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Structure of 1,6-Hexanediammonium Metavanadate

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Abstract. $[H_3N—(CH_2)_6—NH_3]^{2+}.(VO_3^-)_2$, M_r = 316.10, orthorhombic, *Pbca*, a = 5.379 (4), b = 11.074 (9), c = 19.58 (2) Å, V = 1166 (2) Å^3, Z = 4, D_m = 1.76 (1), D_x = 1.800 g cm^{-3}, Mo $K\alpha$, λ = 0.71069 Å, μ = 15.65 cm^{-1}, $F(000)$ = 648, room temperature, final R = 0.0339 for 918 observed reflections. The structure is made up of infinite $(VO_3)_\infty$ chains running parallel to the *a* axis. The doubly protonated hexanediamine molecule is located on a centre of symmetry.

Introduction. Typical structures of anhydrous metavanadates M^IVO_3 comprise infinite chains of corner-sharing VO_4 tetrahedra. Fourfold O-atom coordination of V atoms was found by Synečék & Hanic (1954) in NH_4VO_3, Petrášová, Madar & Hanic (1958) in KVO_3, Shannon & Calvo (1973) in $LiVO_3$, Marumo, Isobe & Iwai (1974) in α-$NaVO_3$, and Ganne, Piffard & Tournoux (1974) in $TlVO_3$.

In the $M^{II}(VO_3)_2$ metavanadate structures there is greater variability among the VO_n coordination polyhedra. Most of the previously investigated metavanadates have a distorted octahedral coordination around the V atoms characterized by five strong and one weak (~2.6 Å) V—O interactions. Examples are β-$Cd(VO_3)_2$ (Bouloux & Galy, 1969), $Mg(VO_3)_2$ (Ng & Calvo, 1972), $Co(VO_3)_2$ (Sauerbrei, 1972), $Zn(VO_3)_2$ (Angenault & Rimsky, 1968; Andreetti, Calestani, Montenero & Bettinelli, 1984), $Cu(VO_3)_2$ (Calvo & Manolescu, 1973), $Pb(VO_3)_2$ (I) (Jordan & Calvo, 1974) and β-$Hg(VO_3)_2$ (Angenault, 1970).

Loss of the sixth weak interaction is characteristic for the structure $Pb(VO_3)_2$ (II) (Calestani, Andreetti, Montenero & Bettinelli, 1985), which consists of columns of double chains of edge-sharing VO_5 square pyramids. This coordination was found in the structure $Co(VO_3)_2$ (Jasper-Tönnies & Müller-Buschbaum, 1984). In the structures $Ca(VO_3)_2$ and α-$Cd(VO_3)_2$ (Bouloux, Perez & Galy, 1972) and α-$Sr(VO_3)_2$ (Karpov, Simonov, Krasnenko & Zabara, 1989) are found trigonal bipyramidal coordinations of the atom V^V.

The structures of $Pb(VO_3)_3$ (III) (Calestani, Andreetti, Montenero, Bettinelli & Rebizant, 1985) and $Ba(VO_3)_2$ (Launay & Thoret, 1973) are different from those of other $M^{II}(VO_3)_2$ systems and consist of chains of corner-sharing VO_4 tetrahedra. The structure of $[H_3N—(CH_2)_6—NH_3]^{2+}.(VO_3^-)_2$ has been investigated to provide further structural data on the metavanadates.

Experimental. 1,6-Hexanediammonium metavanadate was prepared by dissolving V_2O_5 in aqueous solution of hexanediamine with gentle heating. The colourless solution was slowly cooled at room temperature and then filtrated. For preparation of crys-

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tals acetone must be slowly added to the solution. This solution was placed in a refrigerator and over several days hexagonal crystals were formed.

