

Fig. 1. The molecule of $[(C_6H_5CH_2)_2(C_6H_5)P]_2PtCl_2$, showing the atomic numbering.

closest Pt—C(*o*) approach is 3.61 (1) Å, to C(123), too long to indicate incipient *ortho*-metallation. The title compound appears to be significantly more crowded than either of the parallels cited above, from its P—Pt—P angle [103.0 (1) *vs* 94.5 and 96.2°]. It also shows the single large C—P—M angle [here 120.2 (4)°] similar to those of 120.7 and 121.5° found in the notably overcrowded $\{(C_3H_7)_3P\}PdCl_2$ (Alcock, Kemp & Wimmer, 1981). However, the conformation around Pt shows only slight deviations from planarity with Cl and P atoms alternately up and down by ± 0.07 Å. This is probably better viewed as a twist between the

P—Pt—P plane and the Cl—Pt—Cl plane, of 5.1 (2)°. The title compound is also more crowded than the *ortho*-metallated compound for which the PPtP angle is 106.0° and the twist angle is 1.5°.

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Tetrakis(4-ethylpyridinium) Decavanadate

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Abstract. $[NHC_7H_9]_4[V_{10}H_2O_{28}]$, monoclinic, $P2_1/n$, $a = 19.473$ (2), $b = 9.938$ (1), $c = 11.965$ (4) Å, $\beta = 92.26$ (1)°, $V = 2314.8$ Å³, $\mu(Mo K\alpha) = 17.35$ mm⁻¹, D_m (pycnometric method) = $D_x = 2.00$ Mg m⁻³ for

$Z = 2$. The structure was solved by direct methods and refined to a final R value of 0.08 for 2842 observed reflexions. The 4-ethylpyridinium groups are bonded to the oxygens of the decavanadate anion by an intermolecular hydrogen bond between the pyridine nitrogen and the most basic oxygens of the decavanadate group. This group consists of ten distorted VO_6 octahedra that

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share edges and is basically the same as that in the analogous inorganic decavanadates.

Introduction. Yellow-orange crystals of tetrakis-(4-ethylpyridinium) decavanadate, with a nearly hexagonal cross-section, have been synthesized for the first time by one of us (Arrieta, 1980). The chemical analysis of V, C, H and N gave the following calculated (experimental) results: V_2O_5 65.3 (65.2), C 24.2 (24.1), H 3.0 (3.0), N 4.0 (4.3%). Vanadium was analysed as V_2O_5 after calcination for 2h at 823K.

The intensities of 3014 independent reflexions ($\theta_{\max} = 25^\circ$) were collected on a Philips PW 1100 four-circle diffractometer (Mo $K\alpha$ radiation, graphite monochromator, ω - 2θ scan) using a crystal $0.15 \times 0.12 \times 0.02$ mm. The 2842 reflexions with $I_{hkl} \geq 1.5\sigma(I)$ were considered as observed and included in the refinement. The relative intensities were corrected for the usual Lorentz-polarization factors. No absorption correction was made.

The structure was solved by direct methods using the MULTAN 78 computer system (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). The computation of an E map with the phases of the set with the highest 'combined figure of merit' revealed the position

