Neutral and cationic vanadium(III) alkyl and allyl complexes with a cyclopentadienyl-amine ancillary ligand

Liu, GH; Beetstra, DJ; Meetsma, A; Hessen, B; Liu, Guohua; Beetstra, Dirk J.

Published in:
Organometallics

DOI:
10.1021/om049660t

Publication date:
2004

Supporting Material

To: “Neutral and Cationic V(III) Alkyl and Allyl Complexes with a Cyclopentadienyl-Amine Ancillary Ligand” by G. Liu, D.J. Beetstra, A. Meetsma and B. Hessen.

*Part I: Structure of [h^5,h^1-$C_5H_4(CH)_2NMe_2$]$VCl_2(PMe_3)$ (**1**)*

Abstract. $C_{12}H_{23}Cl_2NPV$, M_r = 334.12, monoclinic, $P2_1/n$, a = 10.119(1), b = 11.660(1), c = 13.184(1) Å, ß= 98.75(1)°, V = 1537.4(2) Å^3, Z = 4, D_x = 1. 444 gcm^{-3}, λ(MoK$\bar{a}$) = 0.71073 Å, μ = 10.8 cm^{-1}, F(000) = 696, T = 180 K, *GooF* = 1.073, $wR(F^2)$ = 0.0942 for 3502 reflections and 246 parameters and $R(F)$ = 0.0355 for 2814 reflections obeying $F_o \geq 4.0\ \sigma(F_o)$ criterion of observability.

The asymmetric unit consists of one molecule of the title compound.

Experimental

X-ray diffraction: Crystal and Molecular Structure.

The crystal, a parallelepiped of approximate size 0.250 x 0.250 x 0.150 mm., used for characterization and data collection was glued on top of a glass fiber by using inert-atmosphere handling techniques and was transferred into the cold nitrogen stream of the low temperature unit[1] mounted on an Enraf-Nonius *CAD-4F*[2] diffractometer, interfaced to a *INDY* (*Silicon Graphics*) *UNIX* computer (Mo tube, 50 kV, 40 mA, monochromated Mo-K$\bar{a}$ radiation, $\Delta\omega = 1.00 + 0.34$ tg θ).

Unit cell parameters[3] and orientation matrix were determined from a least-squares treatment of the *SET4*[4] setting angles of 22 reflections in the range 16.49° < θ < 21.84°. The unit cell was identified as monoclinic, space group $P2_1/n$. Reduced cell calculations did not indicate any higher metric lattice symmetry[5] and examination of the final atomic coordinates of the structure did not yield extra metric symmetry elements.[6,7]

The intensities of three standard reflections, monitored every three hours of X-ray exposure time, showed no greater fluctuations during data collection than those expected from Poisson statistics. A 360° ψ-scan for a reflection close to axial (024) showed a variation in intensity of less than 9% about the mean value. Intensity data were corrected for Lorentz and polarization effects, scale variation, for absorption (psi-scans method,[8] as implemented in *PLATON*; the calculated transmission-factor range was: 1.020 - 1.208.) and reduced to F_o^2.[9]

The structure was solved by Patterson methods and extension of the model was accomplished by direct methods applied to difference structure factors using the program *DIRDIF*.[10] The positional and anisotropic displacement parameters for the non-hydrogen atoms were refined.

A subsequent difference Fourier synthesis resulted in the location of all the hydrogen atoms, which coordinates and isotropic displacement parameters were refined. Final refinement on F^2 carried out by full-matrix least-squares techniques. Convergence was reached at $wR(F^2)$ = 0.0942 for 3502 reflections and 246 parameters and $R(F)$ = 0.0355 for 2814 reflections with $F_o \geq 4.0\ \sigma(F_o)$. The final difference Fourier map was essentially featureless: no significant peaks (0.87(8) e/Å^3) having chemical meaning above the general background were observed.

The positional and anisotropic displacement parameters for the non-hydrogen atoms and isotropic displacement parameters for hydrogen atoms were refined on F^2 with full-matrix least-squares procedures minimizing the function $Q = \sum_h [w(|(F_o^2) - k(F_c^2)|)^2]$, where $w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$, $P = [\max(F_o^2,0) + 2F_c^2] / 3$, F_0 and F_c are the observed and calculated structure factor amplitudes, respectively; a (=0.0581) and b (= 0.2038) were refined.

Crystal data and numerical details on data collection and refinement are given in Table 1. Final fractional atomic coordinates, equivalent displacement parameters and anisotropic displacement parameters for the non-hydrogen atoms are given in Table 2. Molecular geometry data are collected in Table 3. Neutral atom scattering factors and anomalous dispersion corrections were taken from *International Tables for Crystallography*[11] All calculations were performed on a Pentium-III / Debian-Linux computer at the University of Groningen with the program packages *SHELXL*[12] (least-square refinements), *PLATON*[13] (calculation of geometric data and the *ORTEP*[15] illustrations) and a locally modified version of the program *PLUTO*[14] (preparation of illustrations).

The monoclinic unit cell contains four discrete units of the title compound separated by normal van der Waals distances[16].

No missed symmetry (*MISSYM*) or solvent-accessible voids were detected by procedures implemented in *PLATON.* [17,18]

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Table 1.

a. Crystal data and details of the structure determination.

Moiety_Formula	$C_{12}H_{23}Cl_2NPV$
Formula_Weight, g.mol^{-1}	334.12
Crystal system	monoclinic
Space group, no.[19]	$P2_1/n$, 14
a, Å	10.119(1)
b, Å	11.660(1)
c, Å	13.184(1)
β, deg	98.75(1)
V, Å^3	1537.4(2)
Formula_*Z*	4
SpaceGroup_*Z*	4
Z' (= Formula_Z / SpaceGroup_*Z)*	1
ρ_{calc}, g.cm^{-3}	1.444
F(000), electrons	696
μ(Mo K$\bar{a}$), cm^{-1}	10.8
Color, habit	blue-green, block
Approx. crystal dimension, mm	0.15 x 0.25 x 0.25

b. Data collection.

λ (Mo K$\bar{a}$), Å	0.71073
Monochromator	Graphite
Temperature, K	180(2)
θ range; min. max., deg	2.34, 27.47
ω/2θ scan, deg	$\Delta\omega = 1.00 + 0.34 \text{ tg } \theta$
Index ranges	h: -13→12; k: 0→15; l: 0→17
Crystal-to-receiving-aperture-distance, mm	173
Horizontal-, vertical-aperture, mm	4.0; 4.0
Reference reflections,	222, 2.6
r.m.s. dev. in %	204, 2.5
	040, 1.3
Drift correction	1.000 – 1.045
X-ray exposure time, h	89.4
Total data	3833
Unique data	3502
Data with criterion: ($F_o \geq 4.0\ \sigma\ (F_o)$)	2814
$R_{int} = \sum [\lvert F_o^2 - F_o^2 \text{(mean)}\rvert] / \sum [F_o^2]$	0.0246
$R_{sig} = \sum \sigma(F_o^2) / \sum [F_o^2]$	0.0251

c. Refinement.

Number of reflections	3502
Number of refined parameters	246
Final agreement factors:	
$wR(F^2) = [\sum [w(F_o^2 - F_c^2)^2] / \sum [w(F_o^2)^2]]^{1/2}$	0.0942
Weighting scheme: *a, b*	0.0581, 0.2038
$w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$	
and $P = [\max(F_o^2,0) + 2F_c^2] / 3$	
$R(F) = \sum (\|\|F_o\| - \|F_c\|\|) / \sum \|F_o\|$	0.0355
For $F_o > 4.0\ \sigma(F_o)$	
$GooF = S = [\sum [w(F_o^2 - F_c^2)^2] / (n-p)]^{1/2}$	1.073
n = number of reflections	
p = number of parameters refined	
Residual electron density in final	
Difference Fourier map, e/Å^3	-0.44, 0.87(8)
Max. (shift/s) final cycle	<0.000
Average (shift/σ) final cycle	0.000

Table 2. Final fractional atomic coordinates and equivalent isotropic displacement parameters with s.u.'s in parentheses.

Atoms of the Asymmetric Unit.

Non-Hydrogen parameters

Atom	*x*	*y*	*z*	U_{eq} (Å^2)*
V	0.04866(4)	0.26745(3)	0.35304(3)	0.01338(11)
Cl(1)	0.07018(6)	0.08487(5)	0.27793(5)	0.02751(17)
Cl(2)	0.11608(5)	0.28291(5)	0.53466(4)	0.02210(17)
P	0.29927(6)	0.25808(5)	0.36948(4)	0.01736(17)
N	-0.15124(19)	0.19556(17)	0.39698(15)	0.0187(5)
C(1)	0.0036(2)	0.4633(2)	0.34711(19)	0.0224(7)
C(2)	0.1003(3)	0.4405(2)	0.28392(19)	0.0224(7)
C(3)	0.0441(3)	0.3687(2)	0.20304(19)	0.0245(7)
C(4)	-0.0883(2)	0.3461(2)	0.21674(18)	0.0232(7)
C(5)	-0.1139(2)	0.4033(2)	0.30637(19)	0.0215(7)
C(6)	-0.2367(3)	0.3918(2)	0.3564(2)	0.0287(8)
C(7)	-0.2176(2)	0.2958(2)	0.4358(2)	0.0224(7)
C(8)	-0.2430(3)	0.1425(3)	0.3111(2)	0.0283(8)
C(9)	-0.1308(3)	0.1075(2)	0.4784(2)	0.0282(8)
C(10)	0.3960(3)	0.3711(3)	0.4426(2)	0.0284(8)
C(11)	0.3660(3)	0.2579(3)	0.2488(2)	0.0282(8)
C(12)	0.3688(3)	0.1288(2)	0.4329(2)	0.0241(7)

Hydrogen parameters

Atom	*x*	*y*	*z*	*Ueq (Å2)**
H(1)	0.018(3)	0.515(3)	0.409(2)	0.044(9)
H(2)	0.184(3)	0.469(2)	0.293(2)	0.027(7)
H(3)	0.087(3)	0.339(3)	0.149(3)	0.044(9)
H(4)	-0.144(3)	0.292(3)	0.172(2)	0.034(8)
H(6)	-0.313(3)	0.375(3)	0.299(2)	0.030(8)
H(6')	-0.253(3)	0.462(3)	0.391(2)	0.043(9)
H(7)	-0.299(3)	0.271(2)	0.454(2)	0.023(7)
H(7')	-0.164(3)	0.321(3)	0.495(2)	0.032(8)
H(8)	-0.199(3)	0.083(3)	0.286(2)	0.028(8)
H(8')	-0.323(3)	0.104(3)	0.339(2)	0.040(9)
H(8'')	-0.272(3)	0.198(3)	0.261(3)	0.039(9)
H(9)	-0.224(3)	0.087(2)	0.500(2)	0.027(7)
H(9')	-0.086(3)	0.045(3)	0.453(2)	0.032(8)
H(9'')	-0.071(3)	0.135(3)	0.536(3)	0.039(9)
H(10)	0.381(4)	0.380(3)	0.508(3)	0.051(10)
H(10')	0.387(4)	0.442(4)	0.412(3)	0.079(14)
H(10'')	0.503(4)	0.357(4)	0.443(3)	0.075(13)
H(11)	0.358(3)	0.333(3)	0.215(2)	0.035(8)
H(11')	0.446(4)	0.230(3)	0.258(3)	0.052(11)
H(11'')	0.320(3)	0.206(3)	0.206(2)	0.023(7)
H(12)	0.343(3)	0.118(3)	0.492(2)	0.029(8)
H(12')	0.331(3)	0.068(3)	0.394(2)	0.035(9)
H(12'')	0.460(4)	0.126(3)	0.437(3)	0.055(11)

*) $U_{eq} = 1/3 \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a_i}.\mathbf{a_j}$ [20]

Anisotropic (displacement) parameters (Å^2)

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
V	0.01233(18)	0.01633(19)	0.01135(18)	-0.00015(13)	0.00141(13)	0.00027(13)
Cl(1)	0.0238(3)	0.0229(3)	0.0365(3)	-0.0125(2)	0.0067(2)	-0.0010(2)
Cl(2)	0.0182(3)	0.0345(3)	0.0129(3)	-0.0022(2)	0.00013(19)	0.0030(2)
P	0.0137(3)	0.0208(3)	0.0182(3)	-0.0008(2)	0.0044(2)	0.0004(2)
N	0.0149(9)	0.0201(9)	0.0207(10)	0.0014(8)	0.0016(7)	-0.0012(8)
C(1)	0.0258(12)	0.0166(10)	0.0241(12)	0.0038(9)	0.0013(10)	0.0016(9)
C(2)	0.0221(12)	0.0219(11)	0.0229(12)	0.0078(9)	0.0027(9)	-0.0009(9)
C(3)	0.0277(13)	0.0297(13)	0.0162(11)	0.0071(10)	0.0032(10)	0.0001(10)
C(4)	0.0228(12)	0.0286(12)	0.0162(11)	0.0057(10)	-0.0038(9)	-0.0016(10)
C(5)	0.0183(11)	0.0195(11)	0.0255(12)	0.0072(9)	-0.0002(9)	0.0053(9)
C(6)	0.0203(12)	0.0266(13)	0.0402(15)	0.0071(12)	0.0076(11)	0.008(1)
C(7)	0.0151(11)	0.0259(12)	0.0275(12)	0.0012(10)	0.0074(9)	0.0019(9)
C(8)	0.0208(12)	0.0306(14)	0.0324(14)	-0.0069(12)	0.0008(11)	-0.0082(11)
C(9)	0.0252(13)	0.0260(13)	0.0343(15)	0.0104(11)	0.0070(11)	0.0012(10)
C(10)	0.0198(12)	0.0330(14)	0.0316(15)	-0.0065(12)	0.0016(10)	-0.0052(10)
C(11)	0.0260(13)	0.0342(15)	0.0278(13)	0.0022(12)	0.0147(11)	0.0021(12)
C(12)	0.0230(12)	0.0274(13)	0.0221(12)	0.0012(10)	0.0041(10)	0.0077(10)

Thermal vibration amplitudes (Å^2)

$$F(\mathbf{h}) = F_o(\mathbf{h}) \exp\left(-2\pi^2 \sum_{i=1}^{3}\sum_{j=1}^{3} h_i h_j a_i^* a_j^* U_{ij}\right)$$

or

$$F(\mathbf{h}) = F_o(\mathbf{h}) \exp\left(-8\pi^2 U_{iso} (\sin(\theta)/\lambda)^2\right)$$

Table 3. Data on the geometry.

