%). μ_{eff} = 1.83 μ_B for the monomer unit (300K). Anal. Calcd for $C_{42}H_{82}Cl_4N_2P_6V_2$.$CH_2Cl_2$: C, 45.72; H, 7.50; N, 2.48. Found: C, 45.85; H, 7.60; N, 2.40.

Synthesis of $[V(N=Ar)Cl_2(dppm)_2]_2$ (4). 150 mg of 1 (0.5048 mM) were dissolved in 2 mL of dichloromethane. 194 mg of dppm (0.5048 mM) were added by portions to this solution at RT. After 2 hours crystals began to appear and the solution was then carefully layered with some pentane. The crystals of **4**.CH_2Cl_2 were collected and dried under vacuum (yield: 225 mg, 62%). μ_{eff} = 1.87 μ_B for the monomer unit (300K). Anal. Calcd for $C_{74}H_{78}Cl_4N_2P_4V_2.CH_2Cl_2$: C, 62.21; H, 5.57; N, 1.93. Found: C, 61.37; H, 5.72; N, 1.87.

2. X-ray data

Crystal Structure Determination of 1-3.

Crystals of [**2**] (red plates), [**3**] (dark blocks), [**4**] (red-brown blocks), were obtained. For the three structures data collection were collected at low temperature (T=180K) on a Stoe Imaging Plate Diffraction System (IPDS) [4] equipped with an Oxford Cryosystems Cryostream Cooler Device or an Oxford Diffraction Kappa CCD Excalibur [2-3] diffractometer equipped with an a cryojet from Oxford Instrument, and using a graphite-monochromated Mo-Kα radiation (λ = 0.71073Å). Final unit cell parameters were obtained by means of a least-squares refinement of a set of 8000 well measured reflections, and a crystal decay was monitored during data collection by measuring 200 reflections by image, no significant fluctuation of intensities has been observed. Structures have been solved by means of Direct Methods using the program SIR92 [Altomare, A.; Cascarano, G.; Giacovazzo, G.; Guagliardi, A.; Burla, M.C.; Polidori, G.; Camalli, M. *J. Appl. Cryst.* **1994**, *27*, 435.], and subsequent difference Fourier maps, models were refined by least-squares procedures on a F^2 by using SHELXL-97 [SHELX97 [Includes SHELXS97, SHELXL97, CIFTAB] - Programs for Crystal Structure Analysis (Release 97-2). G. M. Sheldrick, Institüt für Anorganische Chemie der Universität, Tammanstrasse 4, D-3400 Göttingen, Germany, 1998] integrated in the package WINGX version 1.64 [Farrugia, L.J. *J. Appl. Cryst*. **1999**, *32*, 837-838], and empirical absorption corrections were applied on data [Walker, N.; Stuart, D. *Acta Cryst. A* **1983**, *39*, 158-166]. All hydrogen atoms have been located on differences fourier maps and introduced in the refinement in idealized positions using a rigid groups with an isotropic thermal parameter fixed at 20% higher than those of carbon atoms with which they are connected. For the seven structures all non-hydrogen atoms were anisotropically refined, in the last cycles of refinement a weighting schemes were used, where weights are calculated from the following formula: $w=1/[\sigma^2(Fo^2)+(aP)^2+bP]$ where $P=(Fo^2+2Fc^2)/3$.

Data for 1

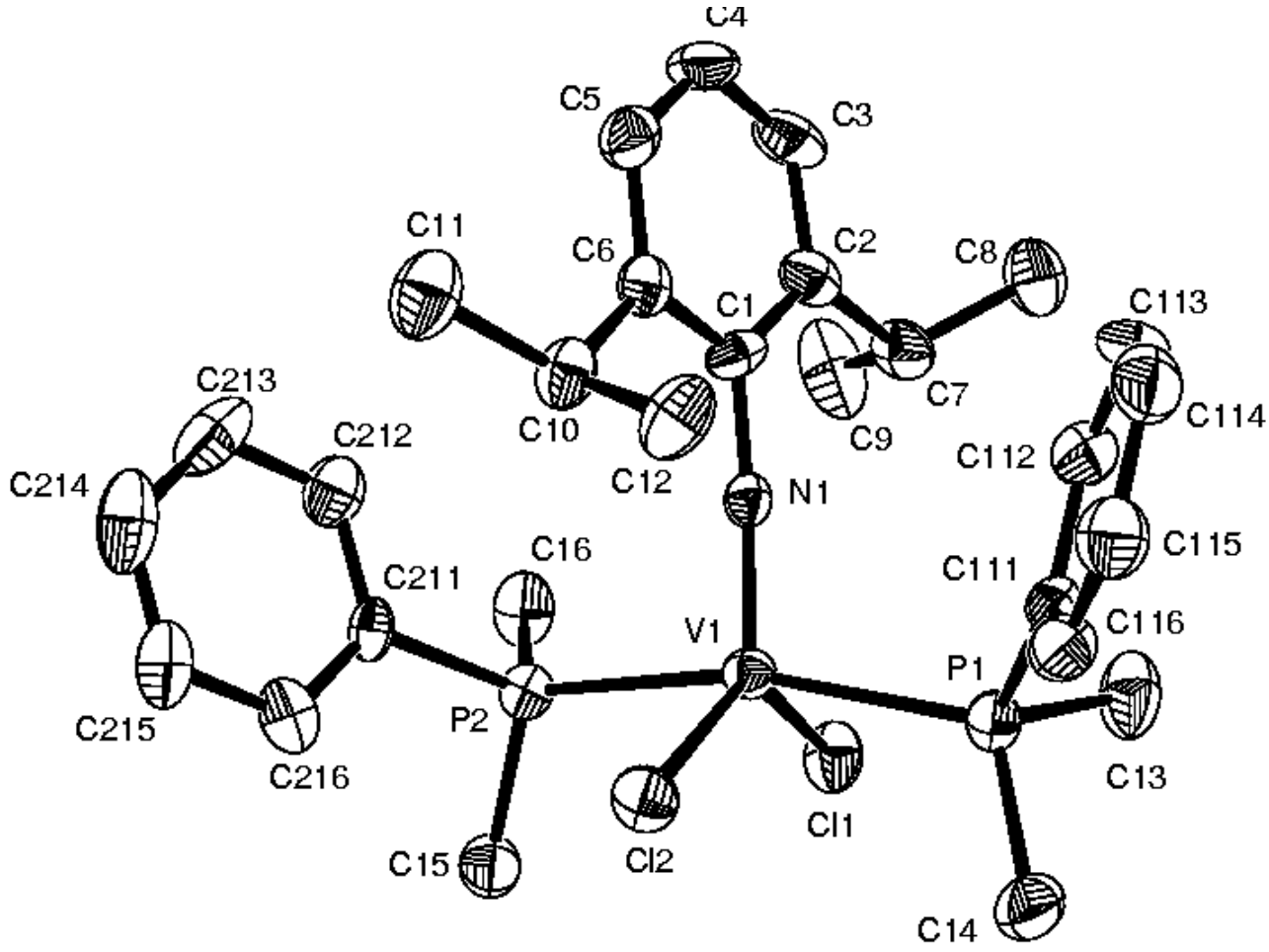
C4
C5
C3
C11
C6
C2
C8
C1
C113
C213
C7
C114
C212
C10
C9
C112
C214
C12
N1
C16
C115
C211
C111
V1
C116
P1
C215
P2
C13
C216
Cl1
Cl2
C15
C14

Table S1 .Crystal data, data collection and structure refinement

Empirical formula	$C_{28}H_{39}Cl_2NP_2V$
Formula weight	573.38
Temperature	180(2)K
Wavelength	0.71073Å
Crystal system, space group	orthorhombic, P b c a
Unit cell dimensions	a = 12.090(2)Å
	b = 16.489(3)Å
	c = 29.815(6)Å
Volume	5943.7(19)$Å^3$
Z, Calculated density	8, 1.282 mg/m^3
Absorption coefficient	0.638mm^{-1}
F(000)	2408
Tube power	2.00kW
Tube voltage	50kV
Tube current	40mA
Collimator size	0.5mm
Detector distance	55mm
Measurement device method	omega scan
Crystal size	(0.22x0.14x0.07)mm
Crystal color	red
Crystal form	plate
Theta range for data collection	(3.21 to 20.81)°
Index ranges	-10<=h<=12, -15<=k<=16, -28<=l<=29
Reflections collected / unique	23753 / 3092 [R(int) = 0.1571]
Completeness to 2theta = 41.62	99.5%
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3092 / 0 / 316
Absorption corrections	Semi-empirical*
Tmin – Tmax	0.464 - 0.825
Goodness-of-fit on F^2	1.256
Scheme of ponderation	Weight = $1/[\sigma^2(F_o^2)+24.433]$
where P = $(F_o^2+2F_c^2)/3$	
Final R indices [I>2σ(I)]	R1 = 0.0817, wR2 = 0.1323
R indices (all data)	R1 = 0.0938, wR2 = 0.1378
Extinction coefficient	0.0003(2)
Largest diff. peak and hole	(0.333 and -0.307)e.$Å^{-3}$

*DIFABS - N. Walker and D. Stuart, Acta Crystallogr., Sect A 1983, 39, 158-166.