Table 1. Atomic coordinates and equivalent isotropic temperature factors

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$$

	x	y	z	$U_{eq}(\text{\AA}^2)$
V(1)	0.5693 (1)	0.0418 (2)	0.5749 (1)	0.023 (2)
V(2)	0.5829 (1)	-0.0929 (2)	0.3396 (1)	0.027 (2)
V(3)	0.4656 (1)	0.2608 (2)	0.4683 (1)	0.027 (2)
V(4)	0.6187 (1)	0.2086 (2)	0.3790 (1)	0.030 (2)
V(5)	0.4901 (1)	0.1306 (2)	0.2363 (1)	0.030 (2)
O(6)	0.5243 (3)	0.0793 (6)	0.4211 (5)	0.008 (5)
O(7)	0.4152 (3)	0.0141 (7)	0.2929 (5)	0.010 (6)
O(8)	0.6347 (3)	0.1410 (7)	0.5428 (5)	0.012 (6)
O(9)	0.5588 (3)	0.3338 (7)	0.4561 (6)	0.016 (6)
O(10)	0.4307 (4)	0.3939 (7)	0.5176 (6)	0.022 (7)
O(11)	0.5370 (3)	-0.0245 (7)	0.2150 (5)	0.011 (6)
O(12)	0.5040 (3)	0.1778 (6)	0.6169 (5)	0.007 (6)
O(13)	0.6847 (4)	0.3011 (7)	0.3644 (6)	0.022 (7)
O(14)	0.6214 (4)	-0.2183 (7)	0.2871 (6)	0.019 (7)
O(15)	0.5997 (3)	-0.1211 (6)	0.5010 (5)	0.013 (6)
O(16)	0.4457 (4)	0.2672 (7)	0.3264 (6)	0.015 (7)
O(17)	0.5700 (3)	0.2300 (7)	0.2486 (5)	0.014 (6)
O(18)	0.6489 (3)	0.0399 (7)	0.3392 (5)	0.013 (6)
O(19)	0.4596 (4)	0.1639 (8)	0.1145 (6)	0.022 (7)
N(20)	0.1948 (5)	0.4434 (11)	0.6283 (8)	0.075 (12)
C(21)	0.1628 (6)	0.5094 (12)	0.5424 (10)	0.046 (13)
C(22)	0.1819 (6)	0.4859 (12)	0.4385 (10)	0.045 (12)
C(23)	0.2348 (6)	0.3946 (13)	0.4166 (10)	0.049 (13)
C(24)	0.2664 (7)	0.3277 (13)	0.5071 (10)	0.053 (14)
C(25)	0.2453 (6)	0.3520 (14)	0.6145 (10)	0.054 (14)
C(26)	0.2541 (9)	0.3704 (16)	0.2979 (12)	0.076 (18)
C(27)	0.2506 (12)	0.2246 (19)	0.2655 (15)	0.103 (24)
N(30)	0.0459 (4)	0.1852 (10)	0.2941 (8)	0.061 (10)
C(31)	0.0644 (6)	0.0556 (12)	0.2758 (10)	0.049 (13)
C(32)	0.0873 (6)	-0.0215 (12)	0.3655 (9)	0.044 (12)
C(33)	0.0923 (5)	0.0300 (12)	0.4714 (9)	0.040 (12)
C(34)	0.0737 (6)	0.1655 (13)	0.4863 (10)	0.051 (13)
C(35)	0.0500 (6)	0.2400 (12)	0.3952 (9)	0.044 (12)
C(36)	0.1165 (7)	-0.0544 (15)	0.5679 (11)	0.064 (15)
C(37)	0.1914 (9)	-0.0725 (30)	0.5708 (19)	0.154 (38)

Table 2. Interatomic angles ($^\circ$)

The superscript (i) refers to the symmetry operation $\bar{x} + 1, \bar{y}, \bar{z} + 1$.