Standard deviations in the last decimal place are given in parentheses.

Interatomic Distances (Å)

V	-Cl(1) 2.3719(7)	P	-C(12) 1.813(3)
V	-Cl(2) 2.3947(7)	N	-C(7) 1.478(3)
V	-P 2.5145(8)	N	-C(8) 1.486(4)
V	-N 2.342(2)	N	-C(9) 1.477(3)
V	-C(1) 2.328(2)	C(1)	-C(2) 1.404(4)
V	-C(2) 2.306(2)	C(1)	-C(5) 1.413(3)
V	-C(3) 2.298(2)	C(2)	-C(3) 1.406(4)
V	-C(4) 2.288(2)	C(3)	-C(4) 1.404(4)
V	-C(5) 2.299(2)	C(4)	-C(5) 1.415(3)
P	-C(10) 1.827(3)	C(5)	-C(6) 1.499(4)
P	-C(11) 1.821(3)	C(6)	-C(7) 1.525(3)

Bond Angles (deg.)

Cl(1)	-V	-Cl(2)	117.05(3)	C(3)	-V	-C(5)	59.72(9)
Cl(1)	-V	-P	80.88(3)	C(4)	-V	-C(5)	35.93(8)
Cl(1)	-V	-N	85.39(5)	V	-P	-C(10)	117.81(10)
Cl(1)	-V	-C(1)	153.70(7)	V	-P	-C(11)	115.34(10)
Cl(1)	-V	-C(2)	125.42(7)	V	-P	-C(12)	113.04(10)
Cl(1)	-V	-C(3)	95.33(6)	C(10)	-P	-C(11)	102.82(14)
Cl(1)	-V	-C(4)	96.60(6)	C(10)	-P	-C(12)	102.55(13)
Cl(1)	-V	-C(5)	127.88(6)	C(11)	-P	-C(12)	103.45(14)
Cl(2)	-V	-P	77.70(2)	V	-N	-C(7)	104.93(13)
Cl(2)	-V	-N	84.55(5)	V	-N	-C(8)	115.07(15)
Cl(2)	-V	-C(1)	89.10(6)	V	-N	-C(9)	113.44(16)
Cl(2)	-V	-C(2)	106.38(7)	C(7)	-N	-C(8)	109.2(2)
Cl(2)	-V	-C(3)	141.94(7)	C(7)	-N	-C(9)	108.60(19)
Cl(2)	-V	-C(4)	143.29(6)	C(8)	-N	-C(9)	105.5(2)
Cl(2)	-V	-C(5)	107.41(7)	V	-C(1)	-C(2)	71.55(13)
P	-V	-N	149.40(5)	V	-C(1)	-C(5)	71.11(13)
P	-V	-C(1)	103.61(6)	C(2)	-C(1)	-C(5)	107.6(2)
P	-V	-C(2)	77.64(8)	V	-C(2)	-C(1)	73.20(14)
P	-V	-C(3)	89.11(8)	V	-C(2)	-C(3)	71.88(14)
P	-V	-C(4)	124.59(6)	C(1)	-C(2)	-C(3)	109.0(3)
P	-V	-C(5)	136.40(6)	V	-C(3)	-C(2)	72.56(14)
N	-V	-C(1)	100.79(7)	V	-C(3)	-C(4)	71.78(14)
N	-V	-C(2)	131.83(9)	C(2)	-C(3)	-C(4)	107.3(2)
N	-V	-C(3)	119.45(9)	V	-C(4)	-C(3)	72.56(14)
N	-V	-C(4)	83.97(7)	V	-C(4)	-C(5)	72.48(13)
N	-V	-C(5)	72.73(7)	C(3)	-C(4)	-C(5)	108.6(2)
C(1)	-V	-C(2)	35.26(9)	V	-C(5)	-C(1)	73.32(13)
C(1)	-V	-C(3)	59.26(9)	V	-C(5)	-C(4)	71.59(13)
C(1)	-V	-C(4)	59.21(8)	V	-C(5)	-C(6)	115.33(16)
C(1)	-V	-C(5)	35.56(8)	C(1)	-C(5)	-C(4)	107.49(19)
C(2)	-V	-C(3)	35.56(9)	C(1)	-C(5)	-C(6)	125.7(2)
C(2)	-V	-C(4)	59.04(9)	C(4)	-C(5)	-C(6)	126.5(2)
C(2)	-V	-C(5)	59.16(9)	C(5)	-C(6)	-C(7)	109.9(2)
C(3)	-V	-C(4)	35.65(9)	N	-C(7)	-C(6)	111.1(2)

*Part II: Structure of [h^5-$C_5H_4(CH)_2NMe_2$]$VMe_2(PMe_3)_2$ (**2**)*

Abstract. $C_{17}H_{38}NP_2V$, M_r = 369.38, monoclinic, $P2_1/c$, a = 14.1732(8), b = 9.6636(5), c = 16.6389(9) Å, ß = 111.624(1)°, V = 2118.5(2) Å^3, Z = 4, D_x = 1.158 gcm^{-3}, F(000) = 800, μ = 6.15 cm^{-1}, λ(MoK$\bar{a}$) = 0.71073 Å, T = 100(1) K, 17844 reflections measured, *GooF* = 1.027, *wR*(F^2) = 0.0692 for 4793 unique reflections and 342 parameters and *R*(F) = 0.0254 for 4335 reflections obeying $F_o \geq 4.0\ \sigma(F_o)$ criterion of observability.
The asymmetric unit consists of one molecule of the title compound.

Experimental

X-ray diffraction: Crystal and Molecular Structure.

A triangle-block-shaped crystal with the dimensions of 0.53 x 0.41 x 0.17 mm was mounted on top of a glass fiber, by using inert-atmosphere handling techniques, and aligned on a *Bruker*[1] *SMART APEX CCD* diffractometer (Platform with full three-circle goniometer). The diffractometer was equipped with a 4K *CCD* detector set 60.0 mm from the crystal. The crystal was cooled to 100(1) K using the *Bruker KRYOFLEX* low-temperature device. Intensity measurements were performed using graphite monochromated Mo-K$\bar{a}$ radiation from a sealed ceramic diffraction tube (*SIEMENS*). Generator settings were 50 KV/ 40 mA. *SMART* was used for preliminary determination of the unit cell constants and data collection control. The intensities of reflections of a hemisphere were collected by a combination of 3 sets of exposures (frames). Each set had a different ϕ angle for the crystal and each exposure covered a range of 0.3° in ω. A total of 1800 frames were collected with an exposure time of 10.0 seconds per frame. The overall data collection time was 9.2 h. Data integration and global cell refinement was performed with the program *SAINT*. The final unit cell was obtained from the *xyz* centroids of 5030 reflections after integration. Intensity data were corrected for Lorentz and polarization effects, scale variation, for decay and absorption: a multi-scan absorption correction was applied, based on the intensities of symmetry-related reflections measured at different angular settings (*SADABS*)[2], and reduced to F_o^2. The program suite *SHELXTL* was used for space group determination (*XPREP*).[1]

The unit cell[3] was identified as monoclinic; space group $P2_1/c$, was derived from the systematic extinctions. Reduced cell calculations did not indicate any higher metric lattice symmetry[4] and examination of the final atomic coordinates of the structure did not yield extra crystallographic or metric symmetry elements.[5,6]

The structure was solved by Patterson methods and extension of the model was accomplished by direct methods applied to difference structure factors using the program *DIRDIF*.[7] The

positional and anisotropic displacement parameters for the non-hydrogen atoms were refined. A subsequent difference Fourier synthesis resulted in the location of all the hydrogen atoms, which coordinates and isotropic displacement parameters were refined.

Final refinement on F^2 carried out by full-matrix least-squares techniques converged at $wR(F^2)$ = 0.0692 for 4793 reflections and $R(F)$ = 0.0254 for 4335 reflections with $F_o \geq 4.0\ \sigma(F_o)$ and 342 parameters. The final difference Fourier map was essentially featureless: no significant peaks (0.4(5) e/Å^3) having chemical meaning above the general background were observed.

The positional and anisotropic displacement parameters for the non-hydrogen atoms and isotropic displacement parameters for hydrogen atoms were refined on F^2 with full-matrix least-squares procedures minimizing the function $Q = \sum_h[w(|(F_o^2) - k(F_c^2)|)^2]$, where $w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$, $P = [\max(F_o^2,0) + 2F_c^2] / 3$, F_0 and F_c are the observed and calculated structure factor amplitudes, respectively; ultimately the suggested a (=0.0421) and b (= 0.4119) were used in the final refinement.

Crystal data and numerical details on data collection and refinement are given in Table 1. Final fractional atomic coordinates, equivalent displacement parameters and anisotropic displacement parameters for the non-hydrogen atoms are given in Table 2. Molecular geometry data are collected in Table 3. Neutral atom scattering factors and anomalous dispersion corrections were taken from *International Tables for Crystallography.*[10]

All refinement calculations and graphics were performed on a Pentium-III / Debian-Linux computer at the University of Groningen with the program packages *SHELXL*[11] (least-square refinements), a locally modified version of the program *PLUTO*[12] (preparation of illustrations) and *PLATON*[9] package (checking the final results for missed symmetry with the *MISSYM* option, solvent accessible voids with the *SOLV* option, calculation of geometric data and the *ORTEP*[9] illustrations).

The asymmetric unit contains one formula unit molecule with no atom setting at special position. The monoclinic unit cell contains four discrete molecules separated by normal van der Waals distances[13]. No classic hydrogen bonds, no missed symmetry (*MISSYM*) or solvent-accessible voids were detected by procedures implemented in *PLATON.*[14,15]

References.

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Table 1.

a. Crystal data and details of the structure determination.

Moiety_Formula	$C_{17}H_{38}NP_2V$
Formula_Weight, g.mol^{-1}	369.38
Crystal system	monoclinic
Space group, no.[16]	*P2*$_1$/*c*, 14
a, Å	14.1732(8)
b, Å	9.6636(5)
c, Å	16.6389(9)
β, deg	111.624(1)
V, Å^3	2118.5(2)
T range unit cell: min.-max., deg; reflections	2.48 - 27.45 ; 5030
Formula_*Z*	4
SpaceGroup_*Z*	4
Z' (= Formula_Z / SpaceGroup_*Z)*	1
ρ_{calc}, g.cm^{-3}	1.158
F(000), electrons	800
μ(Mo K$\bar{a}$), cm^{-1}	6.15
Color, habit	red, triangle-block
Approx. crystal dimension, mm	0.53 x 0.41 x 0.17

b. Data collection.

λ(Mo K$\bar{\alpha}$), Å	0.71073
Monochromator	Graphite
Measurement device type	*CCD* area-detector diffractometer
Detector Area resolution (pixels / mm)	4096 x 4096 / 62 x 62 (binned 512)
Temperature, K	100(1)
Measurement method	φ- and ω-scans
θ range; min. max., deg	2.49, 27.47
Index ranges	h: -18→18; k: -12→12; l: -21→21
Min.- Max. absorption transmission factor	0.7863 – 0.9026
X-ray exposure time, h	9.2
Total data	17844
Unique data	4793
Data with criterion: ($F_o \geq 4.0\ \sigma\ (F_o)$)	4335
$R_{int} = \sum [\lvert F_o^2 - F_o^2\ (\text{mean})\rvert] / \sum [F_o^2]$	0.0198
$R_{sig} = \sum \sigma(F_o^2) / \sum [F_o^2]$	0.0205

c. Refinement.

Number of reflections	4793
Number of refined parameters	342
Final agreement factors:	
$wR(F^2) = [\sum [w(F_o^2 - F_c^2)^2] / \sum [w(F_o^2)^2]]^{1/2}$	0.0692
Weighting scheme: *a, b*	0.0421, 0.4119
$w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$	
And $P = [\max(F_o^2,0) + 2F_c^2] / 3$	
$R(F) = \sum (\|\|F_o\| - \|F_c\|\|) / \sum \|F_o\|$	0.0254
For $F_o > 4.0\ \sigma(F_o)$	
$\text{GooF} = S = [\sum [w(F_o^2 - F_c^2)^2] / (n-p)]^{1/2}$	1.027
n = number of reflections	
p = number of parameters refined	
Residual electron density in final	
Difference Fourier map, e/Å^3	-0.15, 0.40(5)
Max. (shift/s) final cycle	<0.001
Average (shift/σ) final cycle	0.000

Table 2. Final fractional atomic coordinates and equivalent isotropic displacement parameters with s.u.'s in parentheses.
Atoms of the Asymmetric Unit.