Table S2. Atomic coordinates (x10^4) and equivalent isotropic displacement parameters (Å^{2}x10^3), U(eq) is defined as one third of the trace of the orthogonalized Uij tensor

	x	y	z	U(eq)
V	9192(1)	2400(1)	1335(1)	26(1)
Cl(1)	8654(2)	1571(1)	1924(1)	41(1)
Cl(2)	9530(2)	2063(1)	598(1)	36(1)
P(1)	7174(2)	2536(1)	1142(1)	33(1)
P(2)	11085(2)	1894(1)	1507(1)	29(1)
N	9439(4)	3368(3)	1455(2)	25(1)
C(1)	9732(6)	4160(4)	1562(2)	28(2)
C(2)	9406(6)	4493(5)	1978(2)	34(2)
C(3)	9777(7)	5255(5)	2084(3)	46(2)
C(4)	10402(7)	5707(5)	1796(3)	47(2)
C(5)	10680(6)	5389(5)	1375(3)	37(2)
C(6)	10356(6)	4619(4)	1247(2)	30(2)
C(7)	8696(7)	4005(5)	2298(2)	38(2)
C(8)	7748(7)	4506(5)	2495(3)	50(2)
C(9)	9408(8)	3623(6)	2664(3)	74(3)
C(10)	10614(6)	4277(5)	784(2)	34(2)
C(11)	11580(6)	4689(5)	554(3)	50(2)
C(12)	9584(6)	4310(5)	486(2)	45(2)
C(13)	6259(6)	2530(6)	1625(3)	54(2)
C(14)	6600(7)	1752(5)	786(3)	50(2)
C(15)	11172(6)	801(4)	1418(3)	40(2)
C(16)	11540(6)	2020(5)	2082(2)	46(2)
C(111)	6812(6)	3465(4)	860(2)	27(2)
C(112)	6803(6)	4188(5)	1106(3)	38(2)
C(113)	6540(7)	4926(5)	910(3)	46(2)
C(114)	6313(7)	4949(5)	457(3)	48(2)
C(115)	6327(6)	4256(5)	206(3)	42(2)
C(116)	6582(6)	3511(5)	403(3)	37(2)
C(211)	12204(5)	2320(4)	1179(2)	28(2)
C(212)	12725(6)	3030(5)	1313(3)	41(2)
C(213)	13544(7)	3384(6)	1048(3)	55(3)
C(214)	13809(7)	3047(6)	637(3)	53(3)
C(215)	13299(6)	2349(6)	501(3)	45(2)
C(216)	12496(6)	1996(5)	767(2)	38(2)

Table S3. Full list of bond lengths [Å] and angles [°], e.s.d's in parentheses refers to the last significant digit

V	- N	1.663(6)		V	- Cl(2)	2.303(2)	
V	- Cl(1)	2.318(2)		V	- P(2)	2.489(2)	
V	- P(1)	2.517(2)		P(1)	- C(111)	1.802(7)	
P(1)	- C(14)	1.811(8)		P(1)	- C(13)	1.814(7)	
P(2)	- C(16)	1.812(7)		P(2)	- C(211)	1.812(7)	
P(2)	- C(15)	1.823(7)		N	- C(1)	1.391(9)	
C(1)	- C(2)	1.411(10)		C(1)	- C(6)	1.422(10)	
C(2)	- C(3)	1.370(11)		C(2)	- C(7)	1.515(10)	
C(3)	- C(4)	1.365(11)		C(4)	- C(5)	1.400(11)	
C(5)	- C(6)	1.382(10)		C(6)	- C(10)	1.524(10)	
C(7)	- C(9)	1.526(11)		C(7)	- C(8)	1.531(10)	
C(10)	- C(11)	1.517(10)		C(10)	- C(12)	1.531(10)	
C(111)	- C(116)	1.394(10)		C(111)	- C(112)	1.399(10)	
C(112)	- C(113)	1.388(11)		C(113)	- C(114)	1.378(11)	
C(114)	- C(115)	1.366(11)		C(115)	- C(116)	1.398(11)	
C(211)	- C(216)	1.384(9)		C(211)	- C(212)	1.389(10)	
C(212)	- C(213)	1.395(11)		C(213)	- C(214)	1.383(12)	
C(214)	- C(215)	1.368(12)		C(215)	- C(216)	1.383(10)	

N	- V	- Cl(2)	113.92(19)	N	- V	- Cl(1)	116.97(19)
Cl(2)	- V	- Cl(1)	129.03(9)	N	- V	- P(2)	96.5(2)
Cl(2)	- V	- P(2)	87.29(7)	Cl(1)	- V	- P(2)	84.52(7)
N	- V	- P(1)	97.9(2)	Cl(2)	- V	- P(1)	88.59(8)
Cl(1)	- V	- P(1)	87.31(8)	P(2)	- V	- P(1)	165.50(8)
C(111)	- P(1)	- C(14)	103.9(3)	C(111)	- P(1)	- C(13)	103.1(4)
C(14)	- P(1)	- C(13)	103.2(4)	C(111)	- P(1)	- V	114.7(2)
C(14)	- P(1)	- V	116.2(3)	C(13)	- P(1)	- V	114.2(3)
C(16)	- P(2)	- C(211)	103.9(3)	C(16)	- P(2)	- C(15)	103.5(4)
C(211)	- P(2)	- C(15)	105.2(3)	C(16)	- P(2)	- V	115.8(3)
C(211)	- P(2)	- V	116.4(2)	C(15)	- P(2)	- V	110.8(3)
C(1)	- N	- V	175.5(5)	N	- C(1)	- C(2)	119.7(6)
N	- C(1)	- C(6)	118.9(6)	C(2)	- C(1)	- C(6)	121.3(7)
C(3)	- C(2)	- C(1)	117.9(7)	C(3)	- C(2)	- C(7)	121.8(7)
C(1)	- C(2)	- C(7)	120.3(7)	C(4)	- C(3)	- C(2)	122.4(8)
C(3)	- C(4)	- C(5)	119.4(7)	C(6)	- C(5)	- C(4)	121.6(7)
C(5)	- C(6)	- C(1)	117.2(7)	C(5)	- C(6)	- C(10)	122.2(7)
C(1)	- C(6)	- C(10)	120.6(6)	C(2)	- C(7)	- C(9)	110.5(7)
C(2)	- C(7)	- C(8)	112.3(6)	C(9)	- C(7)	- C(8)	111.7(6)

C(11) - C(10) - C(6)	113.7(7)	C(11) - C(10) - C(12)	110.3(6)
C(6) - C(10) - C(12)	110.3(6)	C(116) - C(111) - C(112)	117.7(7)
C(116) - C(111) - P(1)	123.4(6)	C(112) - C(111) - P(1)	118.8(5)
C(113) - C(112) - C(111)	121.8(7)	C(114) - C(113) - C(112)	118.9(7)
C(115) - C(114) - C(113)	120.8(8)	C(114) - C(115) - C(116)	120.5(7)
C(111) - C(116) - C(115)	120.2(7)	C(216) - C(211) - C(212)	117.8(7)
C(216) - C(211) - P(2)	121.3(6)	C(212) - C(211) - P(2)	120.7(6)
C(211) - C(212) - C(213)	120.7(8)	C(214) - C(213) - C(212)	119.9(9)
C(215) - C(214) - C(213)	119.8(8)	C(214) - C(215) - C(216)	120.0(8)
C(215) - C(216) - C(211)	121.7(8)		