V(2)-V(1)	-V(3)	90.4 (1)	O(11)-V(2)-O(18)	92.9 (3)
	-V(4)	60.2 (1)	-O(12 ⁱ)	88.7 (5)
	-V(1 ⁱ)	60.0 (1)	O(15)-V(2)-O(18)	90.9 (3)
	-V(2 ⁱ)	117.7 (1)	-O(12 ⁱ)	78.0 (4)
	-V(3 ⁱ)	57.7 (1)	O(18)-V(2)-O(12 ⁱ)	155.5 (5)
	-V(5 ⁱ)	117.8 (1)	O(6)-V(3)-O(9)	78.2 (2)
V(3)-V(1)	-V(1 ⁱ)	57.5 (1)	-O(10)	171.8 (3)
	-V(2 ⁱ)	56.2 (1)	-O(12)	73.9 (2)
	-V(3 ⁱ)	117.4 (1)	-O(16)	83.0 (3)
	-V(5 ⁱ)	114.8 (1)	-O(15 ⁱ)	79.3 (4)
V(4)-V(1)	-V(1 ⁱ)	89.7 (1)	O(9)-V(3)-O(10)	97.4 (3)
	-V(2 ⁱ)	118.7 (1)	-O(12)	84.7 (3)
	-V(3 ⁱ)	117.9 (1)	-O(16)	94.9 (3)
	-V(5 ⁱ)	176.0 (1)	-O(15 ⁱ)	153.9 (5)
V(1 ⁱ)-V(1)	-V(2 ⁱ)	57.7 (1)	O(10)-V(3)-O(12)	99.0 (3)
	-V(3 ⁱ)	59.8 (1)	-O(16)	104.4 (4)
	-V(5 ⁱ)	86.3 (1)	-O(15 ⁱ)	103.3 (5)
V(2 ⁱ)-V(1)	-V(3 ⁱ)	90.4 (1)	O(12)-V(3)-O(16)	156.5 (3)
	-V(5 ⁱ)	58.6 (1)	-O(15 ⁱ)	76.6 (4)
V(1)-V(2)	-V(4)	58.6 (1)	O(16)-V(3)-O(15 ⁱ)	95.2 (5)
	-V(5)	89.0 (1)	O(6)-V(4)-O(8)	73.0 (2)
	-V(1 ⁱ)	62.3 (1)	-O(9)	76.0 (2)
	-V(3 ⁱ)	60.9 (1)	-O(13)	173.6 (3)
V(4)-V(2)	-V(5)	59.5 (1)	-O(17)	82.0 (3)
	-V(1 ⁱ)	90.2 (1)	-O(18)	79.4 (3)
	-V(3 ⁱ)	119.5 (1)	O(8)-V(4)-O(9)	80.3 (3)
V(5)-V(2)	-V(1 ⁱ)	58.8 (1)	-O(13)	101.4 (3)
	-V(3 ⁱ)	120.4 (1)	-O(17)	154.8 (3)
V(1)-V(3)	-V(4)	56.6 (1)	-O(18)	85.0 (3)
	-V(5)	87.5 (1)	O(9)-V(4)-O(13)	100.5 (3)
	-V(1 ⁱ)	62.7 (1)	-O(17)	91.7 (3)
	-V(2 ⁱ)	62.1 (1)	-O(18)	154.1 (3)
V(4)-V(3)	-V(5)	57.7 (1)	O(13)-V(4)-O(17)	103.6 (4)
	-V(1 ⁱ)	89.3 (1)	-O(18)	103.2 (3)
	-V(2 ⁱ)	118.7 (1)	O(17)-V(4)-O(18)	92.6 (3)
V(5)-V(3)	-V(1 ⁱ)	59.5 (1)	O(6)-V(5)-O(11)	79.7 (3)
	-V(2 ⁱ)	120.8 (1)	-O(16)	75.3 (2)
V(1 ⁱ)-V(3)	-V(2 ⁱ)	61.4 (1)	-O(17)	80.4 (3)