Non-Hydrogen parameters

Atom	*x*	*y*	*z*	U_{eq} (Å^2)*
V	0.25392(1)	0.35769(2)	0.20140(1)	0.01480(6)
P1	0.24420(2)	0.13810(3)	0.12126(2)	0.01869(8)
P2	0.10432(2)	0.48335(3)	0.21020(2)	0.01944(9)
N	0.60088(7)	0.1565(1)	0.50679(6)	0.0202(3)
C1	0.36742(8)	0.47513(12)	0.31821(8)	0.0196(3)
C2	0.35397(9)	0.55261(12)	0.24199(8)	0.0226(3)
C3	0.39190(9)	0.47299(13)	0.18946(8)	0.0223(3)
C4	0.42836(8)	0.34661(12)	0.23276(8)	0.0192(3)
C5	0.41408(8)	0.34775(11)	0.31301(7)	0.0175(3)
C6	0.44979(8)	0.23845(12)	0.38182(7)	0.0191(3)
C7	0.55880(8)	0.26665(12)	0.44340(7)	0.0197(3)
C8	0.70918(9)	0.17886(16)	0.55358(9)	0.0295(4)
C9	0.54966(10)	0.14914(13)	0.56818(8)	0.0241(3)
C10	0.11489(11)	0.08173(18)	0.05829(11)	0.0369(4)
C11	0.30230(11)	0.13164(16)	0.03958(9)	0.0301(4)
C12	0.29707(13)	-0.02090(14)	0.18059(10)	0.0336(4)
C13	-0.01862(10)	0.40442(16)	0.14935(10)	0.0309(4)
C14	0.08102(12)	0.66237(14)	0.17124(12)	0.0348(5)
C15	0.09377(10)	0.50414(15)	0.31614(9)	0.0271(4)
C16	0.17712(10)	0.42753(15)	0.06671(8)	0.0275(4)
C17	0.20428(9)	0.21252(12)	0.28163(8)	0.0189(3)

Hydrogen parameters

Atom	*x*	*y*	*z*	U_{eq} (Å^2)*
H1	0.35235(-)	0.50096(-)	0.36664(-)	0.0222(10)
H2	0.32355(-)	0.64425(-)	0.22844(-)	0.0272(12)
H3	0.38951(-)	0.50063(-)	0.13441(-)	0.0270(11)
H4	0.45618(-)	0.27444(-)	0.21240(-)	0.0258(10)
H6	0.44786(-)	0.15042(-)	0.35498(-)	0.0206(11)
H6'	0.40459(-)	0.23698(-)	0.41430(-)	0.0253(11)
H7	0.60238(-)	0.27233(-)	0.40844(-)	0.0266(11)
H7'	0.56331(-)	0.35630(-)	0.47383(-)	0.0280(11)
H8	0.72216(-)	0.26696(-)	0.58672(-)	0.0338(12)
H8'	0.74243(-)	0.18442(-)	0.51348(-)	0.0378(13)
H8"	0.73506(-)	0.10317(-)	0.59000(-)	0.0427(13)
H9	0.58399(-)	0.08286(-)	0.61241(-)	0.0306(11)
H9'	0.48102(-)	0.12122(-)	0.53994(-)	0.0285(12)
H9"	0.55284(-)	0.23717(-)	0.59782(-)	0.0290(11)
H10	0.08194(-)	0.15055(-)	0.01946(-)	0.0575(16)
H10'	0.08135(-)	0.07112(-)	0.09594(-)	0.0480(14)
H10"	0.11527(-)	-0.00436(-)	0.02782(-)	0.0484(14)
H11	0.28855(-)	0.04312(-)	0.00894(-)	0.0345(12)
H11'	0.37307(-)	0.14148(-)	0.06613(-)	0.0433(14)
H11"	0.27839(-)	0.20113(-)	0.00201(-)	0.0452(14)
H12	0.26497(-)	-0.04703(-)	0.21866(-)	0.0444(13)
H12'	0.36960(-)	-0.00808(-)	0.21032(-)	0.0470(14)
H12"	0.28310(-)	-0.09896(-)	0.14038(-)	0.0362(12)
H13	-0.06955(-)	0.45589(-)	0.15731(-)	0.0563(15)
H13'	-0.02084(-)	0.31364(-)	0.16469(-)	0.0356(12)
H13"	-0.02880(-)	0.40262(-)	0.08975(-)	0.0438(13)
H14	0.07743(-)	0.66417(-)	0.11503(-)	0.0447(13)
H14'	0.13786(-)	0.71788(-)	0.20572(-)	0.0418(13)
H14"	0.01490(-)	0.69312(-)	0.17311(-)	0.0395(13)
H15	0.03277(-)	0.55109(-)	0.31058(-)	0.0371(13)
H15'	0.15110(-)	0.55545(-)	0.35157(-)	0.0429(13)
H15"	0.09403(-)	0.41719(-)	0.34286(-)	0.0331(12)
H16	0.10725(-)	0.39451(-)	0.03645(-)	0.0389(14)
H16'	0.21245(-)	0.39992(-)	0.03073(-)	0.0322(12)
H16"	0.17758(-)	0.53014(-)	0.06383(-)	0.0503(13)
H17	0.14338(-)	0.17830(-)	0.25779(-)	0.0320(13)
H17'	0.20624(-)	0.25852(-)	0.33259(-)	0.0417(13)
H17"	0.25116(-)	0.13806(-)	0.29723(-)	0.0292(12)

*) $U_{eq} = 1/3 \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a_i}.\mathbf{a_j}$ [17]

Anisotropic (displacement) parameters (Å^2)

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
V	0.01532(10)	0.01479(10)	0.01393(11)	0.00130(6)	0.00496(8)	0.00026(6)
P1	0.01781(14)	0.02052(15)	0.01783(15)	-0.00364(11)	0.00669(12)	-0.00254(10)
P2	0.01677(14)	0.01752(15)	0.02334(16)	0.00208(11)	0.00658(12)	0.00171(10)
N	0.0180(4)	0.0199(5)	0.0191(5)	0.0004(4)	0.0027(4)	0.0014(3)
C1	0.0200(5)	0.0180(5)	0.0196(6)	-0.0029(4)	0.0059(4)	-0.0020(4)
C2	0.0220(5)	0.0160(5)	0.0265(6)	0.0027(4)	0.0051(5)	-0.0026(4)
C3	0.0192(5)	0.0264(6)	0.0210(6)	0.0047(5)	0.0070(5)	-0.0048(4)
C4	0.0156(5)	0.0230(6)	0.0192(6)	-0.0009(4)	0.0065(4)	-0.0014(4)
C5	0.0149(5)	0.0184(5)	0.0176(5)	-0.0001(4)	0.0040(4)	-0.0025(4)
C6	0.0175(5)	0.0198(5)	0.0180(5)	0.0021(4)	0.0042(4)	-0.0016(4)
C7	0.0181(5)	0.0206(5)	0.0182(5)	0.0021(4)	0.0041(4)	-0.0025(4)
C8	0.0190(6)	0.0343(7)	0.0290(7)	-0.0013(6)	0.0017(5)	0.0021(5)
C9	0.0277(6)	0.0230(6)	0.0193(6)	0.0027(5)	0.0061(5)	-0.0026(5)
C10	0.0235(6)	0.0455(9)	0.0399(8)	-0.0187(7)	0.0096(6)	-0.0105(6)
C11	0.0343(7)	0.0341(8)	0.0264(7)	-0.0069(6)	0.0166(6)	-0.0029(6)
C12	0.0496(9)	0.0215(6)	0.0324(7)	-0.0018(5)	0.0184(7)	0.0056(6)
C13	0.0189(6)	0.0350(8)	0.0340(8)	0.0014(6)	0.0041(5)	-0.0005(5)
C14	0.0373(8)	0.0227(7)	0.0491(10)	0.0097(6)	0.0213(7)	0.0092(6)
C15	0.0258(6)	0.0282(7)	0.0305(7)	-0.0024(5)	0.0140(5)	0.0025(5)
C16	0.0298(7)	0.0318(7)	0.0198(6)	0.0067(5)	0.0077(5)	0.0043(5)
C17	0.0206(5)	0.0191(5)	0.0171(5)	0.0018(4)	0.0072(4)	0.0031(4)

Thermal vibration amplitudes (Å^2)

$$F(\mathbf{h}) = F_o(\mathbf{h}) \exp\left(-2\pi^2 \sum_{i=1}^{3}\sum_{j=1}^{3} h_i h_j a_i^* a_j^* U_{ij}\right)$$

or

$$F(\mathbf{h}) = F_o(\mathbf{h}) \exp\left(-8\pi^2 U_{iso} (\sin(\theta)/\lambda)^2\right)$$

Table 3. Data on the geometry.

Standard deviations in the last decimal place are given in parentheses.

Interatomic Distances (Å)

V	-P1 2.4829(4)	P2	-C14 1.8342(15)
V	-P2 2.4941(4)	P2	-C15 1.8342(15)
V	-C1 2.3128(12)	N	-C7 1.4621(15)
V	-C2 2.3042(12)	N	-C8 1.4588(17)
V	-C3 2.3220(14)	N	-C9 1.4567(17)
V	-C4 2.3336(13)	C1	-C2 1.4238(17)
V	-C5 2.3460(12)	C1	-C5 1.4150(16)
V	-C16 2.2057(13)	C2	-C3 1.4111(18)
V	-C17 2.2220(13)	C3	-C4 1.4154(17)
P1	-C10 1.8271(17)	C4	-C5 1.4224(17)
P1	-C11 1.8314(16)	C5	-C6 1.5018(16)
P1	-C12 1.8308(15)	C6	-C7 1.5312(16)
P2	-C13 1.8283(16)		

Hydrogen parameters:

C1	-H1 0.9403(-)	C11	-H11'' 0.8947(-)
C2	-H2 0.9743(-)	C12	-H12 0.9404(-)
C3	-H3 0.9427(-)	C12	-H12' 0.9706(-)
C4	-H4 0.9247(-)	C12	-H12'' 0.9789(-)
C6	-H6 0.9565(-)	C13	-H13 0.9254(-)
C6	-H6' 0.9787(-)	C13	-H13' 0.9174(-)
C7	-H7 0.9938(-)	C13	-H13'' 0.9482(-)
C7	-H7' 0.9936(-)	C14	-H14 0.9179(-)
C8	-H8 0.9940(-)	C14	-H14' 0.9619(-)
C8	-H8' 0.9497(-)	C14	-H14'' 0.9945(-)
C8	-H8'' 0.9343(-)	C15	-H15 0.9502(-)
C9	-H9 0.9616(-)	C15	-H15' 0.9490(-)
C9	-H9' 0.9503(-)	C15	-H15'' 0.95(-)
C9	-H9'' 0.9760(-)	C16	-H16 0.9841(-)
C10	-H10 0.9249(-)	C16	-H16' 0.9494(-)
C10	-H10' 0.9211(-)	C16	-H16'' 0.9929(-)
C10	-H10'' 0.9753(-)	C17	-H17 0.8714(-)
C11	-H11 0.9780(-)	C17	-H17' 0.9487(-)
C11	-H11' 0.9401(-)	C17	-H17'' 0.9484(-)

Bond Angles (deg.)