Table S4. Anisotropic displacement parameters ($Å^2x10^3$), the anisotropic displacement factor exponent takes the form: $exp[-2p^2(U_{11}h^2a^{*2} + U_{22}k_2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}klb^*c^*)]$

	U11	U22	U33	U23	U13	U12
V	28(1)	25(1)	24(1)	-1(1)	1(1)	-1(1)
Cl(1)	38(1)	46(1)	40(1)	14(1)	8(1)	0(1)
Cl(2)	40(1)	43(1)	24(1)	-5(1)	1(1)	-1(1)
P(1)	27(1)	32(1)	41(1)	0(1)	1(1)	0(1)
P(2)	29(1)	31(1)	27(1)	3(1)	-1(1)	0(1)
N	22(3)	27(4)	25(3)	2(3)	3(3)	0(3)
C(1)	27(4)	21(5)	38(5)	7(4)	-16(4)	-4(4)
C(2)	37(5)	32(5)	33(5)	-5(4)	-8(4)	7(4)
C(3)	59(6)	36(6)	43(5)	-18(4)	0(5)	8(5)
C(4)	45(5)	26(5)	68(6)	-13(5)	-11(5)	-5(4)
C(5)	28(4)	34(5)	50(5)	6(4)	-7(4)	0(4)
C(6)	22(4)	34(5)	33(4)	1(4)	-9(4)	4(4)
C(7)	47(5)	40(5)	28(4)	-6(4)	10(4)	1(4)
C(8)	46(6)	63(6)	42(5)	-3(5)	11(4)	8(5)
C(9)	66(7)	94(8)	61(6)	22(6)	17(5)	33(6)
C(10)	31(4)	39(5)	32(4)	11(4)	2(4)	-2(4)
C(11)	40(5)	61(6)	48(5)	16(5)	1(4)	-4(5)
C(12)	41(5)	58(6)	35(5)	11(4)	-1(4)	-13(5)
C(13)	36(5)	60(6)	65(6)	22(5)	12(4)	1(5)
C(14)	37(5)	32(5)	81(6)	1(5)	-11(5)	-2(4)
C(15)	39(5)	35(5)	47(5)	5(4)	3(4)	2(4)
C(16)	40(5)	67(6)	30(5)	1(4)	-2(4)	1(5)
C(111)	22(4)	27(5)	32(5)	-4(4)	4(3)	-7(3)
C(112)	36(5)	38(5)	40(5)	-3(4)	-2(4)	-3(4)

C(113)	44(5)	21(5)	71(7)	-12(4)	6(5)	0(4)
C(114)	37(5)	35(5)	71(7)	15(5)	6(5)	4(4)
C(115)	38(5)	47(6)	40(5)	4(5)	0(4)	1(4)
C(116)	34(5)	39(5)	39(5)	-5(4)	2(4)	-5(4)
C(211)	17(4)	39(5)	27(4)	5(4)	-2(3)	2(4)
C(212)	31(5)	50(6)	44(5)	3(4)	-7(4)	0(4)
C(213)	38(5)	54(6)	74(7)	23(5)	-16(5)	-17(5)
C(214)	30(5)	86(8)	44(6)	28(5)	1(4)	10(5)
C(215)	31(5)	64(7)	40(5)	10(5)	7(4)	4(5)
C(216)	36(5)	47(5)	29(5)	5(4)	1(4)	11(4)

Table S5. Hydrogen coordinates (x10^4) and isotropic displacement parameters (Å^{2}x10^3)

	x	y	z	U(eq)
H(3)	9591	5476	2368	55
H(4)	10647	6233	1881	56
H(5)	11100	5710	1173	45
H(7)	8357	3552	2122	46
H(8A)	7333	4765	2252	75
H(8B)	8051	4924	2694	75
H(8C)	7255	4151	2667	75
H(9A)	9986	3289	2526	110
H(9B)	8944	3282	2856	110
H(9C)	9751	4051	2844	110
H(10)	10814	3693	824	41
H(11A)	11781	4385	284	75
H(11B)	12214	4707	758	75
H(11C)	11370	5243	470	75
H(12A)	9752	4068	194	67
H(12B)	9359	4877	444	67
H(12C)	8982	4008	629	67
H(13A)	6313	2005	1778	81
H(13B)	5496	2618	1525	81
H(13C)	6475	2963	1832	81
H(14A)	7023	1725	506	75
H(14B)	5826	1879	719	75
H(14C)	6642	1228	940	75
H(15A)	10638	526	1614	61
H(15B)	11921	614	1490	61

H(15C)	11003	677	1104	61
H(16A)	10983	1794	2286	68
H(16B)	11640	2598	2146	68
H(16C)	12243	1735	2125	68
H(112)	6982	4173	1416	46
H(113)	6516	5408	1084	55
H(114)	6146	5453	318	57
H(115)	6161	4282	-105	50
H(116)	6599	3033	225	45
H(212)	12521	3277	1589	50
H(213)	13920	3855	1150	66
H(214)	14343	3300	450	64
H(215)	13496	2106	223	54
H(216)	12135	1518	665	45

Data for 2

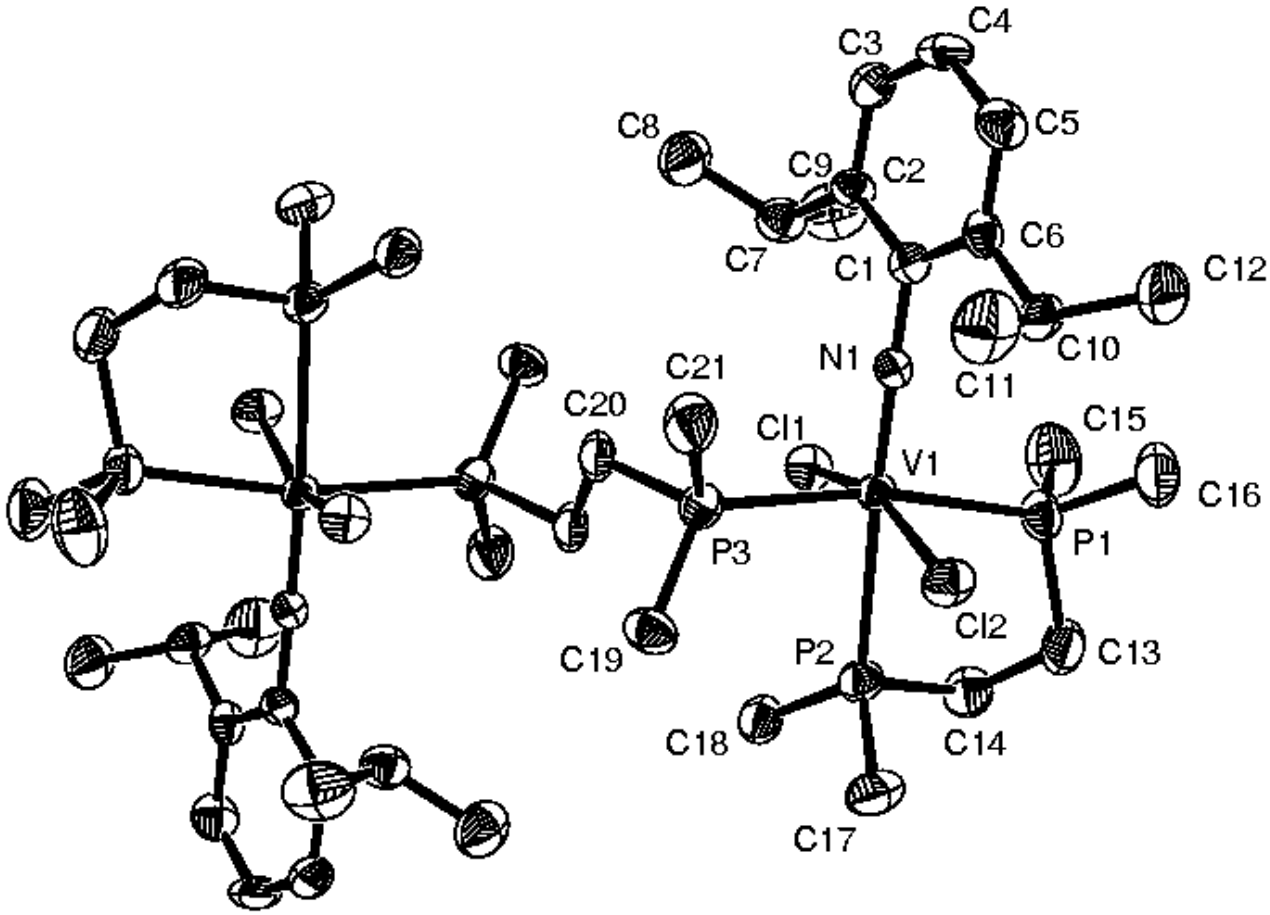
C4
C3
C5
C8
C9
C2
C6
C7
C1
C12
C10
C11
N1
C21
C20
Cl1
C15
V1
C16
P3
P1
Cl2
C19
P2
C13
C18
C14
C17