V(1)-V(4)	-V(2)	61.2 (1)	-O(19)	174.6 (3)
	-V(3)	60.9 (1)	-O(7 ⁱ)	74.8 (4)
	-V(5)	90.9 (1)	O(11)-V(5)-O(16)	154.5 (3)
V(2)-V(4)	-V(3)	90.1 (1)	-O(17)	92.2 (3)
	-V(5)	60.0 (1)	-O(19)	102.7 (3)
V(3)-V(4)	-V(5)	59.0 (1)	-O(7 ⁱ)	86.2 (5)
V(2)-V(5)	-V(3)	92.9 (1)	O(16)-V(5)-O(17)	88.5 (3)
	-V(4)	60.5 (1)	-O(19)	101.7 (3)
	-V(1 ⁱ)	62.6 (1)	-O(7 ⁱ)	82.6 (5)
V(3)-V(5)	-V(4)	63.3 (1)	O(17)-V(5)-O(19)	104.2 (4)
	-V(1 ⁱ)	60.3 (1)	-O(7 ⁱ)	155.1 (5)
V(4)-V(5)	-V(1 ⁱ)	93.1 (1)	V(1)-O(6)-V(2)	92.5 (2)
O(6)-V(1)	-O(7)	163.1 (5)	-V(3)	97.2 (2)
	-O(8)	89.4 (3)	-V(4)	89.0 (2)
	-O(6 ⁱ)	78.0 (4)	V(1)-O(6)-V(5)	171.1 (3)
O(7)-V(1)	-O(8)	107.4 (5)	-V(1 ⁱ)	102.0 (4)
	-O(6 ⁱ)	85.2 (4)	V(2)-O(6)-V(3)	169.5 (3)
O(12)-V(1)	-O(6)	81.0 (2)	-V(4)	84.8 (2)
	-O(7)	94.7 (5)	-V(5)	83.6 (2)
	-O(8)	99.3 (3)	-V(1 ⁱ)	91.6 (4)
	-O(15)	155.9 (3)	V(3)-O(6)-V(4)	91.4 (2)
O(15)-V(1)	-O(6)	82.5 (3)	-V(5)	86.2 (2)
	-O(7)	96.0 (5)	-V(1 ⁱ)	90.2 (4)
	-O(8)	97.9 (3)	V(4)-O(6)-V(5)	82.7 (2)
	-O(6 ⁱ)	79.7 (4)	-V(1 ⁱ)	168.6 (5)
O(6 ⁱ)-V(1)	-O(8)	167.3 (5)	V(5)-O(6)-V(1 ⁱ)	86.2 (3)
O(6)-V(2)	-O(11)	80.5 (3)	V(1)-O(7)-V(5 ⁱ)	113.6 (5)
	-O(14)	176.8 (3)	V(1)-O(8)-V(4)	108.6 (3)
	-O(15)	75.8 (2)	V(3)-O(9)-V(4)	111.7 (3)
	-O(18)	80.0 (3)	V(5)-O(11)-V(2)	115.5 (3)
	-O(12 ⁱ)	76.2 (4)	V(1)-O(12)-V(3)	105.9 (3)
O(14)-V(2)	-O(11)	101.0 (3)	-V(2 ⁱ)	110.4 (5)
	-O(15)	102.3 (3)	V(3)-O(12)-V(2)	96.4 (4)
	-O(18)	102.8 (3)	V(1)-O(15)-V(2)	106.6 (3)
	-O(12 ⁱ)	101.0 (5)	-V(3 ⁱ)	107.3 (5)
O(15)-V(2)	-O(11)	155.0 (3)	V(2)-O(15)-V(3 ⁱ)	101.8 (5)
			V(3)-O(16)-V(5)	115.4 (4)