A crystal with dimensions 0.4 × 0.3 × 0.18 mm was chosen for the measurements. D_m was determined by flotation in dioxane diluted by bromoform. The space group was determined from Weissenberg photographs. Unit-cell parameters were determined by least-squares fit of positional angles of 15 reflections with $8 < 2\theta < 18°$. Intensity data were measured on a Syntex $P2_1$ diffractometer using graphite-monochromated Mo $K\alpha$ radiation and ω–2θ scan mode with variable scan rate 4.88 to 29.3° min^{-1} in 2θ. Two standard reflections were monitored every 98 reflections, no significant systematic fluctuation was detected. Background measured at each end of the scan interval for one half of the scan time. 1647 measured reflections [$0 \leq 2\theta \leq 55°$, max. $(\sin\theta)/\lambda = 0.651$ Å^{-1} (h 0→6, k 0→14, l 0→25)] were averged to 1343 unique and 918 observed [$|F_o| > 3.92\sigma(F)$] reflections ($R_{\text{int}} = 0.02$, for standard reflections). Data were not corrected for absorption effects. The structure was solved by Patterson and Fourier methods with program *XFPS*89 (Pavelčík & Sivý, 1989). Refinement on $|F_o|$ by block-diagonal matrix least squares with anisotropic displacement parameters. All H atoms were located in $\Delta\rho$ maps and refined isotropically. The three strongest reflections, affected by extinction, were omitted from refinement. 109 parameters were refined. Convergence was obtained at $(\Delta/\sigma)_{\text{max}} = 0.0385$. $R = 0.0339$ and $wR = 0.0384$ for observed data ($R = 0.0594$ for all reflections); $S = 1.16$; $(\Delta\rho)_{\text{max}} = 0.341$, $(\Delta\rho)_{\text{min}} = -0.298$ e Å^{-3}. A weighting scheme $w^{-1} = \sigma^2(F) + (0.02|F_o|)^2$ was used. Scattering factors for neutral atoms with anomalous-dispersion correction for the V atom were from *International Tables for X-ray Crystallography* (1974, Vol. IV). Final atomic coordinates and equivalent isotropic displacement factors for nonhydrogen atoms are given in Table 1.*

Discussion. The crystal structure of $[NH_3(CH_2)_6NH_3](VO_3)_2$ projected onto the (100) plane is shown in Fig. 1. The metavanadate anion is formed by corner-sharing VO_4 tetrahedra which are connected *via* the O(1) atoms into infinite chains running parallel to the *a* axis. The bond lengths between V and the bridging O(1) atoms are much longer, 1.789 (2) Å, than those between V and the unshared O(2) and

Table 1. *Final atomic coordinates* (× 10^4) *and equivalent isotropic displacement parameters* B_{eq} (Å^2) *with e.s.d.'s in parentheses*

$$B_{\text{eq}} = (4/3)\sum_{i=1}^{3} B_{ii}a_i^2.$$

	x	y	z	B_{eq}
V	2098 (1)	1543 (1)	2061 (1)	1.61 (1)
O(1)	203 (1)	1057 (1)	2760 (1)	2.37 (2)
O(2)	1981 (1)	3014 (1)	2010 (1)	2.48 (2)
O(3)	1058 (2)	972 (1)	1359 (1)	3.02 (2)
N(1)	2195 (2)	1030 (1)	8390 (1)	2.49 (2)
C(1)	4059 (2)	107 (1)	9724 (1)	3.44 (3)
C(2)	3735 (3)	1404 (1)	9549 (1)	3.70 (3)
C(3)	1735 (2)	1657 (1)	9040 (1)	2.96 (3)

Table 2. *Interatomic distances* (Å); *bond angles* (°) *and hydrogen-bond geometry with e.s.d.'s in parentheses*

V—O(1)	1.789 (2)	V—O(3)	1.613 (3)
V—O(2)	1.633 (2)	V—O(1^{i})	1.789 (3)
C(3)—C(2)	1.492 (3)	C(1)—C(2)	1.488 (2)
C(1)—C(1^{ii})	1.499 (3)	C(3)—N(1)	1.471 (3)
O(1)—V—O(2)	108.97 (4)	O(2)—V—O(3)	109.00 (4)
O(1)—V—O(3)	109.64 (8)	O(2)—V—O(1^{i})	110.37 (6)
O(1)—V—O(1^{i})	106.95 (11)	O(3)—V—O(1^{i})	111.85 (10)
V(1)—O(1)—V^{iii}	126.20 (10)		
C(2)—C(1)—C(1^{ii})	113.50 (12)	N(1)—C(3)—C(2)	111.58 (11)
C(1)—C(2)—C(3)	114.83 (12)		

X—H···Y	X···Y	Y···H	X—H···Y
N(1)—H(7)···O(2^{iv})	2.905 (4)	2.170 (12)	137.3 (13)
N(1)—H(8)···O(2^{v})	2.892 (3)	2.000 (15)	154.6 (11)
N(1)—H(9)···O(3^{vi})	2.867 (3)	2.162 (12)	144.7 (10)

Symmetry code: (i) $\frac{1}{2} + x, y, \frac{1}{2} - z$; (ii) $1 - x, -y, 2 - z$; (iii) $x - \frac{1}{2}, y, \frac{1}{2} - z$; (iv) $x, \frac{1}{2} - y, \frac{1}{2} + z$; (v) $\frac{1}{2} + x, \frac{1}{2} - y, 1 - z$; (vi) $-x, -y, 1 - z$.