Table S1 .Crystal data, data collection and structure refinement

Empirical formula	$C_{22}H_{43}Cl_4NP_3V$
Formula weight	607.22
Temperature	180(2)K
Wavelength	0.71073Å
Crystal system, space group	monoclinic, P $2_1/c$
Unit cell dimensions	a = 10.769(5)Å
	b = 15.902(5)Å
	c = 18.413(5)Å
	β = 90.660(5)°
Volume	3153(2)A3
Z, Calculated density	4, 1.279mg/m^3
Absorption coefficient	0.817mm^{-1}
F(000)	1272
Crystal size	(0.35x0.25x0.12)mm
Crystal color	dark red
Crystal form	platelet
Tube power	2.00kW
Tube voltage	50kV
Tube current	40mA
Collimator size	0.5mm
Detector distance	55mm
Measurement device method	omega scan
Theta range for data collection	(3.36 to 20.81)°
Index ranges	-10<=h<=10, -15<=k<=15, -18<=l<=17
Reflections collected / unique	11812 / 3289 [R(int) = 0.0982]
Completeness to 2theta = 41.62	99.3%
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3289 / 0 / 310
Absorption corrections	Semi-empirical*
Tmin – Tmax	0.504 - 0.843
Goodness-of-fit on F^2	1.070
Scheme of ponderation	Weight = $1/[\sigma^2(F_o^2)+(0.0231P)^2+16.539]$
where P = $(F_o^2+2F_c^2)/3$	
Final R indices [I>2σ(I)]	R1 = 0.0686, wR2 = 0.1307
R indices (all data)	R1 = 0.0836, wR2 = 0.1394
Extinction coefficient	0.0005(5)
Largest diff. peak and hole	(0.513 and -0.393)e.$Å^{-3}$

*DIFABS - N. Walker and D. Stuart, Acta Crystallogr., Sect A 1983, 39, 158-166.

Table S2. Atomic coordinates (x10^4) and equivalent isotropic displacement parameters (Å^{2}x10^3), U(eq) is defined as one third of the trace of the orthogonalized Uij tensor

	x	y	z	U(eq)
V	7355(1)	1195(1)	3766(1)	26(1)
N	7328(5)	2148(3)	4172(3)	24(1)
P(1)	7806(2)	1690(1)	2517(1)	40(1)
P(2)	7366(2)	-263(1)	3027(1)	37(1)
P(3)	6994(2)	413(1)	4921(1)	34(1)
Cl(1)	5222(2)	1082(1)	3386(1)	38(1)
Cl(2)	9506(2)	855(1)	3885(1)	41(1)
C(1)	7300(6)	2950(4)	4495(3)	27(2)
C(2)	6160(6)	3281(4)	4727(3)	29(2)
C(3)	6161(7)	4081(4)	5028(4)	40(2)
C(4)	7214(7)	4546(5)	5091(4)	45(2)
C(5)	8337(7)	4213(5)	4883(4)	42(2)
C(6)	8419(6)	3403(4)	4593(3)	31(2)
C(7)	4952(6)	2802(5)	4615(4)	41(2)
C(8)	4103(7)	2862(5)	5274(4)	57(2)
C(9)	4275(8)	3120(6)	3927(5)	67(3)
C(10)	9663(6)	3020(5)	4412(4)	40(2)
C(11)	10290(8)	2713(6)	5107(5)	68(3)
C(12)	10491(8)	3630(5)	4006(4)	58(2)
C(13)	8284(8)	806(5)	1956(4)	54(2)
C(14)	7389(8)	63(5)	2065(4)	50(2)
C(15)	6549(9)	2169(6)	2004(5)	74(3)
C(16)	9053(8)	2442(5)	2417(4)	60(2)
C(17)	8699(7)	-969(5)	3091(4)	52(2)
C(18)	6086(7)	-1012(5)	3032(4)	43(2)
C(19)	7574(7)	-648(4)	5043(4)	43(2)
C(20)	5382(7)	330(4)	5218(3)	39(2)
C(21)	7709(8)	971(5)	5677(4)	56(2)

***CH_2Cl_2**

	x	y	z	U(eq)
C(1S)	2289(9)	754(7)	2570(5)	84(3)
Cl(1S)	2610(20)	-290(17)	2193(12)	164(9)

Cl(2S)	2090(20)	1454(15)	1831(11)	166(10)
Cl(3S)	2401(19)	1654(11)	2073(11)	123(6)
Cl(4S)	2150(17)	-154(13)	2087(9)	131(6)

Table S3. Full list of bond lengths [Å] and angles [°], e.s.d's in parentheses refers to the last significant digit

V	- N	1.691(5)	V	- Cl(2)	2.387(2)		
V	- Cl(1)	2.400(2)	V	- P(1)	2.484(2)		
V	- P(3)	2.497(2)	V	- P(2)	2.689(2)		
N	- C(1)	1.407(8)	P(1)	- C(16)	1.809(8)		
P(1)	- C(15)	1.810(8)	P(1)	- C(13)	1.823(8)		
P(2)	- C(18)	1.821(7)	P(2)	- C(17)	1.824(8)		
P(2)	- C(14)	1.847(7)	P(3)	- C(19)	1.813(7)		
P(3)	- C(21)	1.815(7)	P(3)	- C(20)	1.830(7)		
C(1)	- C(2)	1.406(9)	C(1)	- C(6)	1.414(9)		
C(2)	- C(3)	1.388(9)	C(2)	- C(7)	1.519(9)		
C(3)	- C(4)	1.358(10)	C(4)	- C(5)	1.378(10)		
C(5)	- C(6)	1.397(10)	C(6)	- C(10)	1.512(10)		
C(7)	- C(8)	1.531(10)	C(7)	- C(9)	1.540(10)		
C(10)	- C(11)	1.520(11)	C(10)	- C(12)	1.521(10)		
C(13)	- C(14)	1.539(10)	C(20)	- C(20)#1	1.552(13)		

***CH_2Cl_2**

C(1S)	- Cl(1S)	1.83(3)	C(1S)	- Cl(2S)	1.77(2)		
C(1S)	- Cl(3S)	1.705(19)	C(1S)	- Cl(4S)	1.70(2)		

N	- V	- Cl(2)	100.60(18)	N	- V	- Cl(1)	100.09(18)
Cl(2)	- V	- Cl(1)	159.13(8)	N	- V	- P(1)	97.44(17)
Cl(2)	- V	- P(1)	87.52(8)	Cl(1)	- V	- P(1)	87.17(7)
N	- V	- P(3)	93.81(17)	Cl(2)	- V	- P(3)	88.23(7)
Cl(1)	- V	- P(3)	93.06(7)	P(1)	- V	- P(3)	168.54(8)
N	- V	- P(2)	175.78(17)	Cl(2)	- V	- P(2)	80.84(7)
Cl(1)	- V	- P(2)	78.33(7)	P(1)	- V	- P(2)	78.62(7)
P(3)	- V	- P(2)	90.19(7)	C(1)	- N	- V	178.8(4)
C(16)	- P(1)	- C(15)	102.7(4)	C(16)	- P(1)	- C(13)	103.7(4)
C(15)	- P(1)	- C(13)	104.1(4)	C(16)	- P(1)	- V	117.2(3)
C(15)	- P(1)	- V	117.6(3)	C(13)	- P(1)	- V	109.9(3)
C(18)	- P(2)	- C(17)	101.1(4)	C(18)	- P(2)	- C(14)	102.0(3)
C(17)	- P(2)	- C(14)	102.5(4)	C(18)	- P(2)	- V	123.5(2)
C(17)	- P(2)	- V	120.4(3)	C(14)	- P(2)	- V	104.1(2)