Table 2 (cont.)

V(4)–O(17)–V(5)	114.7 (4)	V(2)–O(18)–V(4)	114.9 (3)
C(21)–N(20)–C(25)	123 (1)	C(31)–N(30)–C(35)	122 (1)
N(20)–C(21)–C(22)	119 (1)	N(30)–C(31)–C(32)	119 (1)
C(21)–C(22)–C(23)	121 (1)	C(31)–C(32)–C(33)	122 (1)
C(22)–C(23)–C(24)	118 (1)	C(32)–C(33)–C(34)	118 (1)
C(22)–C(23)–C(26)	120 (1)	C(32)–C(33)–C(36)	121 (1)
C(24)–C(23)–C(26)	123 (1)	C(34)–C(33)–C(36)	121 (1)
C(23)–C(24)–C(25)	120 (1)	C(33)–C(34)–C(35)	120 (1)
C(24)–C(25)–N(20)	118 (1)	C(34)–C(35)–N(30)	120 (1)
C(23)–C(26)–C(27)	113 (1)	C(33)–C(36)–C(37)	112 (1)

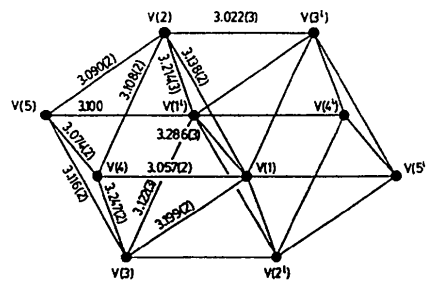


Fig. 2. A view of the vanadium frame in the decavanadate molecule. (Distances in Å.)

of the V atom and its six nearest oxygen neighbours of the polyanion. The remaining atoms of the molecule (N and C atoms) were found by successive refinements and Fourier synthesis.

The atom parameters were refined using the *SHELX* 76 program (Sheldrick, 1976) until the shifts were smaller than the corresponding standard deviations. During the first five cycles isotropic thermal parameters were used and the *R* index reached the value 0.11; during the five following cycles, anisotropic thermal parameters were used and the *R* index dropped to the final value of 0.08. The H atoms were not taken into account in this work.

The final atomic positions are given in Table 1.* The V–O distances are given in Fig. 1. Fig. 2 shows the V–V distances and Fig. 3 shows a projection of the whole molecule on the *yz* plane as well as the C–C, C–N and N–O distances. The bond angles are given in Table 2.

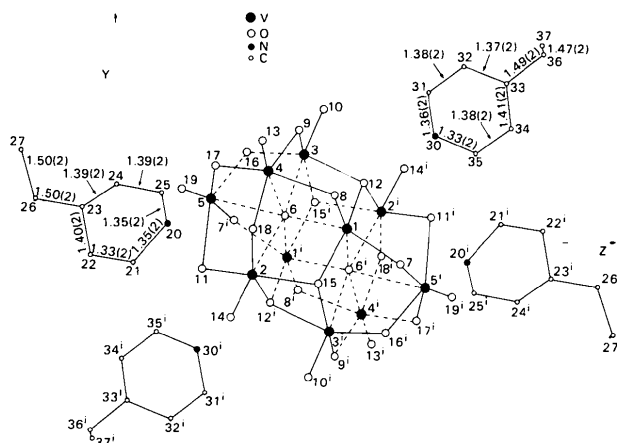


Fig. 3. A complete view of the decavanadate molecule with the surrounding ethylpyridinium molecules.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36868 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

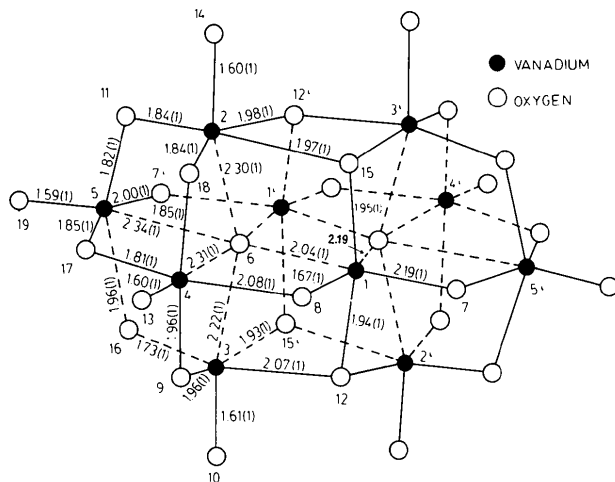


Fig. 1. A general view of the decavanadate molecule showing the V–O distances (Å).