P1	-V	-P2	124.58(1)	V	-P1	-C10	114.09(6)
P1	-V	-C1	138.98(3)	V	-P1	-C11	118.46(5)
P1	-V	-C2	139.68(3)	V	-P1	-C12	119.80(5)
P1	-V	-C3	104.24(3)	C10	-P1	-C11	101.22(7)
P1	-V	-C4	85.72(3)	C10	-P1	-C12	100.59(8)
P1	-V	-C5	103.66(3)	C11	-P1	-C12	99.49(8)
P1	-V	-C16	78.87(4)	V	-P2	-C13	114.94(5)
P1	-V	-C17	79.39(3)	V	-P2	-C14	118.99(6)
P2	-V	-C1	93.14(3)	V	-P2	-C15	118.81(5)
P2	-V	-C2	91.69(3)	C13	-P2	-C14	100.78(8)
P2	-V	-C3	122.17(3)	C13	-P2	-C15	100.33(7)
P2	-V	-C4	149.63(3)	C14	-P2	-C15	99.70(7)
P2	-V	-C5	124.68(3)	C7	-N	-C8	110.27(10)
P2	-V	-C16	78.39(4)	C7	-N	-C9	111.42(10)
P2	-V	-C17	78.92(3)	C8	-N	-C9	109.35(10)
C1	-V	-C2	35.92(4)	V	-C1	-C2	71.71(7)
C1	-V	-C3	59.30(4)	V	-C1	-C5	73.61(7)
C1	-V	-C4	58.88(4)	C2	-C1	-C5	108.37(10)
C1	-V	-C5	35.35(4)	V	-C2	-C1	72.37(7)
C1	-V	-C16	129.32(5)	V	-C2	-C3	72.93(7)
C1	-V	-C17	94.65(4)	C1	-C2	-C3	107.97(10)
C2	-V	-C3	35.52(5)	V	-C3	-C2	71.55(8)
C2	-V	-C4	59.01(4)	V	-C3	-C4	72.75(7)
C2	-V	-C5	59.34(4)	C2	-C3	-C4	107.83(11)
C2	-V	-C16	93.88(5)	V	-C4	-C3	71.86(7)
C2	-V	-C17	129.56(4)	V	-C4	-C5	72.78(7)
C3	-V	-C4	35.40(4)	C3	-C4	-C5	108.64(10)
C3	-V	-C5	59.18(4)	V	-C5	-C1	71.04(7)
C3	-V	-C16	83.23(5)	V	-C5	-C4	71.83(7)
C3	-V	-C17	144.83(5)	V	-C5	-C6	126.03(8)
C4	-V	-C5	35.39(4)	C1	-C5	-C4	107.19(10)
C4	-V	-C16	109.07(5)	C1	-C5	-C6	126.8(1)
C4	-V	-C17	112.37(5)	C4	-C5	-C6	125.85(10)
C5	-V	-C16	142.01(5)	C5	-C6	-C7	111.0(1)
C5	-V	-C17	85.80(4)	N	-C7	-C6	113.32(10)
C16	-V	-C17	131.05(5)				

*Part III: Structure of {[h^5,h^2-C$_5$H$_4$(CH)$_2$N(CH$_2$)Me]V(PMe$_3$)$_2$}[BPh$_4$] (**3**)*

Abstract. $[C_{15}H_{31}NP_2V]^+.[C_{24}H_{20}B]^-$, M_r = 657.54, orthorhombic, *Pna*2$_1$, *a* = 23.287(1), *b* = 15.1524(7), *c* = 10.2341(4) Å, *V* = 3611.1(3) Å^3, *Z* = 4, D_x = 1.209 gcm^{-3}, *F*(000) = 1400, μ = 3.91 cm^{-1}, λ(MoK$\bar{a}$) = 0.71073 Å, T = 100(1) K, 32392 reflections measured, *GooF* = 1.037, *wR*(*F*^{2}) = 0.1449 for 8792 unique reflections and 574 parameters, 1 restraints and *R*(*F*) = 0.0582 for 7193 reflections obeying $F_o \geq 4.0\ \sigma(F_o)$ criterion of observability.
The asymmetric unit consists of two moieties: a cationic V-complex and a tetraphenylborate anion.

Experimental

X-ray diffraction: Crystal and Molecular Structure.

A crystal with the dimensions of 0.14 x 0.12 x 0.09 mm was mounted on top of a glass fiber, by using inert-atmosphere handling techniques, and aligned on a *Bruker*[1] *SMART APEX CCD* diffractometer (Platform with full three-circle goniometer). The diffractometer was equipped with a 4K *CCD* detector set 60.0 mm from the crystal. The crystal was cooled to 100(1) K using the *Bruker KRYOFLEX* low-temperature device. Intensity measurements were performed using graphite monochromated Mo-K$\bar{a}$ radiation from a sealed ceramic diffraction tube (*SIEMENS*). Generator settings were 50 KV/ 40 mA. *SMART* was used for preliminary determination of the unit cell constants and data collection control. The intensities of reflections of a hemisphere were collected by a combination of 3 sets of exposures (frames). Each set had a different ϕ angle for the crystal and each exposure covered a range of 0.3° in ω. A total of 1800 frames were collected with an exposure time of 10.0 seconds per frame. The overall data collection time was 8.0 h. Data integration and global cell refinement was performed with the program *SAINT*. The final unit cell was obtained from the *xyz* centroids of 7898 reflections after integration. Intensity data were corrected for Lorentz and polarization effects, scale variation, for decay and absorption: a multi-scan absorption correction was applied, based on the intensities of symmetry-related reflections measured at different angular settings (*SADABS*)[2], and reduced to F_o^2. The program suite *SHELXTL* was used for space group determination (*XPREP*).[1]

The unit cell[3] was identified as orthorhombic; space group *Pna*2$_1$, was derived from the systematic extinctions. The $|E|$ distribution statistics showed unambiguous a non-centrosymmetric space group.[4] Reduced cell calculations did not indicate any higher metric lattice symmetry[5] and examination of the final atomic coordinates of the structure did not yield obvious extra crystallographic or metric symmetry elements.[6,7]

The structure was solved by Patterson methods and extension of the model was accomplished by direct methods applied to difference structure factors using the program *DIRDIF*.[8] The positional and anisotropic displacement parameters for the non-hydrogen atoms were refined. A subsequent difference Fourier synthesis resulted in the location of most hydrogen atoms; the remaining hydrogen atoms were generated by geometrical considerations. The hydrogen atom coordinates and isotropic displacement parameters were refined. The hydrogen atoms connected to C17, C18 and C19 did not refine well, so ultimately these were constrained to idealized geometries and allowed to ride on their carrier atoms with an isotropic displacement parameter related to the equivalent displacement parameter of their carrier atoms.

Final refinement on F^2 carried out by full-matrix least-squares techniques converged at $wR(F^2)$ = 0.1449 for 8792 reflections and $R(F)$ = 0.0582 for 7193 reflections with $F_o \geq 4.0\ \sigma(F_o)$ and 574 parameters and 1 restraints. The final difference Fourier map was essentially featureless: no significant peaks (1.06(8) e/Å^3 in the neighborhood of C17) having chemical meaning above the general background were observed.

The polarity of the structure of the crystal actually chosen was determined by Flack's[9,10,11,12] **x**-refinement (**x** = 0.02(3)).

The positional and anisotropic displacement parameters for the non-hydrogen atoms and isotropic displacement parameters for hydrogen atoms were refined on F^2 with full-matrix least-squares procedures minimizing the function $Q = \sum_h [w(|(F_o^2) - k(F_c^2)|)^2]$, where $w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$, $P = [\max(F_o^2,0) + 2F_c^2] / 3$, F_0 and F_c are the observed and calculated structure factor amplitudes, respectively; ultimately the suggested a (=0.0759) and b (= 1.66) were used in the final refinement.

Crystal data and numerical details on data collection and refinement are given in Table 1. Final fractional atomic coordinates, equivalent displacement parameters and anisotropic displacement parameters for the non-hydrogen atoms are given in Table 2. Molecular geometry data are collected in Table 3. Neutral atom scattering factors and anomalous dispersion corrections were taken from *International Tables for Crystallography.*[14]

All refinement calculations and graphics were performed on a Pentium-III / Debian-Linux computer at the University of Groningen with the program packages *SHELXL*[15] (least-square refinements), a locally modified version of the program *PLUTO*[16] (preparation of illustrations) and *PLATON*[17] package (checking the final results for missed symmetry with the *MISSYM* option, solvent accessible voids with the *SOLV* option, calculation of geometric data and the *ORTEP*[16] illustrations).

Each asymmetric unit contains one formula unit, consisting of two moieties: a cationic V-complex and tetraphenylborate anion, with no atom setting at special position. The

orthorhombic unit cell contains eight discrete units, four cations and four anions moieties separated by normal van der Waals distances[18].

No classic hydrogen bonds, no missed symmetry (*MISSYM*), but potential solvent-accessible area (voids of 46.2 $Å^3$ / unit cell) were detected by procedures implemented in *PLATON.*[19,20]

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Table 1.

a. Crystal data and details of the structure determination.

Moiety_Formula	$[C_{15}H_{31}NP_2V]^+.[C_{24}H_{20}B]^-$
Formula_Weight, g.mol^{-1}	657.54
Crystal system	orthorhombic
Space group, no.[21]	*Pna*2$_1$, 33
a, Å	23.287(1)
b, Å	15.1524(7)
c, Å	10.2341(4)
V, Å^3	3611.1(3)
T range unit cell: min.-max., deg; reflections	2.21 - 24.56 ; 7898
Formula_*Z*	4
SpaceGroup_*Z*	4
Z' (= Formula_Z / SpaceGroup_*Z)*	1
ρ_{calc}, g.cm^{-3}	1.209
F(000), electrons	1400
μ(Mo K$\bar{a}$), cm^{-1}	3.91
Color, habit	red, block
Approx. crystal dimension, mm	0.14 x 0.12 x 0.09

b. Data collection.

λ(Mo K$\bar{\alpha}$), Å	0.71073
Monochromator	Graphite
Measurement device type	*CCD* area-detector diffractometer
Detector Area resolution (pixels / mm)	4096 x 4096 / 62 x 62 (binned 512)
Temperature, K	100(1)
Measurement method	φ- and ω-scans
θ range; min. max., deg	2.21, 28.28
Index ranges	h: -31→31; k: -19→19; l: -13→12
Min.- Max. absorption transmission factor	0.8452 – 0.9652
X-ray exposure time, h	8.0
Total data	32392
Unique data	8792
Data with criterion: ($F_o \geq 4.0\ \sigma\ (F_o)$)	7193
$R_{int} = \sum [\lvert F_o^2 - F_o^2\ (\text{mean})\rvert] / \sum [F_o^2]$	0.0629
$R_{sig} = \sum \sigma(F_o^2) / \sum [F_o^2]$	0.0749

c. Refinement.

Number of reflections	8792
Number of refined parameters	574
Number of restraints	1
Final agreement factors:	
$wR(F^2) = [\sum [w(F_o^2 - F_c^2)^2] / \sum [w(F_o^2)^2]]^{1/2}$	0.1449
Weighting scheme: *a, b*	0.0759, 1.66
$w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$	
And $P = [\max(F_o^2,0) + 2F_c^2] / 3$	
$R(F) = \sum (\|\|F_o\| - \|F_c\|\|) / \sum \|F_o\|$	0.0582
For $F_o > 4.0\ \sigma(F_o)$	
Absolute-Structure parameter Flack's **x**	0.02(3)
GooF = $S = [\sum [w(F_o^2 - F_c^2)^2] / (n-p)]^{1/2}$	1.037
n = number of reflections	
p = number of parameters refined	
Residual electron density in final	
Difference Fourier map, e/Å^3	-0.58, 1.06(8)
Max. (shift/s) final cycle	<0.001
Average (shift/σ) final cycle	0.000

Table 2. Final fractional atomic coordinates and equivalent isotropic displacement parameters with s.u.'s in parentheses.
Atoms of the Asymmetric Unit.

Non-Hydrogen parameters

Residue: 1.

Atom	*x*	*y*	*z*	U_{eq} (Å^2)*
V1	0.08493(2)	0.12905(4)	0.49570(6)	0.02070(17)
P11	0.13691(4)	0.08934(6)	0.70289(9)	0.0226(3)
P12	0.04734(4)	0.27116(6)	0.57782(10)	0.0259(3)
N1	0.15560(12)	0.1383(2)	0.3735(3)	0.0221(9)
C11	0.06325(16)	-0.0129(2)	0.4425(4)	0.0223(10)
C12	0.02229(16)	0.0142(3)	0.5345(4)	0.0260(11)
C13	-0.00974(15)	0.0840(2)	0.4810(4)	0.0238(10)
C14	0.01183(14)	0.1012(2)	0.3533(3)	0.0205(10)
C15	0.05749(14)	0.0414(2)	0.3299(3)	0.0203(10)
C16	0.09957(17)	0.0456(3)	0.2188(4)	0.0239(11)
C17	0.15777(18)	0.0718(3)	0.2666(4)	0.0377(14)
C18	0.12340(18)	0.2145(3)	0.3568(4)	0.0353(12)
C19	0.21424(18)	0.1480(3)	0.4292(5)	0.0447(16)
C110	0.1515(2)	-0.0285(3)	0.7104(5)	0.0387(14)
C111	0.20593(18)	0.1359(3)	0.7541(4)	0.0333(12)
C112	0.0942(2)	0.1046(3)	0.8511(4)	0.0347(14)
C113	0.1003(2)	0.3444(3)	0.6526(5)	0.0357(16)
C114	-0.0103(2)	0.2704(3)	0.6994(5)	0.0357(14)
C115	0.0155(2)	0.3385(3)	0.4478(5)	0.0377(14)

Residue: 2.

C21	0.35395(14)	0.1542(2)	0.2534(3)	0.0195(9)
C22	0.35643(15)	0.0769(3)	0.3316(4)	0.0256(11)
C23	0.35474(16)	0.0803(3)	0.4659(4)	0.0281(11)
C24	0.35119(16)	0.1598(3)	0.5323(4)	0.0330(13)
C25	0.35124(16)	0.2374(3)	0.4588(4)	0.0284(11)
C26	0.35214(15)	0.2340(3)	0.3238(4)	0.0227(10)
C27	0.42930(14)	0.1620(2)	0.0680(3)	0.0196(9)
C28	0.45696(15)	0.2441(2)	0.0684(4)	0.0227(10)
C29	0.51595(16)	0.2525(3)	0.0574(4)	0.0276(11)
C210	0.55041(17)	0.1791(3)	0.0455(4)	0.0361(13)
C211	0.52546(18)	0.0974(3)	0.0459(5)	0.0457(16)
C212	0.46595(18)	0.0897(3)	0.0581(5)	0.0370(14)
C213	0.33382(13)	0.0631(2)	0.0241(3)	0.0185(9)
C214	0.35248(17)	0.0340(3)	-0.0982(4)	0.0265(11)
C215	0.32723(19)	-0.0367(3)	-0.1641(4)	0.0313(12)
C216	0.28102(19)	-0.0808(3)	-0.1087(4)	0.0337(12)
C217	0.26095(17)	-0.0532(3)	0.0101(5)	0.0309(11)
C218	0.28637(14)	0.0176(2)	0.0772(4)	0.0238(10)
C219	0.32406(14)	0.2333(2)	0.0219(3)	0.0186(9)
C220	0.27161(15)	0.2647(2)	0.0714(4)	0.0261(10)
C221	0.23793(15)	0.3266(2)	0.0037(5)	0.0331(11)
C222	0.25634(17)	0.3590(3)	-0.1156(4)	0.0294(11)

C223	0.30727(17)	0.3290(3)	-0.1672(4)	0.0299(11)
C224	0.34038(15)	0.2671(2)	-0.0991(4)	0.0236(10)
B2	0.36007(16)	0.1529(3)	0.0933(4)	0.0191(11)

Hydrogen parameters:

Residue: 1.