C(19)	- P(3)	- C(21)	102.6(4)	C(19)	- P(3)	- C(20)	102.8(4)
C(21)	- P(3)	- C(20)	101.6(4)	C(19)	- P(3)	- V	120.8(2)
C(21)	- P(3)	- V	110.0(3)	C(20)	- P(3)	- V	116.6(2)
C(2)	- C(1)	- N	119.4(6)	C(2)	- C(1)	- C(6)	121.1(6)
N	- C(1)	- C(6)	119.5(6)	C(3)	- C(2)	- C(1)	117.9(6)
C(3)	- C(2)	- C(7)	120.7(6)	C(1)	- C(2)	- C(7)	121.4(6)
C(4)	- C(3)	- C(2)	122.0(7)	C(3)	- C(4)	- C(5)	120.1(7)
C(4)	- C(5)	- C(6)	121.3(7)	C(5)	- C(6)	- C(1)	117.4(6)
C(5)	- C(6)	- C(10)	121.1(6)	C(1)	- C(6)	- C(10)	121.5(6)
C(2)	- C(7)	- C(8)	112.3(6)	C(2)	- C(7)	- C(9)	110.1(6)
C(8)	- C(7)	- C(9)	110.5(6)	C(6)	- C(10)	- C(11)	109.3(6)
C(6)	- C(10)	- C(12)	112.2(6)	C(11)	- C(10)	- C(12)	111.2(7)
C(14)	- C(13)	- P(1)	109.7(5)	C(13)	- C(14)	- P(2)	110.8(5)
C(20)#1	- C(20)	- P(3)	113.2(6)				

Symmetry transformations used to generate equivalent atoms: #1 -x+1,-y,-z+1

***CH_2Cl_2**

Cl(2S)	- C(1S)	- Cl(1S)	107.4(12)	Cl(4S)	- C(1S)	- Cl(3S)	116.0(11)

Table S4. Anisotropic displacement parameters ($Å^2x10^3$), the anisotropic displacement factor exponent takes the form: $exp[-2p^2(U_{11}h^2a^{*2} + U_{22}k_2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}klb^*c^*)]$

	U11	U22	U33	U23	U13	U12
V	26(1)	27(1)	25(1)	-2(1)	1(1)	-1(1)
N	21(3)	26(3)	27(3)	2(3)	-2(2)	-1(2)
P(1)	48(1)	41(1)	30(1)	3(1)	1(1)	-9(1)
P(2)	41(1)	36(1)	32(1)	-8(1)	3(1)	-2(1)
P(3)	35(1)	38(1)	28(1)	-1(1)	0(1)	0(1)
Cl(1)	33(1)	40(1)	42(1)	-3(1)	-5(1)	-2(1)
Cl(2)	27(1)	45(1)	51(1)	-6(1)	0(1)	4(1)
C(1)	31(4)	29(4)	22(4)	5(3)	-4(3)	6(3)
C(2)	23(4)	25(4)	40(4)	0(3)	-2(3)	4(3)
C(3)	32(5)	39(5)	48(5)	-1(4)	10(4)	3(4)
C(4)	51(6)	27(4)	57(5)	-11(4)	8(4)	8(4)
C(5)	42(5)	39(5)	46(4)	-4(4)	-3(4)	-10(4)
C(6)	35(5)	34(4)	24(4)	2(3)	-1(3)	-9(4)
C(7)	35(5)	37(4)	49(5)	-1(4)	-1(4)	5(4)
C(8)	47(5)	60(6)	64(5)	-12(4)	13(4)	-14(4)
C(9)	58(6)	74(7)	70(6)	-2(5)	-17(5)	22(5)

C(10)	22(4)	41(5)	56(5)	-10(4)	13(4)	-6(4)
C(11)	41(5)	86(7)	79(6)	9(5)	-2(5)	11(5)
C(12)	50(6)	59(6)	66(6)	-4(5)	15(4)	-14(4)
C(13)	65(6)	56(5)	43(5)	-8(4)	20(4)	-18(5)
C(14)	68(6)	50(5)	32(4)	-15(4)	5(4)	-13(4)
C(15)	75(7)	84(7)	63(6)	31(5)	-10(5)	-3(6)
C(16)	66(6)	63(6)	52(5)	4(4)	15(4)	-23(5)
C(17)	53(6)	50(5)	53(5)	-15(4)	8(4)	8(4)
C(18)	46(5)	45(5)	40(4)	-9(4)	8(4)	-11(4)
C(19)	56(5)	33(4)	39(4)	4(3)	-6(4)	8(4)
C(20)	42(5)	42(5)	32(4)	5(3)	10(3)	-9(4)
C(21)	74(6)	57(6)	35(4)	-2(4)	2(4)	-13(5)

***CH_2Cl_2**

	U11	U22	U33	U23	U13	U12
C(1S)	65(7)	104(9)	83(7)	12(6)	-21(6)	-19(6)
Cl(1S)	200(20)	161(11)	135(9)	26(7)	-86(11)	-43(12)
Cl(2S)	130(15)	260(20)	112(11)	89(12)	37(10)	61(12)
Cl(3S)	99(8)	114(7)	155(15)	71(8)	3(8)	17(5)
Cl(4S)	126(11)	153(13)	115(10)	-60(10)	-3(8)	-53(8)

Table S5. Hydrogen coordinates ($x10^4$) and isotropic displacement parameters ($Å^2x10^3$)

	x	y	z	U(eq)
H(3)	5401	4310	5195	48
H(4)	7177	5102	5279	54
H(5)	9070	4540	4938	51
H(7)	5162	2196	4540	49
H(8A)	3364	2513	5192	85
H(8B)	3851	3448	5345	85
H(8C)	4550	2662	5708	85
H(9A)	3575	2747	3814	101
H(9B)	4852	3123	3519	101
H(9C)	3968	3692	4010	101
H(10)	9511	2521	4092	47
H(11A)	11086	2449	4990	103
H(11B)	9754	2301	5345	103
H(11C)	10435	3190	5433	103

H(12A)	11211	3328	3816	88
H(12B)	10775	4074	4339	88
H(12C)	10022	3883	3603	88
H(13A)	8280	975	1438	65
H(13B)	9139	634	2092	65
H(14A)	7652	-416	1760	60
H(14B)	6541	230	1908	60
H(15A)	5842	1782	1981	111
H(15B)	6828	2294	1511	111
H(15C)	6296	2692	2242	111
H(16A)	9810	2221	2648	91
H(16B)	8824	2973	2649	91
H(16C)	9203	2539	1899	91
H(17A)	9461	-654	2993	78
H(17B)	8604	-1422	2734	78
H(17C)	8747	-1210	3580	78
H(18A)	5302	-713	2946	65
H(18B)	6060	-1293	3505	65
H(18C)	6207	-1431	2650	65
H(19A)	8476	-654	4978	64
H(19B)	7181	-1020	4684	64
H(19C)	7378	-844	5533	64
H(20A)	5376	178	5739	46
H(20B)	4978	887	5165	46
H(21A)	8610	997	5608	83
H(21B)	7532	674	6131	83
H(21C)	7373	1543	5700	83