Discussion. The decavanadate group consists of ten distorted VO_6 octahedra that share edges. One half of the molecule is related to the other by a centre of symmetry located between O(6) and O(6'). Within the molecule there are two O atoms, each surrounded by six V atoms at the corners of an octahedron. The VO_6 octahedra show a considerable distortion due to the mutual repulsion of the V atoms with the consequence that the V(4)–V(1)–V(5') angle is reduced from 180 to $176.0(1)^\circ$ and the V(2)–O(6)–V(3) angle to $169.5(3)$. In order to understand this distortion Kepert (1973) has interpreted it by taking into account the V–O distances *versus* the number of V atoms bonded to each of the O atoms. This interpretation agrees with our experimental results. The maximum and minimum distances for oxygen bonded to six V atoms are 2.34 and 2.05 Å (mean 2.24 Å), to three V atoms 2.08 and 1.93 Å (mean 1.98 Å), to two V atoms 2.01 and 1.67 Å (mean 1.86 Å), to one V atom 1.61 and 1.59 Å (mean 1.60 Å). These distances are in agreement with those found by Evans (1966), Shallow, Ahmed & Barnes (1966) and Durif, Averbuch-Pouchot & Guitel (1980) in inorganic decavanadates.

Other distances of interest are those between the N atom of the pyridine ring and the O atoms of the decavanadate group. The N(30)–O(12) and N(20)–O(18) distances are respectively 2.63 (2) and 2.73 (2) Å. These distances are typical for very strong intermolecular hydrogen bonds.

Our X-ray structural data agree with the results found by Klemperer & Shum (1977), who have determined the protonation positions in the $V_{10}O_{28}^{6-}$ ion by means of ^{17}O NMR, and, according to their work, the sequence of negative charge on the oxygens in our decavanadate ion would be $O(10) = O(14) \simeq O(19) = O(13) < O(7) = O(8) < O(17) < O(9) = O(16) = O(11) = O(18) < O(15) = O(12) < O(16)$. As O(6) is inaccessible to the protons and, of course, to the larger groups such as the 4-ethylpyridinium group, the sequence predicts O(12) and O(18) as the most basic O atoms, O(12) being more basic than O(18). This, added to the steric effects of such a large group, explains why the N–O(12) distance is slightly shorter than the N–O(18) distance.

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catena- μ -[1,2-Bis(diphenylphosphino)ethane]-dicyanomercure(II)

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Abstract. $C_{28}H_{24}HgN_2P_2$, $M_r = 651.05$, triclinic, space group $P\bar{1}$, $a = 9.586$ (2), $b = 11.793$ (2), $c = 12.343$ (2) Å, $\alpha = 104.55$ (1), $\beta = 107.69$ (1), $\gamma = 97.74$ (1)°, $V = 1252.6$ (4) Å³, $Z = 2$, $d_c = 1.726$, $d_x = 1.70$ (1) g cm⁻³ (flotation in ZnI₂ solution), Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å, $\mu = 62.86$ cm⁻¹, $F(000) = 627.422$. The structure was determined by a least-squares procedure from the measured angular positions of 15 reflections. Final $R = 0.056$ for 3704 independent observed reflections. The structure is built up by packing of infinite chains of $[Hg(CN)_2\{P(C_6H_5)_2CH_2\}_2]_n$. The two Hg–P bonds are significantly different: 2.606 (3) and 2.534 (4) Å. No short contacts occur between the chains in the crystal.

Introduction. Lattice constants and intensity measurements were performed on a computer-controlled

automatic Syntex P2₁ diffractometer using graphite-monochromatized Mo $K\alpha$ radiation and a colourless crystal of dimensions 0.04 × 0.12 × 0.15 mm.

Intensities of the reflections up to $\theta = 28^\circ$ were measured by the ω -scan technique at a scan rate automatically chosen between 2.0 and 29.3° min⁻¹ with a scan range of 1.0°. Background counts were taken, at 1.0° from the peak position, for the scan time. The intensity of three standard reflections, measured for every 100, remained constant throughout the run.

The intensity data were processed as previously described (Bachechi, Zambonelli & Marcotrigiano, 1977), using an uncertainty factor $p = 0.004$, calculated from the variance of the standard reflections (McCandlish, Stout & Andrews, 1975). The values of I and $\sigma(I)$ were corrected for Lorentz, polarization and shape-anisotropy effects.