H11	0.08887(-)	-0.05704(-)	0.43961(-)	0.0246(18)
H12	0.01380(-)	-0.01397(-)	0.62000(-)	0.045(2)
H13	-0.04220(-)	0.11112(-)	0.50953(-)	0.0353(19)
H14	-0.00371(-)	0.14216(-)	0.29055(-)	0.0183(16)
H16	0.08366(-)	0.07781(-)	0.16666(-)	0.057(2)
H16'	0.10271(-)	-0.00187(-)	0.16913(-)	0.045(2)
H17	0.17813(-)	0.01862(-)	0.29837(-)	0.04552(-)
H17'	0.18008(-)	0.09632(-)	0.19265(-)	0.04552(-)
H18	0.10331(-)	0.22136(-)	0.27215(-)	0.04244(-)
H18'	0.14018(-)	0.27003(-)	0.39064(-)	0.04244(-)
H19	0.24104(-)	0.16509(-)	0.35986(-)	0.06708(-)
H19'	0.22645(-)	0.09173(-)	0.46735(-)	0.06708(-)
H19"	0.21386(-)	0.19359(-)	0.49714(-)	0.06708(-)
H110	0.17139(-)	-0.04714(-)	0.78200(-)	0.046(2)
H110'	0.17781(-)	-0.04532(-)	0.64138(-)	0.052(2)
H110"	0.11599(-)	-0.05641(-)	0.69585(-)	0.0308(19)
H111	0.20046(-)	0.21279(-)	0.74893(-)	0.0242(17)
H111'	0.24275(-)	0.14350(-)	0.70546(-)	0.074(3)
H111"	0.21693(-)	0.10411(-)	0.84004(-)	0.057(2)
H112	0.04404(-)	0.06831(-)	0.84016(-)	0.240(6)
H112'	0.09424(-)	0.15978(-)	0.86362(-)	0.051(2)
H112"	0.11750(-)	0.08801(-)	0.93064(-)	0.0306(19)
H113	0.12721(-)	0.36029(-)	0.58929(-)	0.0142(17)
H113'	0.11782(-)	0.32704(-)	0.72747(-)	0.0250(19)
H113"	0.08498(-)	0.38659(-)	0.67153(-)	0.080(3)
H114	-0.00108(-)	0.23728(-)	0.76970(-)	0.045(2)
H114'	-0.04237(-)	0.25417(-)	0.65823(-)	0.052(2)
H114"	-0.01823(-)	0.32834(-)	0.72451(-)	0.042(2)
H115	0.00623(-)	0.39167(-)	0.46310(-)	0.041(2)
H115'	-0.01488(-)	0.30113(-)	0.40101(-)	0.041(2)
H115"	0.05573(-)	0.35206(-)	0.39630(-)	0.067(3)

Residue: 2.

H22	0.35909(-)	0.01452(-)	0.28513(-)	0.0250(16)
H23	0.35553(-)	0.03498(-)	0.50872(-)	0.0235(16)
H24	0.35018(-)	0.17198(-)	0.62826(-)	0.0243(17)
H25	0.34889(-)	0.29687(-)	0.49195(-)	0.0403(18)
H26	0.35224(-)	0.28288(-)	0.28142(-)	0.0036(14)
H28	0.43112(-)	0.30233(-)	0.06409(-)	0.0317(18)
H29	0.53123(-)	0.30759(-)	0.05128(-)	0.0203(16)
H210	0.58856(-)	0.18328(-)	0.03610(-)	0.034(2)
H211	0.55084(-)	0.04961(-)	0.03467(-)	0.058(2)
H212	0.45347(-)	0.04842(-)	0.03167(-)	0.078(3)
H214	0.38277(-)	0.06843(-)	-0.13367(-)	0.0138(16)
H215	0.34354(-)	-0.05289(-)	-0.24077(-)	0.0138(15)
H216	0.26255(-)	-0.11669(-)	-0.14084(-)	0.037(2)
H217	0.23899(-)	-0.07605(-)	0.04207(-)	0.0254(19)

H218	0.27338(-)	0.03418(-)	0.16640(-)	0.0114(15)
H220	0.25946(-)	0.24222(-)	0.14959(-)	0.0233(17)
H221	0.19800(-)	0.33638(-)	0.05472(-)	0.047(2)
H222	0.23399(-)	0.40990(-)	-0.17037(-)	0.0387(19)
H223	0.31974(-)	0.34691(-)	-0.24512(-)	0.0103(16)
H224	0.37471(-)	0.25491(-)	-0.14091(-)	0.0148(17)

*) $U_{eq} = 1/3 \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a_i}.\mathbf{a_j}$ [22]

Anisotropic (displacement) parameters (Å^2)

Residue: 1.

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
V1	0.0217(3)	0.0264(3)	0.0140(3)	-0.0032(3)	0.0024(3)	-0.0129(2)
P11	0.0214(4)	0.0252(4)	0.0212(5)	-0.0042(4)	-0.0010(4)	-0.0028(4)
P12	0.0376(5)	0.0202(4)	0.0199(5)	-0.0001(4)	-0.0038(4)	-0.0068(4)
N1	0.0148(13)	0.0310(16)	0.0205(16)	-0.0063(12)	0.0050(11)	-0.0056(11)
C11	0.0206(16)	0.0226(18)	0.0237(18)	0.0033(14)	-0.0039(14)	-0.0075(14)
C12	0.0259(18)	0.0334(19)	0.0188(19)	0.0050(14)	-0.0031(13)	-0.0159(15)
C13	0.0222(16)	0.0270(18)	0.0222(19)	-0.0032(15)	0.0008(14)	-0.0110(13)
C14	0.0227(17)	0.0207(17)	0.0182(18)	-0.0039(13)	-0.0038(13)	-0.0043(13)
C15	0.0205(16)	0.0255(17)	0.0150(17)	-0.0045(13)	-0.0024(13)	-0.0068(13)
C16	0.0302(19)	0.0259(19)	0.0157(19)	-0.0034(15)	0.0013(15)	-0.0008(15)
C17	0.033(2)	0.047(3)	0.033(2)	-0.0059(19)	0.0035(18)	0.0069(18)
C18	0.036(2)	0.033(2)	0.037(2)	0.0066(18)	0.0011(18)	-0.0008(17)
C19	0.027(2)	0.064(3)	0.043(3)	-0.001(2)	0.0008(19)	-0.004(2)
C110	0.040(2)	0.034(2)	0.042(3)	-0.002(2)	-0.018(2)	0.0037(18)
C111	0.030(2)	0.040(2)	0.030(2)	0.0013(17)	-0.0059(17)	-0.0069(16)
C112	0.042(2)	0.038(3)	0.024(2)	-0.0010(18)	0.0086(18)	0.0002(19)
C113	0.046(3)	0.027(2)	0.034(3)	-0.0082(19)	-0.002(2)	-0.0120(19)
C114	0.036(2)	0.040(3)	0.031(2)	0.001(2)	-0.0023(19)	0.0015(18)
C115	0.054(3)	0.027(2)	0.032(2)	0.0047(17)	-0.007(2)	0.003(2)

Residue: 2.

C21	0.0150(15)	0.0218(16)	0.0216(18)	-0.0012(13)	0.0033(12)	-0.0010(12)
C22	0.0238(18)	0.028(2)	0.025(2)	0.0046(15)	-0.0006(14)	-0.0013(14)
C23	0.0284(19)	0.036(2)	0.020(2)	0.0101(15)	-0.0035(14)	-0.0029(15)
C24	0.0241(18)	0.058(3)	0.017(2)	-0.0038(16)	0.0003(14)	-0.0071(17)
C25	0.0241(17)	0.034(2)	0.027(2)	-0.0065(15)	0.0010(14)	-0.0042(15)
C26	0.0213(16)	0.0219(18)	0.025(2)	0.0022(14)	0.0060(14)	-0.0011(13)
C27	0.0173(15)	0.0228(16)	0.0186(18)	0.0023(14)	0.0009(13)	-0.0017(12)
C28	0.0262(17)	0.0239(16)	0.0179(18)	-0.0029(14)	0.0025(14)	-0.0024(14)
C29	0.0276(18)	0.036(2)	0.0193(19)	-0.0058(15)	0.0053(14)	-0.0132(15)
C210	0.0192(18)	0.056(3)	0.033(2)	-0.0065(19)	0.0040(15)	-0.0044(17)
C211	0.027(2)	0.043(2)	0.067(4)	0.000(2)	0.005(2)	0.0100(18)
C212	0.028(2)	0.022(2)	0.061(3)	0.005(2)	0.0008(19)	0.0029(16)
C213	0.0190(15)	0.0160(14)	0.0204(19)	0.0036(12)	-0.0001(12)	0.0027(12)
C214	0.035(2)	0.0238(18)	0.0206(19)	0.0036(14)	-0.0002(15)	-0.0031(15)
C215	0.044(2)	0.029(2)	0.021(2)	-0.0010(16)	-0.0048(18)	-0.0017(17)
C216	0.039(2)	0.030(2)	0.032(2)	-0.0004(17)	-0.0171(18)	-0.0064(17)

C217	0.0226(17)	0.034(2)	0.036(2)	0.0070(19)	-0.0058(18)	-0.0103(15)
C218	0.0190(16)	0.0255(17)	0.027(2)	0.0008(15)	-0.0013(15)	-0.0009(13)
C219	0.0218(15)	0.0150(15)	0.0191(19)	-0.0033(12)	-0.0012(12)	-0.0005(11)
C220	0.0253(17)	0.0231(17)	0.030(2)	-0.0023(16)	0.0(16)	0.0015(14)
C221	0.0242(17)	0.0292(19)	0.046(2)	-0.007(2)	-0.007(2)	0.0065(14)
C222	0.0319(19)	0.0222(18)	0.034(2)	-0.0011(16)	-0.0154(17)	0.0032(14)
C223	0.038(2)	0.0257(19)	0.026(2)	0.0059(15)	-0.0089(17)	-0.0055(16)
C224	0.0221(17)	0.0239(18)	0.0248(19)	-0.0005(14)	-0.0005(15)	-0.0021(14)
B2	0.0177(17)	0.0206(18)	0.019(2)	0.0031(14)	0.0052(15)	-0.0005(14)

Thermal vibration amplitudes (Å^2)

$$F(\mathbf{h}) = F_o(\mathbf{h}) \exp\left(-2\pi^2 \sum_{i=1}^{3}\sum_{j=1}^{3} h_i h_j a_i^* a_j^* U_{ij}\right)$$

or

$$F(\mathbf{h}) = F_o(\mathbf{h}) \exp\left(-8\pi^2 U_{iso} (\sin(\theta)/\lambda)^2\right)$$

Table 3. Data on the geometry.

Standard deviations in the last decimal place are given in parentheses.

Residue: 1.

Interatomic Distances (Å)

V1	-P112.5146(11)	P12	-C1141.830(5)
V1	-P122.4717(11)	P12	-C1151.833(5)
V1	-N1 2.072(3)	N1	-C171.488(5)
V1	-C112.275(3)	N1	-C181.387(5)
V1	-C122.305(4)	N1	-C191.487(5)
V1	-C132.313(3)	C11	-C121.402(6)
V1	-C142.280(3)	C11	-C151.422(5)
V1	-C152.248(3)	C12	-C131.405(5)
V1	-C182.121(4)	C13	-C141.424(5)
P11	-C1101.819(5)	C14	-C151.417(4)
P11	-C1111.832(4)	C15	-C161.502(5)
P11	-C1121.829(4)	C16	-C171.495(6)
P12	-C1131.827(5)		

Hydrogen parameters:

C11	-H110.8967(-)	C111	-H111'0.9981(-)
C12	-H120.9934(-)	C111	-H111"1.0350(-)
C13	-H130.9086(-)	C111	-H1111.1732(-)
C14	-H140.9636(-)	C112	-H112'0.8459(-)
C16	-H160.8125(-)	C112	-H112"1.0101(-)
C16	-H16'0.8838(-)	C112	-H1121.2959(-)
C17	-H170.9899(-)	C113	-H113'0.9071(-)
C17	-H17'0.9903(-)	C113	-H113"0.7573(-)
C18	-H18 0.99(-)	C113	-H1130.933(-)
C18	-H18'0.9903(-)	C114	-H114'0.892(-)
C19	-H190.9799(-)	C114	-H114"0.9332(-)
C19	-H19'0.9799(-)	C114	-H1140.9031(-)
C19	-H19"0.9802(-)	C115	-H115'1.0249(-)
C110	-H110'0.9692(-)	C115	-H115"1.0944(-)
C110	-H110"0.9406(-)	C115	-H1150.8486(-)
C110	-H1100.9117(-)		

Bond Angles (deg.)