***CH_2Cl_2**

	x	y	z	U(eq)
H(1S1)	1529	738	2867	101
H(1S2)	2991	940	2883	101

Data for 3

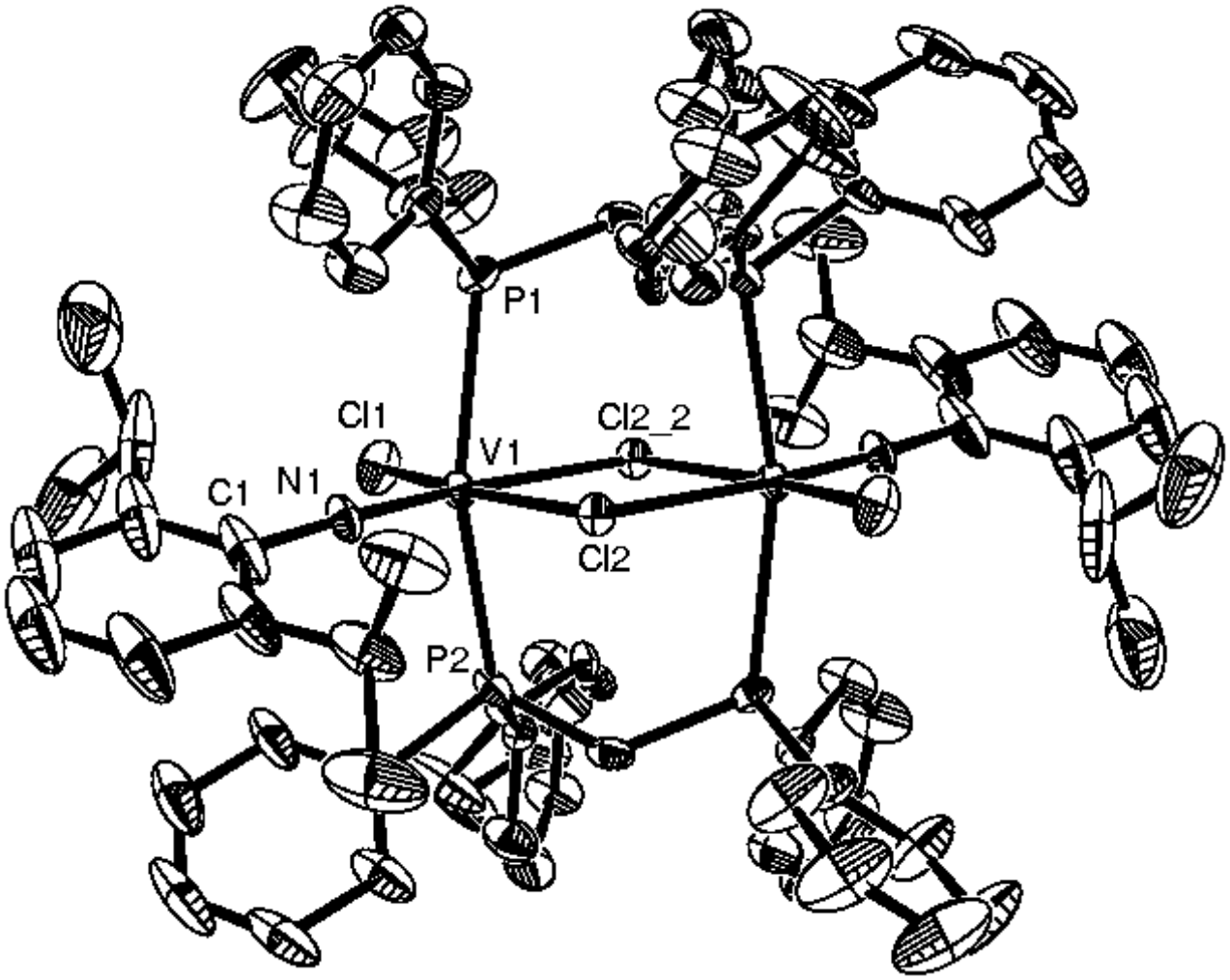

Table S1 .Crystal data, data collection and structure refinement

Empirical formula	$C_{38}H_{39}Cl_4NP_2V$
Formula weight	764.38
Temperature	180(2)K
Wavelength	0.71073Å
Crystal system, space group	triclinic, P $\bar{1}$
Unit cell dimensions	a = 11.745(2)Å α = 108.70(3)°
	b = 13.517(3)Å β = 111.31(3)°
	c = 13.862(3)Å γ = 92.33(3)°
Volume	1910.9(7)Å^3
Z, Calculated density	2, 1.328mg/m^3
Absorption coefficient	0.650mm^{-1}
F(000)	790
Crystal size	(0.35x0.20x0.15)mm
Crystal color	red
Crystal form	parallelepiped
Tube power	1.50kW
Tube voltage	50kV
Tube current	36mA
Collimator size	0.5mm
Detector distance	70.0mm
2theta range	(3.3 - 52.1)°
d(hkl) range	(12.453 - 0.809)Å
ϕ movement mode	rotation
ϕ start	0.0°
ϕ end	249.6°
ϕ incr.	1.3°
Number of exposures	192
Irradiation / exposure	6.00min
Measurement duration	33h
Theta range for data collection	(2.26 to 22.21)°
Index ranges	-12<=h<=12, -14<=k<=14, -14<=l<=14
Reflections collected / unique	12495 / 4576 [R(int) = 0.0307]
Completeness to 2θ = 44.42	94.8%
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4576 / 87 / 491
Absorption corrections	Semi-empirical*
Tmin – Tmax	0.397 - 0.794
Goodness-of-fit on F^2	1.077
Scheme of ponderation where P = $(F_o^2+2F_c^2)/3$	Weight = $1/[\sigma^2(F_o^2)+(0.0982P)^2+0.890P]$
Final R indices [I>2σ(I)]	R1 = 0.0507, wR2 = 0.1355
R indices (all data)	R1 = 0.0626, wR2 = 0.1450
Largest diff. peak and hole	(0.820 and -0.820)e.Å^{-3}

*DIFABS - N. Walker and D. Stuart, Acta Crystallogr., Sect A 1983, 39, 158-166.

Table S2. Atomic coordinates (x10^4) and equivalent isotropic displacement parameters (Å^{2}x10^3), U(eq) is defined as one third of the trace of the orthogonalized Uij tensor

	x	y	z	U(eq)
V	1389(1)	5811(1)	1325(1)	17(1)
P(1)	2020(1)	3952(1)	1135(1)	21(1)
P(2)	519(1)	7393(1)	916(1)	24(1)
Cl(1)	3250(1)	6513(1)	1337(1)	33(1)
Cl(2)	-713(1)	4962(1)	765(1)	20(1)
N	1695(4)	6318(3)	2692(3)	24(1)
C(1)	1963(6)	6815(4)	3820(4)	46(1)
C(2)	3219(7)	7169(5)	4565(4)	63(2)
C(3)	3450(9)	7714(6)	5680(5)	83(2)
C(4)	2508(9)	7892(6)	6025(5)	87(2)
C(5)	1285(9)	7533(5)	5312(5)	75(2)
C(6)	980(7)	6981(4)	4175(4)	55(1)
C(21)	4256(7)	6950(7)	4186(5)	86(3)
C(22)	4962(9)	6147(6)	4624(8)	103(3)
C(23)	5202(8)	7964(8)	4511(9)	112(4)
C(61)	-357(6)	6554(4)	3393(5)	52(2)
C(62)	-803(7)	5474(5)	3379(7)	66(2)
C(63)	-1273(9)	7289(6)	3605(7)	83(3)
C(111)	3595(5)	3889(4)	1209(4)	36(1)
C(112)	3876(6)	3964(6)	339(6)	58(2)
C(113)	5054(6)	3906(7)	360(7)	77(2)
C(114)	5963(7)	3808(7)	1246(8)	87(3)
C(115)	5723(6)	3765(7)	2119(7)	81(2)
C(116)	4535(5)	3783(5)	2101(5)	54(2)
C(121)	1946(4)	3353(3)	2120(4)	22(1)
C(122)	2120(5)	2314(4)	2002(4)	41(1)
C(123)	2027(5)	1874(4)	2740(4)	39(1)
C(124)	1773(5)	2451(4)	3603(5)	42(1)
C(125)	1637(7)	3494(5)	3766(5)	56(2)
C(126)	1716(5)	3936(4)	3016(4)	39(1)
C(131)	1182(4)	2873(3)	-231(4)	27(1)
C(211)	819(5)	8489(3)	2221(4)	37(1)
C(212)	2044(6)	8783(4)	2998(4)	50(2)
C(213)	2356(8)	9571(5)	4025(5)	67(2)
C(214)	1449(10)	10065(5)	4287(5)	76(3)

C(215)	257(8)	9800(4)	3538(5)	63(2)
C(216)	-76(7)	8998(4)	2487(5)	51(2)
C(21A)	1170(20)	8010(9)	158(15)	18(5)
C(22A)	1096(14)	7419(8)	-880(11)	18(4)
C(23A)	1519(18)	7873(12)	-1487(13)	24(4)
C(24A)	2023(16)	8918(14)	-1052(11)	54(6)
C(25A)	2118(19)	9530(8)	-6(12)	82(6)
C(26A)	1672(17)	9090(8)	589(11)	66(5)
C(21B)	890(30)	8090(9)	123(18)	29(7)
C(22B)	1492(14)	7651(10)	-546(14)	35(5)
C(23B)	1800(20)	8174(14)	-1162(15)	49(7)
C(24B)	1503(16)	9145(11)	-1097(11)	40(4)
C(25B)	890(13)	9605(7)	-433(9)	50(3)
C(26B)	589(12)	9094(7)	182(9)	40(3)

***CH_2Cl_2**

	x	y	z	U(eq)
C(100)	4871(10)	8804(9)	1235(8)	140(3)
Cl(1A)	6488(5)	8965(5)	2165(6)	132(2)
Cl(2A)	4455(9)	10081(7)	1688(9)	262(6)
Cl(1B)	5847(9)	8774(5)	499(11)	221(5)
Cl(2B)	5315(11)	9560(6)	2572(6)	207(3)

Table S3. Full list of bond lengths [Å] and angles [°], e.s.d's in parentheses refers to the last significant digit

V	- N	1.685(3)	V	- Cl(1)	2.3396(14)
V	- Cl(2)	2.4128(15)	V	- Cl(2)#1	2.5365(15)
V	- P(2)	2.5452(15)	V	- P(1)	2.6068(15)
P(1)	- C(121)	1.822(5)	P(1)	- C(111)	1.823(5)
P(1)	- C(131)	1.855(4)	P(2)	- C(21B)	1.805(13)
P(2)	- C(131)#1	1.835(5)	P(2)	- C(211)	1.835(4)
P(2)	- C(21A)	1.864(11)	Cl(2)	- V#1	2.5365(15)
N	- C(1)	1.396(6)	C(1)	- C(2)	1.410(10)
C(1)	- C(6)	1.411(9)	C(2)	- C(3)	1.398(9)
C(2)	- C(21)	1.497(11)	C(3)	- C(4)	1.358(12)
C(4)	- C(5)	1.371(12)	C(5)	- C(6)	1.408(8)
C(6)	- C(61)	1.504(10)	C(21)	- C(22)	1.538(13)
C(21)	- C(23)	1.559(10)	C(61)	- C(62)	1.522(9)
C(61)	- C(63)	1.537(9)	C(111)	- C(116)	1.379(8)

C(111) - C(112)	1.391(8)	C(112) - C(113)	1.380(8)
C(113) - C(114)	1.352(11)	C(114) - C(115)	1.356(11)
C(115) - C(116)	1.388(9)	C(121) - C(126)	1.368(7)
C(121) - C(122)	1.393(7)	C(122) - C(123)	1.371(8)
C(123) - C(124)	1.345(8)	C(124) - C(125)	1.381(8)
C(125) - C(126)	1.380(8)	C(131) - P(2)#1	1.835(5)
C(211) - C(216)	1.376(8)	C(211) - C(212)	1.392(9)
C(212) - C(213)	1.383(8)	C(213) - C(214)	1.376(12)
C(214) - C(215)	1.353(12)	C(215) - C(216)	1.411(8)
C(21A) - C(22A)	1.372(12)	C(21A) - C(26A)	1.397(13)
C(22A) - C(23A)	1.395(11)	C(23A) - C(24A)	1.355(15)
C(24A) - C(25A)	1.381(14)	C(25A) - C(26A)	1.383(12)
C(21B) - C(22B)	1.372(12)	C(21B) - C(26B)	1.398(13)
C(22B) - C(23B)	1.395(11)	C(23B) - C(24B)	1.355(15)
C(24B) - C(25B)	1.379(14)	C(25B) - C(26B)	1.382(12)

***CH_2Cl_2**

C(100) - Cl(2B)	1.671(11)	C(100) - Cl(1B)	1.784(12)
C(100) - Cl(2A)	1.792(13)	C(100) - Cl(1A)	1.823(11)

N	- V	- Cl(1)	100.13(14)	N	- V	- Cl(2)	95.50(14)
Cl(1)	- V	- Cl(2)	164.07(5)	N	- V	- Cl(2)#1	174.59(14)
Cl(1)	- V	- Cl(2)#1	84.78(6)	Cl(2)	- V	- Cl(2)#1	79.49(6)
N	- V	- P(2)	95.03(14)	Cl(1)	- V	- P(2)	87.54(5)
Cl(2)	- V	- P(2)	88.09(5)	Cl(2)#1	- V	- P(2)	82.84(5)
N	- V	- P(1)	100.88(14)	Cl(1)	- V	- P(1)	92.62(5)
Cl(2)	- V	- P(1)	87.37(5)	Cl(2)#1	- V	- P(1)	81.04(5)
P(2)	- V	- P(1)	163.80(5)	C(121)	- P(1)	- C(111)	102.9(2)
C(121)	- P(1)	- C(131)	103.3(2)	C(111)	- P(1)	- C(131)	98.1(2)
C(121)	- P(1)	- V	118.74(14)	C(111)	- P(1)	- V	114.56(18)
C(131)	- P(1)	- V	116.34(15)	C(21B)	- P(2)	- C(131)#1	99.8(9)
C(21B)	- P(2)	- C(211)	101.3(5)	C(131)#1	- P(2)	- C(211)	102.6(2)
C(131)#1	- P(2)	- C(21A)	109.1(8)	C(211)	- P(2)	- C(21A)	103.6(5)
C(21B)	- P(2)	- V	127.7(7)	C(131)#1	- P(2)	- V	112.57(15)
C(211)	- P(2)	- V	109.83(18)	C(21A)	- P(2)	- V	117.7(7)
V	- Cl(2)	- V#1	100.51(6)	C(1)	- N	- V	175.6(3)
N	- C(1)	- C(2)	118.4(6)	N	- C(1)	- C(6)	119.4(5)
C(2)	- C(1)	- C(6)	122.2(5)	C(3)	- C(2)	- C(1)	116.8(7)
C(3)	- C(2)	- C(21)	121.5(7)	C(1)	- C(2)	- C(21)	121.7(5)
C(4)	- C(3)	- C(2)	121.4(8)	C(3)	- C(4)	- C(5)	122.3(6)
C(4)	- C(5)	- C(6)	119.5(8)	C(5)	- C(6)	- C(1)	117.8(7)

C(5)	- C(6)	- C(61)	119.9(6)	C(1)	- C(6)	- C(61)	122.3(4)
C(2)	- C(21)	- C(22)	111.4(7)	C(2)	- C(21)	- C(23)	113.8(8)
C(22)	- C(21)	- C(23)	109.0(6)	C(6)	- C(61)	- C(62)	111.8(6)
C(6)	- C(61)	- C(63)	115.1(6)	C(62)	- C(61)	- C(63)	108.6(5)
C(116)	- C(111)	- C(112)b	118.1(5)	C(116)	- C(111)	- P(1)	122.7(4)
C(112)	- C(111)	- P(1)	119.1(4)	C(113)	- C(112)	- C(111)	120.7(6)
C(114)	- C(113)	- C(112)	120.1(7)	C(113)	- C(114)	- C(115)	120.3(6)
C(114)	- C(115)	- C(116)	120.7(7)	C(111)	- C(116)	- C(115)	120.0(6)
C(126)	- C(121)	- C(122)	118.1(5)	C(126)	- C(121)	- P(1)	119.5(4)
C(122)	- C(121)	- P(1)	122.5(3)	C(123)	- C(122)	- C(121)	121.2(5)
C(124)	- C(123)	- C(122)	120.0(5)	C(123)	- C(124)	- C(125)	120.2(5)
C(126)	- C(125)	- C(124)	119.9(5)	C(121)	- C(126)	- C(125)	120.6(5)
P(2)#1	- C(131)	- P(1)	121.5(2)	C(216)	- C(211)	- C(212)	119.1(5)
C(216)	- C(211)	- P(2)	124.7(5)	C(212)	- C(211)	- P(2)	116.2(4)
C(213)	- C(212)	- C(211)	120.6(7)	C(214)	- C(213)	- C(212)	119.8(7)
C(215)	- C(214)	- C(213)	120.5(6)	C(214)	- C(215)	- C(216)	120.3(7)
C(211)	- C(216)	- C(215)	119.7(7)	C(22A)	- C(21A)	- C(26A)	118.0(8)
C(22A)	- C(21A)	- P(2)	120.2(9)	C(26A)	- C(21A)	- P(2)	121.7(9)
C(21A)	- C(22A)	- C(23A)	121.4(9)	C(24A)	- C(23A)	- C(22A)	120.0(11)
C(23A)	- C(24A)	- C(25A)	119.8(9)	C(24A)	- C(25A)	- C(26A)	120.5(8)
C(25A)	- C(26A)	- C(21A)	120.3(8)	C(22B)	- C(21B)	- C(26B)	117.9(8)
C(22B)	- C(21B)	- P(2)	120.3(10)	C(26B)	- C(21B)	- P(2)	121.7(11)
C(21B)	- C(22B)	- C(23B)	121.6(10)	C(24B)	- C(23B)	- C(22B)	119.8(11)
C(23B)	- C(24B)	- C(25B)	119.8(9)	C(24B)	- C(25B)	- C(26B)	120.7(8)
C(25B)	- C(26B)	- C(21B)	120.1(8)				