P11	-V1	-P12	95.29(4)	V1	-P11	-C112	114.04(15)
P11	-V1	-N1	98.21(9)	C110	-P11	-C111	101.7(2)
P11	-V1	-C11	94.73(10)	C110	-P11	-C112	101.0(2)
P11	-V1	-C12	88.78(10)	C111	-P11	-C112	101.0(2)
P11	-V1	-C13	116.3(1)	V1	-P12	-C113	115.63(15)
P11	-V1	-C14	148.65(8)	V1	-P12	-C114	119.04(15)
P11	-V1	-C15	129.20(9)	V1	-P12	-C115	112.41(16)
P11	-V1	-C18	120.52(12)	C113	-P12	-C114	102.4(2)
P12	-V1	-N1	115.32(9)	C113	-P12	-C115	103.8(2)
P12	-V1	-C11	145.72(10)	C114	-P12	-C115	101.6(2)
P12	-V1	-C12	112.03(11)	V1	-N1	-C17	115.2(2)
P12	-V1	-C13	86.66(8)	V1	-N1	-C18	72.6(2)
P12	-V1	-C14	96.56(8)	V1	-N1	-C19	120.3(3)

P12	-V1	-C15	132.13(9)	C17	-N1	-C18	119.4(3)
P12	-V1	-C18	81.12(12)	C17	-N1	-C19	108.5(3)
N1	-V1	-C11	95.50(13)	C18	-N1	-C19	117.5(3)
N1	-V1	-C12	131.13(14)	V1	-C11	-C12	73.3(2)
N1	-V1	-C13	137.55(13)	V1	-C11	-C15	70.61(17)
N1	-V1	-C14	102.69(12)	C12	-C11	-C15	108.1(3)
N1	-V1	-C15	79.05(12)	V1	-C12	-C11	71.0(2)
N1	-V1	-C18	38.62(14)	V1	-C12	-C13	72.6(2)
C11	-V1	-C12	35.63(14)	C11	-C12	-C13	108.7(4)
C11	-V1	-C13	59.60(12)	V1	-C13	-C12	72.0(2)
C11	-V1	-C14	60.43(12)	V1	-C13	-C14	70.70(19)
C11	-V1	-C15	36.65(13)	C12	-C13	-C14	107.9(3)
C11	-V1	-C18	120.69(16)	V1	-C14	-C13	73.18(19)
C12	-V1	-C13	35.43(13)	V1	-C14	-C15	70.50(18)
C12	-V1	-C14	59.87(13)	C13	-C14	-C15	107.6(3)
C12	-V1	-C15	60.27(13)	V1	-C15	-C11	72.74(19)
C12	-V1	-C18	147.51(16)	V1	-C15	-C14	73.02(18)
C13	-V1	-C14	36.12(13)	V1	-C15	-C16	111.2(2)
C13	-V1	-C15	60.36(13)	C11	-C15	-C14	107.7(3)
C13	-V1	-C18	122.63(15)	C11	-C15	-C16	125.2(3)
C14	-V1	-C15	36.48(11)	C14	-C15	-C16	126.2(3)
C14	-V1	-C18	90.00(14)	C15	-C16	-C17	110.8(3)
C15	-V1	-C18	88.56(14)	N1	-C17	-C16	112.9(3)
V1	-P11	-C110	111.12(16)	V1	-C18	-N1	68.8(2)
V1	-P11	-C111	124.85(14)				

Residue: 2.

Interatomic Distances (Å)

C21	-C221.420(5)	C213	-C2141.396(5)
C21	-C261.408(5)	C213	-C2181.411(5)
C21	-B2 1.645(5)	C213	-B2 1.651(5)
C22	-C231.376(6)	C214	-C2151.396(6)
C23	-C241.386(6)	C215	-C2161.388(6)
C24	-C251.396(6)	C216	-C2171.368(6)
C25	-C261.383(6)	C217	-C2181.405(6)
C27	-C281.401(4)	C219	-C2201.405(5)
C27	-B2 1.639(5)	C219	-C2241.393(5)
C28	-C291.384(5)	C219	-B2 1.650(5)
C210	-C291.377(6)	C220	-C2211.405(5)
C210	-C2111.368(6)	C221	-C2221.384(6)
C211	-C2121.396(6)	C222	-C2231.376(6)
C212	-C271.392(5)	C223	-C2241.400(5)

Hydrogen parameters:

C22	-H221.0599(-)	C214	-H2140.9495(-)
C23	-H230.8148(-)	C215	-H2150.9056(-)
C24	-H240.9995(-)	C216	-H2160.7674(-)
C25	-H250.9644(-)	C217	-H2170.6989(-)
C26	-H260.8583(-)	C218	-H2180.9940(-)
C28	-H281.0689(-)	C220	-H2200.9146(-)
C29	-H290.9096(-)	C221	-H2211.0767(-)
C210	-H2100.8958(-)	C222	-H2221.0862(-)

C211	-H211	0.9417(-)		C223	-H223	0.891(-)	
C212	-H212	0.7409(-)		C224	-H224	0.9254(-)	

Bond Angles (deg.)

C22	-C21	-C26	114.9(3)	C213	-C214	-C215	123.0(4)
C22	-C21	-B2	123.2(3)	C214	-C215	-C216	119.9(4)
C26	-C21	-B2	121.5(3)	C215	-C216	-C217	118.7(4)
C21	-C22	-C23	122.1(4)	C216	-C217	-C218	121.6(4)
C22	-C23	-C24	121.6(4)	C213	-C218	-C217	121.0(4)
C23	-C24	-C25	117.9(4)	C220	-C219	-C224	115.7(3)
C24	-C25	-C26	120.5(4)	C220	-C219	-B2	122.2(3)
C21	-C26	-C25	122.9(4)	C224	-C219	-B2	121.8(3)
C28	-C27	-C212	114.7(3)	C219	-C220	-C221	122.2(4)
C28	-C27	-B2	121.8(3)	C220	-C221	-C222	119.9(4)
C212	-C27	-B2	123.3(3)	C221	-C222	-C223	119.2(4)
C27	-C28	-C29	122.5(3)	C222	-C223	-C224	120.3(4)
C28	-C29	-C210	120.8(4)	C219	-C224	-C223	122.6(3)
C29	-C210	-C211	118.9(4)	C21	-B2	-C27	104.0(3)
C210	-C211	-C212	119.8(4)	C21	-B2	-C213	113.9(3)
C27	-C212	-C211	123.3(4)	C21	-B2	-C219	112.9(3)
C214	-C213	-C218	115.8(3)	C27	-B2	-C213	111.5(3)
C214	-C213	-B2	122.0(3)	C27	-B2	-C219	111.6(3)
C218	-C213	-B2	121.8(3)	C213	-B2	-C219	103.3(3)

Hydrogen parameters:

C21	-C22	-H22	119.02(-)	C213	-C214	-H214	113.6(-)
C23	-C22	-H22	118.9(-)	C215	-C214	-H214	123.36(-)
C22	-C23	-H23	120.33(-)	C214	-C215	-H215	116.74(-)
C24	-C23	-H23	118.05(-)	C216	-C215	-H215	123.28(-)
C23	-C24	-H24	130.08(-)	C215	-C216	-H216	126.92(-)
C25	-C24	-H24	111.96(-)	C217	-C216	-H216	113.96(-)
C24	-C25	-H25	126.69(-)	C216	-C217	-H217	120.97(-)
C26	-C25	-H25	112.78(-)	C218	-C217	-H217	117.25(-)
C21	-C26	-H26	118.84(-)	C213	-C218	-H218	117.94(-)
C25	-C26	-H26	118.22(-)	C217	-C218	-H218	120.92(-)
C27	-C28	-H28	118.3(-)	C219	-C220	-H220	117.27(-)
C29	-C28	-H28	118.65(-)	C221	-C220	-H220	120.48(-)
C28	-C29	-H29	118.57(-)	C220	-C221	-H221	109.56(-)
C210	-C29	-H29	120.48(-)	C222	-C221	-H221	130.28(-)
C29	-C210	-H210	122.03(-)	C221	-C222	-H222	123.96(-)
C211	-C210	-H210	119.06(-)	C223	-C222	-H222	116.72(-)
C210	-C211	-H211	115.41(-)	C222	-C223	-H223	121.6(-)
C212	-C211	-H211	124.72(-)	C224	-C223	-H223	118.04(-)
C27	-C212	-H212	116.77(-)	C219	-C224	-H224	124.99(-)
C211	-C212	-H212	115.29(-)	C223	-C224	-H224	112.29(-)

*Part IV: Structure of {[h^5,h^1-$C_5H_4(CH)_2NMe_2$]V(h^3-C_3H_5)(PMe_3)}[BPh_4] (**6**)*

Abstract. $[C_{15}H_{28}NPV]^+.[C_{24}H_{20}B]^-$, M_r = 623.54, orthorhombic, *Pna*2$_1$, *a* = 23.038(2), *b* = 15.084(1), *c* = 9.8631(8) Å, *V* = 3427.5(5) Å^3, *Z* = 4, D_x = 1.208 gcm^{-3}, *F*(000) = 1328, μ = 3.63 cm^{-1}, λ(MoK$\bar{a}$) = 0.71073 Å, T = 100(1) K, 19082 reflections measured, *GooF* = 1.217, *wR*(F^2) = 0.2127 for 5951 unique reflections and 463 parameters, 1 restraints and *R*(*F*) = 0.0855 for 4043 reflections obeying $F_o \geq 4.0\ \sigma(F_o)$ criterion of observability.

The asymmetric unit consists of two moieties: a cationic V-complex and tetraphenylborate anion.

Comment

The scattering power of the crystals investigated was very weak: nearby an half of the unique (till T = 26.37 °) merged reflections obey the $F_o \geq 4.0\ \sigma(F_o)$ criterion of observability. This implies that the mean *s.u*. is large compared to the mean magnitude of the (even more than double of the squared) structure factor. The weak scattering power might be the result of a disorder problem.

Experimental

X-ray diffraction: Crystal and Molecular Structure.

A crystal with the dimensions of 0.40 x 0.38 x 0.09 mm was mounted on top of a glass fiber, by using inert-atmosphere handling techniques, and aligned on a *Bruker*[1] *SMART APEX CCD* diffractometer (Platform with full three-circle goniometer). The diffractometer was equipped with a 4K *CCD* detector set 60.0 mm from the crystal. The crystal was cooled to 100(1) K using the *Bruker KRYOFLEX* low-temperature device. Intensity measurements were performed using graphite monochromated Mo-K$\bar{a}$ radiation from a sealed ceramic diffraction tube (*SIEMENS*). Generator settings were 50 KV/ 40 mA. *SMART*[1] was used for preliminary determination of the unit cell constants and data collection control. The intensities of reflections of a hemisphere were collected by a combination of 3 sets of exposures (frames). Each set had a different ϕ angle for the crystal and each exposure covered a range of 0.3° in ω. A total of 1720 frames were collected with an exposure time of 10.0 seconds per frame. The overall data collection time was 7.5 h. Data integration and global cell refinement was performed with the program *SAINT*.[1] The final unit cell was obtained from the *xyz* centroids of 4521 reflections after integration. Intensity data were corrected for Lorentz and polarization effects, scale variation, for decay and absorption: a multi-scan absorption correction was applied, based on the intensities of symmetry-related reflections measured at different angular

settings (*SADABS*)[2], and reduced to F_o^2. The program suite *SHELXTL* was used for space group determination (*XPREP*).[1]

The unit cell[3] was identified as orthorhombic; space group $Pna2_1$, was derived from the systematic extinctions (There was observed a weak violation to the *a*-glide). The $|E|$ distribution statistics were indicative of a non-centrosymmetric space group.[4] Reduced cell calculations did not indicate any higher metric lattice symmetry[5] and examination of the final atomic coordinates of the structure did not yield obvious extra crystallographic or metric symmetry elements.[6,7]

The structure was solved by Patterson methods and extension of the model was accomplished by direct methods applied to difference structure factors using the program *DIRDIF*.[8] The positional and anisotropic displacement parameters for the non-hydrogen atoms were refined. Refinement was complicated by a disorder problem: a residual peak of ~3.7 e/Å^3 at a distance of ~1 Å from V1a was observed; this peak was refined as partly occupied by a V atom (finally refined s.o.f. = 0.22(1)). As a consequence the ligands should also be shifted; this could not be modeled, but some atoms showed unrealistic displacement parameters when allowed to vary anisotropically, suggesting dynamic disorder (dynamic means that the smeared electron density is due to fluctuations of the atomic positions within each unit cell) as a consequence of the disorder. This is in line with the weak scattering power of the crystals investigated.

Hydrogen atoms were constrained to idealized geometries and allowed to ride on their carrier atoms with an isotropic displacement parameter related to the equivalent displacement parameter of their carrier atoms.

Final refinement on F^2 carried out by full-matrix least-squares techniques converged at $wR(F^2)$ = 0.2127 for 5951 reflections and $R(F)$ = 0.0855 for 4043 reflections with $F_o \geq 4.0\ \sigma(F_o)$ and 463 parameters and 1 restraints. The final difference Fourier map was essentially featureless: no significant peaks (0.72(10) e/Å^3) having chemical meaning above the general background were observed.