***CH_2Cl_2**

Cl(2B)	- C(100)	- Cl(1B)	122.5(8)	Cl(2A)	- C(100)	- Cl(1A)	104.8(6)

Symmetry transformations used to generate equivalent atoms: #1 -x,-y+1,-z

Table S4. Anisotropic displacement parameters ($Å^2x10^3$), the anisotropic displacement factor exponent takes the form: $exp[-2p^2(U_{11}h^2a^{*2} + U_{22}k_2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}klb^*c^*)]$

	U11	U22	U33	U23	U13	U12
V	20(1)	18(1)	13(1)	5(1)	7(1)	0(1)
P(1)	21(1)	28(1)	22(1)	13(1)	13(1)	11(1)
P(2)	40(1)	15(1)	17(1)	4(1)	13(1)	2(1)
Cl(1)	22(1)	45(1)	35(1)	19(1)	10(1)	-4(1)
Cl(2)	21(1)	25(1)	17(1)	7(1)	11(1)	3(1)

N	36(2)	19(2)	14(2)	4(2)	8(2)	-5(2)
C(1)	92(3)	22(2)	19(2)	4(2)	20(2)	-18(2)
C(2)	102(3)	42(3)	21(2)	8(2)	5(2)	-35(3)
C(3)	126(4)	61(3)	25(2)	5(2)	6(2)	-43(3)
C(4)	149(4)	58(3)	27(3)	-1(3)	24(2)	-25(3)
C(5)	140(4)	48(3)	30(2)	-3(2)	43(2)	-15(3)
C(6)	114(3)	28(2)	24(2)	2(2)	38(2)	-8(3)
C(21)	60(4)	131(7)	29(3)	28(4)	-15(3)	-54(5)
C(22)	104(7)	71(5)	85(6)	23(5)	-8(5)	-39(5)
C(23)	65(5)	131(8)	124(8)	97(7)	-19(5)	-32(5)
C(61)	89(5)	40(3)	47(3)	10(3)	53(3)	17(3)
C(62)	86(5)	44(3)	89(5)	20(4)	60(4)	17(3)
C(63)	151(8)	65(4)	91(5)	43(4)	97(6)	54(5)
C(111)	28(3)	51(3)	46(3)	28(3)	25(2)	19(2)
C(112)	45(4)	99(5)	71(4)	57(4)	43(3)	32(3)
C(113)	51(4)	127(7)	109(6)	72(5)	63(5)	46(4)
C(114)	40(4)	135(7)	139(8)	85(6)	61(5)	45(4)
C(115)	35(4)	141(7)	101(6)	82(6)	28(4)	39(4)
C(116)	28(3)	90(5)	63(4)	50(4)	19(3)	21(3)
C(121)	18(2)	28(2)	23(2)	12(2)	8(2)	6(2)
C(122)	66(4)	35(3)	29(3)	15(3)	21(3)	19(3)
C(123)	59(4)	30(3)	31(3)	19(2)	14(3)	10(2)
C(124)	47(3)	50(3)	44(3)	31(3)	24(3)	6(3)
C(125)	102(5)	50(4)	48(3)	28(3)	53(4)	27(3)
C(126)	65(4)	31(3)	37(3)	18(3)	33(3)	13(3)
C(131)	40(3)	24(2)	23(2)	10(2)	18(2)	18(2)
C(211)	73(4)	13(2)	23(3)	5(2)	20(3)	3(2)
C(212)	86(5)	20(3)	28(3)	0(2)	16(3)	-8(3)
C(213)	116(6)	28(3)	32(3)	4(3)	12(4)	-15(4)
C(214)	174(9)	19(3)	27(3)	1(3)	38(5)	-3(4)
C(215)	137(7)	26(3)	41(4)	10(3)	52(5)	24(4)
C(216)	102(5)	26(3)	34(3)	10(3)	38(3)	17(3)
C(21A)	28(12)	14(8)	25(9)	14(6)	19(9)	4(6)
C(22A)	17(9)	19(6)	15(7)	5(5)	6(7)	-4(5)
C(23A)	28(7)	14(7)	28(9)	6(6)	11(8)	-2(7)
C(24A)	80(15)	36(11)	74(12)	23(9)	58(11)	16(9)
C(25A)	167(18)	19(6)	75(11)	7(7)	78(12)	-17(8)
C(26A)	138(15)	21(6)	72(9)	7(6)	85(11)	5(8)
C(21B)	26(11)	21(9)	28(9)	5(7)	-1(6)	5(6)
C(22B)	35(12)	25(7)	48(12)	21(8)	13(11)	0(7)
C(23B)	71(17)	43(14)	44(13)	16(11)	36(13)	0(11)

C(24B)	65(11)	24(8)	50(9)	30(7)	31(7)	6(7)
C(25B)	79(10)	19(5)	44(7)	18(5)	11(7)	3(6)
C(26B)	60(8)	26(6)	40(6)	13(5)	26(6)	8(6)

***CH_2Cl_2**

	U11	U22	U33	U23	U13	U12
C(100)	110(5)	120(6)	157(7)	22(6)	47(4)	-27(5)
Cl(1A)	104(4)	105(4)	164(6)	53(4)	25(4)	-15(3)
Cl(2A)	195(7)	142(5)	213(8)	-67(6)	-63(6)	35(5)
Cl(1B)	190(8)	110(4)	438(13)	78(7)	228(10)	6(5)
Cl(2B)	242(7)	131(5)	148(5)	25(4)	2(5)	-82(5)

Table S5. Hydrogen coordinates ($x10^4$) and isotropic displacement parameters ($Å^2x10^3$)

	x	y	z	U(eq)
H(3)	4284	7966	6207	99
H(4)	2703	8279	6787	104
H(5)	648	7657	5583	90
H(21)	3877	6624	3357	103
H(22A)	5526	5919	4254	155
H(22B)	4367	5529	4471	155
H(22C)	5442	6480	5426	155
H(23A)	5917	7756	4342	168
H(23B)	5481	8371	5307	168
H(23C)	4801	8402	4089	168
H(61)	-408	6449	2630	62
H(62A)	-1688	5241	2896	99
H(62B)	-676	5527	4134	99
H(62C)	-331	4957	3097	99
H(63A)	-2103	6983	3016	124
H(63B)	-1001	7987	3611	124
H(63C)	-1300	7367	4324	124
H(112)	3250	4057	-275	70
H(113)	5228	3935	-250	93
H(114)	6773	3770	1258	104
H(115)	6374	3722	2749	97
H(116)	4368	3723	2704	65
H(122)	2308	1903	1398	50

H(123)	2141	1162	2642	47
H(124)	1687	2139	4103	50
H(125)	1490	3908	4392	68
H(126)	1609	4650	3124	46
H(13A)	1501	3011	-754	32
H(13B)	1439	2206	-146	32
H(212)	2670	8439	2821	60
H(213)	3194	9771	4549	80
H(214)	1662	10596	4998	92
H(215)	-359	10156	3722	76
H(216)	-915	8810	1965	61
H(22D)	748	6685	-1191	21
H(23D)	1454	7449	-2207	29
H(24A)	2311	9229	-1466	64
H(25A)	2493	10258	307	98
H(26A)	1708	9525	1295	80
H(22E)	1710	6973	-592	42
H(23E)	2211	7850	-1625	59
H(24B)	1717	9509	-1508	48
H(25B)	673	10282	-398	60
H(26B)	179	9424	646	48