The absolute structure of the molecule actually chosen was determined by Flack's[9,10,11,12] refinement (**x** = 0.08(6)).

The positional and anisotropic displacement parameters for the non-hydrogen atoms and isotropic displacement parameters for hydrogen atoms were refined on F^2 with full-matrix least-squares procedures minimizing the function $Q = \sum_h[w(|(F_o^2) - k(F_c^2)|)^2]$, where $w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$, $P = [\max(F_o^2,0) + 2F_c^2] / 3$, F_0 and F_c are the observed and calculated structure factor amplitudes, respectively; the *a* (=0.1) and *b* (= 0.0) were used in the final refinement.

Crystal data and numerical details on data collection and refinement are given in Table 1. Final fractional atomic coordinates, equivalent displacement parameters and anisotropic displacement parameters for the non-hydrogen atoms are given in Table 2. Molecular geometry data are collected in Table 3. Neutral atom scattering factors and anomalous dispersion corrections were taken from *International Tables for Crystallography.*[14]

All refinement calculations and graphics were performed on a Pentium-III / Debian-Linux computer at the University of Groningen with the program packages *SHELXL*[15] (least-square refinements), a locally modified version of the program *PLUTO*[16] (preparation of illustrations) and *PLATON*[17] package (checking the final results for missed symmetry with the *MISSYM* option, solvent accessible voids with the *SOLV* option, calculation of geometric data and the *ORTEP*[17] illustrations).

Each asymmetric unit contains one formula unit, consisting of two moieties: a cationic V-complex and tetraphenylborate anion, with no atom setting at special position. The orthorhombic unit cell contains eight discrete units, four cations and four anions moieties separated by normal van der Waals distances[18].

No classic hydrogen bonds, no missed symmetry (*MISSYM*), but potential solvent-accessible area (voids of 122.9 $Å^3$ / unit cell) were detected by procedures implemented in *PLATON.*[19,20]

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Table 1.

a. Crystal data and details of the structure determination.

Moiety_Formula	$[C_{15}H_{28}NPV]^{+}.[C_{24}H_{20}B]^{-}$
Formula_Weight, g.mol^{-1}	623.54
Crystal system	orthorhombic
Space group, no.[21]	*Pna*2$_1$, 33
a, Å	23.038(2)
b, Å	15.084(1)
c, Å	9.8631(8)
V, Å^3	3427.5(5)
T range unit cell: min.-max., deg; reflections	2.22 - 22.17 ; 4521
Formula_*Z*	4
SpaceGroup_*Z*	4
Z' (= Formula_Z / SpaceGroup_*Z)*	1
ρ_{calc}, g.cm^{-3}	1.208
F(000), electrons	1328
μ(Mo K$\bar{a}$), cm^{-1}	3.63
Color, habit	red, platelet
Approx. crystal dimension, mm	0.40 x 0.38 x 0.09

b. Data collection.

λ(Mo K$\bar{\alpha}$), Å	0.71073
Monochromator	Graphite
Measurement device type	*CCD* area-detector diffractometer
Detector Area resolution (pixels / mm)	4096 x 4096 / 62 x 62 (binned 512)
Temperature, K	100(1)
Measurement method	φ- and ω-scans
θ range; min. max., deg	2.22, 25.02
Index ranges	h: -24→27; k: -16→17; l: -11→11
Min.- Max. absorption transmission factor	0.7800 – 0.9682
X-ray exposure time, h	7.5
Total data	19082
Unique data	5951
Data with criterion: ($F_o \geq 4.0\ \sigma\ (F_o)$)	4043
$R_{int} = \sum [\lvert F_o^2 - F_o^2\ (\text{mean})\rvert] / \sum [F_o^2]$	0.0856
$R_{sig} = \sum \sigma(F_o^2) / \sum [F_o^2]$	0.1044

c. Refinement.

Number of reflections	5951
Number of refined parameters	463
Number of restraints	1
Final agreement factors:	
$wR(F^2) = [\sum [w(F_o^2 - F_c^2)^2] / \sum [w(F_o^2)^2]]^{1/2}$	0.2127
Weighting scheme: *a, b*	0.1, 0.0
$w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$	
And $P = [\max(F_o^2,0) + 2F_c^2] / 3$	
$R(F) = \sum (\|\|F_o\| - \|F_c\|\|) / \sum \|F_o\|$	0.0855
For $F_o > 4.0\ \sigma(F_o)$	
Absolute-Structure parameter Flack's **x**	0.08(6)
GooF = $S = [\sum [w(F_o^2 - F_c^2)^2] / (n-p)]^{1/2}$	1.217
n = number of reflections	
p = number of parameters refined	
Residual electron density in final	
Difference Fourier map, e/Å^3	-0.52, 0.72(10)
Max. (shift/s) final cycle	0.355
Average (shift/σ) final cycle	0.193

Table 2. Final fractional atomic coordinates and equivalent isotropic displacement parameters with s.u.'s in parentheses.
Atoms of the Asymmetric Unit.

Non-Hydrogen parameters

Residue: 1.

Atom	*x*	*y*	*z*	U_{eq} (Å^2)*
V1a	0.08782(7)	0.37685(15)	0.5060(2)	0.0185(6)
V1b	0.1010(4)	0.3507(6)	0.5839(15)	0.033(4)
P1	0.04882(9)	0.21913(14)	0.4879(3)	0.0453(7)
N1	0.1681(3)	0.3255(5)	0.6090(6)	0.045(3)
C11	0.1450(4)	0.4819(6)	0.3949(9)	0.051(3)
C12	0.0844(4)	0.4914(6)	0.3548(8)	0.046(3)
C13	0.0674(4)	0.4157(6)	0.2862(9)	0.049(3)
C14	0.1169(4)	0.3566(5)	0.2829(8)	0.047(3)
C15	0.1596(3)	0.3989(5)	0.3583(8)	0.033(3)
C16	0.2178(4)	0.3521(6)	0.3892(10)	0.054(3)
C17	0.2054(4)	0.2829(6)	0.5016(12)	0.071(3)
C18	0.1549(4)	0.2576(6)	0.7196(9)	0.055(3)
C19	0.2030(4)	0.3935(6)	0.6722(10)	0.056(3)
C110	0.0937(4)	0.1281(6)	0.4237(10)	0.063(4)
C111	-0.0066(4)	0.2153(7)	0.3667(11)	0.070(4)
C112	0.0162(4)	0.1674(8)	0.6322(10)	0.075(4)
C113	0.0717(3)	0.4722(6)	0.6901(10)	0.050(3)
C114	0.0268(4)	0.4083(8)	0.682(1)	0.066(4)
C115	-0.0067(4)	0.4051(6)	0.5651(10)	0.058(3)

[a] Indicates an s.o.f. of 0.78(1)

[b] Indicates an s.o.f. of 0.22(1)

Residue: 2.

C21	0.1721(3)	0.7399(4)	0.5715(7)	0.021(2)
C22	0.2270(3)	0.7128(4)	0.5238(8)	0.029(3)
C23	0.2632(3)	0.6531(5)	0.5968(8)	0.031(2)
C24	0.2443(3)	0.6162(4)	0.7169(8)	0.033(3)
C25	0.1893(3)	0.6403(4)	0.7648(8)	0.030(3)
C26	0.1562(3)	0.6999(4)	0.6939(8)	0.026(2)
C27	0.0649(3)	0.8120(4)	0.5191(8)	0.0267(19)
C28	0.0359(3)	0.7290(5)	0.5128(8)	0.029(2)
C29	-0.0244(3)	0.7202(5)	0.5101(9)	0.035(2)
C210	-0.0586(3)	0.7947(5)	0.5151(8)	0.039(3)
C211	-0.0315(3)	0.8784(5)	0.5246(8)	0.038(3)
C212	0.0274(3)	0.8869(5)	0.5230(8)	0.028(2)
C213	0.1631(3)	0.9088(4)	0.5731(7)	0.025(2)
C214	0.1418(3)	0.9490(4)	0.6932(8)	0.027(2)
C215	0.1681(3)	1.0168(5)	0.7609(7)	0.030(3)
C216	0.2206(3)	1.0481(5)	0.7159(8)	0.038(3)
C217	0.2454(3)	1.0138(5)	0.5964(9)	0.043(3)

C218	0.2162(3)	0.9449(5)	0.5322(7)	0.031(2)
C219	0.1423(3)	0.8218(4)	0.3349(7)	0.023(2)
C220	0.1396(3)	0.9016(5)	0.2589(7)	0.025(2)
C221	0.1399(3)	0.9024(5)	0.1173(7)	0.031(3)
C222	0.1441(3)	0.8230(6)	0.0447(8)	0.033(3)
C223	0.1463(3)	0.7441(6)	0.1170(8)	0.036(3)
C224	0.1463(3)	0.7439(5)	0.2606(8)	0.029(3)
B2	0.1357(3)	0.8204(4)	0.5049(9)	0.022(2)

Hydrogen parameters:

Residue: 1.

H16	0.24699(-)	0.39561(-)	0.42058(-)	0.06444(-)
H16'	0.23282(-)	0.32242(-)	0.30677(-)	0.06444(-)
H17	0.18506(-)	0.23101(-)	0.46255(-)	0.08456(-)
H17'	0.24231(-)	0.26227(-)	0.54201(-)	0.08456(-)
H18	0.19102(-)	0.22896(-)	0.74834(-)	0.08282(-)
H18'	0.12810(-)	0.21281(-)	0.68420(-)	0.08282(-)
H18"	0.13707(-)	0.28761(-)	0.79733(-)	0.08282(-)
H19	0.23549(-)	0.36595(-)	0.72040(-)	0.08400(-)
H19'	0.17911(-)	0.42701(-)	0.73650(-)	0.08400(-)
H19"	0.21799(-)	0.43370(-)	0.60243(-)	0.08400(-)
H110	0.06858(-)	0.08023(-)	0.39078(-)	0.09430(-)
H110'	0.11847(-)	0.10561(-)	0.49680(-)	0.09430(-)
H110"	0.11803(-)	0.14961(-)	0.34916(-)	0.09430(-)
H111	0.16895(-)	0.52491(-)	0.43760(-)	0.06080(-)
H111	-0.02022(-)	0.15414(-)	0.35680(-)	0.10442(-)
H111'	0.00833(-)	0.23662(-)	0.27956(-)	0.10442(-)
H111"	-0.03884(-)	0.25309(-)	0.39598(-)	0.10442(-)
H112	-0.01548(-)	0.20480(-)	0.66607(-)	0.11329(-)
H112'	0.04535(-)	0.15979(-)	0.70349(-)	0.11329(-)
H112"	0.00068(-)	0.10937(-)	0.60605(-)	0.11329(-)
H121	0.06048(-)	0.54130(-)	0.37281(-)	0.05456(-)
H131	0.03021(-)	0.40466(-)	0.24824(-)	0.05904(-)
H141	0.11951(-)	0.30080(-)	0.23857(-)	0.05579(-)
H1131	0.07836(-)	0.51159(-)	0.61658(-)	0.05940(-)
H1131'	0.09524(-)	0.47566(-)	0.76909(-)	0.05940(-)
H1141	0.01977(-)	0.36854(-)	0.75489(-)	0.07937(-)
H1151	0.00079(-)	0.44522(-)	0.49295(-)	0.07022(-)
H1151'	-0.03705(-)	0.36283(-)	0.55734(-)	0.07022(-)

Residue: 2.

H22	0.24033(-)	0.73528(-)	0.43939(-)	0.035(3)
H23	0.30057(-)	0.63869(-)	0.56271(-)	0.037(3)
H24	0.26791(-)	0.57553(-)	0.76565(-)	0.040(3)
H25	0.17493(-)	0.61525(-)	0.84650(-)	0.036(3)
H26	0.11953(-)	0.71537(-)	0.73100(-)	0.031(3)
H28	0.05882(-)	0.67667(-)	0.51035(-)	0.035(3)
H29	-0.04171(-)	0.66311(-)	0.50493(-)	0.042(3)
H210	-0.09965(-)	0.79000(-)	0.51213(-)	0.047(3)
H211	-0.05483(-)	0.93005(-)	0.53239(-)	0.045(3)
H212	0.04397(-)	0.94462(-)	0.52450(-)	0.034(3)

H214	0.10641(-)	0.92693(-)	0.72930(-)	0.033(3)
H215	0.15020(-)	1.04223(-)	0.83847(-)	0.036(3)
H216	0.24031(-)	1.09294(-)	0.76549(-)	0.046(3)
H217	0.28067(-)	1.03676(-)	0.56097(-)	0.051(3)
H218	0.23382(-)	0.92021(-)	0.45373(-)	0.037(3)
H220	0.13749(-)	0.95644(-)	0.30600(-)	0.029(3)
H221	0.13732(-)	0.95715(-)	0.07010(-)	0.038(3)
H222	0.14543(-)	0.82309(-)	-0.05160(-)	0.039(3)
H223	0.14789(-)	0.68936(-)	0.06939(-)	0.042(3)
H224	0.14902(-)	0.68894(-)	0.30727(-)	0.035(3)

*) $U_{eq} = 1/3 \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a_i}.\mathbf{a_j}$ ^{22}

Anisotropic (displacement) parameters (Å^2)

Residue: 1.

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
V1a	0.0172(7)	0.0166(9)	0.0216(15)	-0.0017(10)	-0.0035(9)	-0.0010(7)
V1b	0.027(4)	0.018(4)	0.053(9)	-0.004(5)	0.006(4)	-0.008(3)
P1	0.0387(11)	0.0530(13)	0.0441(13)	0.0024(12)	0.0036(11)	-0.0089(10)
N1	0.048(4)	0.059(5)	0.029(4)	0.012(3)	-0.003(3)	-0.010(4)
C11	0.070(7)	0.050(6)	0.032(5)	0.023(4)	-0.004(4)	-0.017(5)
C12	0.051(6)	0.056(6)	0.030(5)	0.015(4)	-0.006(4)	0.010(4)
C13	0.050(5)	0.050(5)	0.048(5)	0.013(5)	-0.009(4)	0.008(4)
C14	0.067(6)	0.040(5)	0.033(5)	0.002(4)	0.009(4)	-0.001(4)
C15	0.046(5)	0.018(4)	0.036(4)	0.012(3)	-0.028(4)	-0.001(3)
C16	0.042(5)	0.051(6)	0.069(6)	0.028(5)	0.004(4)	0.015(4)
C17	0.048(5)	0.071(6)	0.093(7)	-0.032(7)	-0.038(6)	0.015(5)
C18	0.057(6)	0.065(6)	0.044(6)	0.007(4)	-0.026(4)	0.009(5)
C19	0.054(5)	0.055(6)	0.059(6)	0.004(5)	-0.016(5)	-0.017(5)
C110	0.087(7)	0.053(6)	0.049(6)	-0.004(5)	-0.005(5)	-0.005(5)
C111	0.070(7)	0.070(7)	0.070(7)	-0.009(6)	0.010(5)	-0.022(6)
C112	0.034(5)	0.148(10)	0.044(6)	0.030(7)	-0.004(4)	-0.006(6)
C113	0.036(5)	0.062(6)	0.051(5)	-0.027(5)	-0.003(4)	0.003(4)
C114	0.048(6)	0.110(9)	0.040(5)	-0.019(6)	0.015(5)	0.038(6)
C115	0.041(5)	0.067(6)	0.067(7)	-0.005(5)	0.019(5)	-0.009(5)

Residue: 2.

C21	0.028(4)	0.009(3)	0.025(4)	0.000(3)	0.002(3)	-0.002(3)
C22	0.030(4)	0.023(4)	0.034(5)	0.003(4)	0.000(4)	0.002(3)
C23	0.027(4)	0.032(4)	0.033(4)	0.003(3)	0.002(3)	0.003(3)
C24	0.039(4)	0.014(4)	0.047(5)	0.003(4)	-0.016(4)	0.007(3)
C25	0.047(5)	0.022(4)	0.022(4)	0.008(3)	-0.001(3)	0.008(3)
C26	0.022(4)	0.026(4)	0.031(4)	-0.003(3)	-0.002(3)	0.004(3)
C27	0.025(3)	0.024(3)	0.031(4)	0.013(3)	0.002(3)	-0.003(3)
C28	0.033(4)	0.033(4)	0.021(4)	-0.007(4)	0.001(4)	0.002(3)
C29	0.031(4)	0.043(4)	0.032(4)	0.001(5)	-0.001(4)	-0.014(3)
C210	0.026(4)	0.067(6)	0.024(4)	0.004(5)	0.000(4)	0.003(4)
C211	0.034(4)	0.046(5)	0.033(5)	0.013(4)	0.012(4)	0.023(4)
C212	0.034(4)	0.027(4)	0.024(4)	0.008(4)	-0.005(3)	0.010(3)
C213	0.037(4)	0.014(3)	0.023(4)	0.007(3)	-0.001(3)	0.004(3)

C214	0.038(4)	0.019(4)	0.025(4)	0.009(3)	0.005(3)	0.006(3)
C215	0.047(5)	0.029(4)	0.015(4)	0.000(3)	-0.006(3)	0.012(3)
C216	0.049(5)	0.030(4)	0.036(5)	0.000(4)	-0.009(4)	-0.015(4)
C217	0.040(5)	0.049(5)	0.039(5)	-0.004(4)	0.004(4)	-0.017(4)
C218	0.036(4)	0.035(4)	0.021(4)	-0.005(3)	0.007(3)	-0.004(3)
C219	0.010(3)	0.021(4)	0.037(4)	-0.002(3)	0.002(3)	-0.001(3)
C220	0.021(4)	0.021(4)	0.032(4)	0.009(3)	0.008(3)	0.005(3)
C221	0.022(4)	0.043(5)	0.028(4)	0.010(4)	-0.001(3)	0.003(3)
C222	0.016(3)	0.060(6)	0.022(4)	-0.007(4)	-0.001(3)	-0.006(3)
C223	0.026(4)	0.041(5)	0.040(5)	-0.008(4)	0.001(3)	-0.010(4)
C224	0.017(4)	0.036(5)	0.035(5)	0.001(4)	-0.001(3)	-0.008(3)
B2	0.022(3)	0.019(4)	0.026(4)	-0.001(4)	0.001(4)	0.002(3)

Thermal vibration amplitudes ($Å^2$)

$$F(\mathbf{h}) = F_o(\mathbf{h}) \exp\left(-2\pi^2 \sum_{i=1}^{3}\sum_{j=1}^{3} h_i h_j a_i^* a_j^* U_{ij}\right)$$

or

$$F(\mathbf{h}) = F_o(\mathbf{h}) \exp\left(-8\pi^2 U_{iso} (\sin(\theta)/\lambda)^2\right)$$

Table 3. Data on the geometry.

Standard deviations in the last decimal place are given in parentheses.

Residue: 1.

Interatomic Distances (Å)

V1a	-P1	2.549(3)	
V1a	-N1	2.248(7)	
V1a	-C11	2.334(9)	
V1a	-C12	2.284(9)	
V1a	-C13	2.295(9)	
V1a	-C14	2.320(8)	
V1a	-C15	2.229(8)	
V1a	-C113	2.346(10)	
V1a	-C114	2.284(10)	
V1a	-C115	2.294(9)	
P1	-C110	1.832(9)	
P1	-C111	1.75(1)	
P1	-C112	1.789(11)	
V1b	-P1	2.506(10)	
V1b	-N1	1.611(12)	
V1b	-C18	2.303(15)	
V1b	-C113	2.216(14)	
V1b	-C114	2.148(14)	
N1	-C17	1.508(12)	
N1	-C18	1.527(11)	
N1	-C19	1.445(12)	
C11	-C12	1.458(13)	
C11	-C15	1.346(12)	
C12	-C13	1.384(13)	
C13	-C14	1.448(13)	
C14	-C15	1.389(11)	
C15	-C16	1.546(12)	
C16	-C17	1.549(14)	
C113	-C114	1.416(13)	
C114	-C115	1.388(14)	

Bond Angles (deg.)

P1	-V1a	-N1	90.0(2)	C15	-V1a	-C115	147.4(3)
P1	-V1a	-C11	143.1(2)	C113	-V1a	-C114	35.6(3)
P1	-V1a	-C12	130.4(2)	C113	-V1a	-C115	62.6(3)
P1	-V1a	-C13	95.7(2)	C114	-V1a	-C115	35.3(3)
P1	-V1a	-C14	85.0(2)	V1a	-P1	-C110	121.7(3)
P1	-V1a	-C15	110.8(2)	V1a	-P1	-C111	109.6(4)
P1	-V1a	-C113	124.8(2)	V1a	-P1	-C112	120.0(4)
P1	-V1a	-C114	91.7(3)	C110	-P1	-C111	98.7(5)
P1	-V1a	-C115	81.8(2)	C110	-P1	-C112	100.7(5)
N1	-V1a	-C11	89.0(3)	C111	-P1	-C112	102.9(5)
N1	-V1a	-C12	125.8(3)	V1a	-N1	-C17	107.4(5)
N1	-V1a	-C13	133.1(3)	V1a	-N1	-C18	113.0(5)
N1	-V1a	-C14	98.4(3)	V1a	-N1	-C19	114.1(6)
N1	-V1a	-C15	74.7(3)	C17	-N1	-C18	109.2(7)
N1	-V1a	-C113	89.5(3)	C17	-N1	-C19	106.8(7)
N1	-V1a	-C114	103.6(3)	C18	-N1	-C19	106.2(6)
N1	-V1a	-C115	136.9(3)	V1a	-C11	-C12	69.7(5)
C11	-V1a	-C12	36.8(3)	V1a	-C11	-C15	68.6(5)
C11	-V1a	-C13	59.9(3)	C12	-C11	-C15	105.0(7)
C11	-V1a	-C14	58.8(3)	V1a	-C12	-C11	73.5(5)
C11	-V1a	-C15	34.2(3)	V1a	-C12	-C13	72.8(5)
C11	-V1a	-C113	92.1(3)	C11	-C12	-C13	108.8(8)
C11	-V1a	-C114	124.3(4)	V1a	-C13	-C12	72.0(5)
C11	-V1a	-C115	121.9(3)	V1a	-C13	-C14	72.7(5)
C12	-V1a	-C13	35.2(3)	C12	-C13	-C14	107.2(8)
C12	-V1a	-C14	59.4(3)	V1a	-C14	-C13	70.7(5)

C12	-V1a	-C15	59.1(3)	V1a	-C14	-C15	68.7(4)
C12	-V1a	-C113	92.1(3)	C13	-C14	-C15	105.3(7)
C12	-V1a	-C114	108.5(4)	V1a	-C15	-C11	77.2(5)
C12	-V1a	-C115	89.6(3)	V1a	-C15	-C14	75.9(5)
C13	-V1a	-C14	36.6(3)	V1a	-C15	-C16	116.5(6)
C13	-V1a	-C15	59.8(3)	C11	-C15	-C14	113.2(7)
C13	-V1a	-C113	122.8(3)	C11	-C15	-C16	126.1(7)
C13	-V1a	-C114	122.6(3)	C14	-C15	-C16	120.7(7)
C13	-V1a	-C115	89.9(3)	C15	-C16	-C17	106.8(7)
C14	-V1a	-C15	35.5(3)	N1	-C17	-C16	108.7(7)
C14	-V1a	-C113	149.4(3)	V1a	-C113	-C114	69.8(6)
C14	-V1a	-C114	157.8(3)	V1a	-C114	-C113	74.6(5)
C14	-V1a	-C115	122.7(3)	V1a	-C114	-C115	72.8(6)
C15	-V1a	-C113	122.1(3)	C113	-C114	-C115	118.5(9)
C15	-V1a	-C114	157.3(4)	V1a	-C115	-C114	71.9(5)

[a] Indicates an s.o.f. of 0.78(1)

[b] Indicates an s.o.f. of 0.22(1)

Residue: 2.

Interatomic Distances (Å)

C21	-C22	1.41(1)	
C21	-C26	1.398(10)	
C21	-B2	1.615(9)	
C22	-C23	1.423(10)	
C23	-C24	1.379(11)	
C24	-C25	1.40(1)	
C25	-C26	1.371(10)	
C27	-C28	1.42(1)	
C27	-B2	1.642(10)	
C28	-C29	1.396(10)	
C210	-C29	1.373(10)	
C210	-C211	1.412(11)	
C211	-C212	1.363(10)	
C212	-C27	1.423(10)	
C213	-C214	1.418(10)	
C213	-C218	1.399(10)	
C213	-B2	1.621(9)	
C214	-C215	1.363(10)	
C215	-C216	1.372(10)	
C216	-C217	1.408(11)	
C217	-C218	1.391(11)	
C219	-C220	1.419(10)	
C219	-C224	1.388(10)	
C219	-B2	1.684(11)	
C220	-C221	1.397(10)	
C221	-C222	1.399(11)	
C222	-C223	1.388(12)	
C223	-C224	1.416(11)	

Bond Angles (deg.)

C22	-C21	-C26	113.5(6)	C213	-C214	-C215	125.2(6)
C22	-C21	-B2	123.2(6)	C214	-C215	-C216	119.4(7)
C26	-C21	-B2	122.6(6)	C215	-C216	-C217	120.1(7)
C21	-C22	-C23	122.7(7)	C216	-C217	-C218	117.4(7)
C22	-C23	-C24	120.3(6)	C213	-C218	-C217	125.7(7)
C23	-C24	-C25	118.1(6)	C220	-C219	-C224	116.2(6)
C24	-C25	-C26	120.1(7)	C220	-C219	-B2	122.2(5)
C21	-C26	-C25	125.3(7)	C224	-C219	-B2	121.4(6)
C28	-C27	-C212	114.6(6)	C219	-C220	-C221	122.4(7)
C28	-C27	-B2	122.1(6)	C220	-C221	-C222	120.3(7)
C212	-C27	-B2	123.0(5)	C221	-C222	-C223	118.3(7)
C27	-C28	-C29	123.6(7)	C222	-C223	-C224	121.0(8)
C28	-C29	-C210	119.5(7)	C219	-C224	-C223	121.8(7)
C29	-C210	-C211	118.7(6)	C21	-B2	-C27	115.0(5)
C210	-C211	-C212	121.6(7)	C21	-B2	-C213	104.3(5)
C27	-C212	-C211	122.0(7)	C21	-B2	-C219	111.6(5)
C214	-C213	-C218	112.2(6)	C27	-B2	-C213	114.5(5)
C214	-C213	-B2	124.3(6)	C27	-B2	-C219	100.1(6)
C218	-C213	-B2	122.8(6)	C213	-B2	-C219	111.6(5)