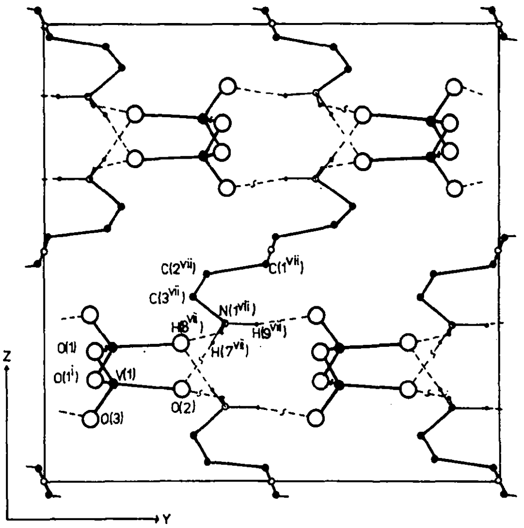

Fig. 1. Projection of the $[H_3N(CH_2)_6NH_3](VO_3)_2$ crystal structure onto the (100) plane. Symmetry code: (vii) $x, \frac{1}{2} - y, z - \frac{1}{2}$.

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*Lists of structure factors, anisotropic displacement parameters and hydrogen positions have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54912 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB0238]

O(3) atoms, 1.633 (2) and 1.613 (3) Å (see Table 2). The O—V—O angles are in the range 106.95 (11) to 111.85 (10)°, indicating a rather undistorted tetrahedral environment of the V atom.

Interatomic distances and bond angles in the VO_4 tetrahedra are in good agreement with those found by Bensch, Hug, Reller & Oswald (1987). They are within the range observed in other metavanadates. The doubly protonated molecules of the hexanediamine are located between the $(VO_3)_\infty$ chains. As indicated by the dotted lines in Fig. 1, a hydrogen-bond network is formed between H(7), H(8), H(9) and O(2^{iv}), O(2^{v}), O(3^{vi}) oxygen atoms, which are terminal O atoms of VO_4 tetrahedra. The geometries for the strongest hydrogen bonds are also given in Table 2.

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[(Methyldiphenylphosphonio)methanide]tris(pentafluorophenyl)gold(III) Acetone Solvate

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Abstract. $[Au(C_{14}H_{15}P)(C_{18}F_{15})].C_3H_6O$, $M_r = 970.45$, triclinic, $P\bar{1}$, $a = 11.258$ (3), $b = 13.586$ (3), $c = 13.969$ (3) Å, $\alpha = 108.77$ (2), $\beta = 101.69$ (2), $\gamma = 112.07$ (1)°, $V = 1742$ (1) Å^3, $Z = 2$, $D_x = 1.850$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 4.4$ mm^{-1}, $F(000) = 936$, $T = 293$ K, $R = 0.036$ for 6980 reflections. The coordination at the Au atom is square planar, with Au—C(ylide) 2.109 (6), Au—C_6F_5 2.055–2.069 (4–6) Å and P—C(ylide) 1.773 (6) Å. No *trans* effect is observed.

Introduction. Organogold chemistry is one of the main topics of research in the groups of Professors R. Usón and A. Laguna, University of Zaragoza, Spain; for recent review articles see Usón (1986), Laguna & Laguna (1990). In particular, they have reported the synthesis of the [tris(pentafluorophenyl)](ylide)gold(III) complexes $(C_6F_5)_3Au(CH_2PPh_3)$ (Usón, Laguna, Laguna & Usón, 1983) and, in a short communication, $(C_6F_5)_3Au(CH_2PPh_2Me)$ (Usón, Laguna, Laguna, Usón & Gimeno, 1987). Here we present the crystal structure of the latter compound.

Experimental. Colourless tablets were obtained from acetone/hexane. A crystal 0.6 × 0.2 × 0.1 mm was mounted in a glass capillary. Using a Stoe four-circle diffractometer, 9141 intensities were registered to $2\theta_{max} = 55°$ with monochromated Mo $K\alpha$ radiation; θ-scan technique. Of 7968 unique reflections ($R_{int} = 0.033$, index ranges h −14 to 14, k −16 to 17, l 0 to 18), $6980 > 4\sigma(F)$ were considered observed. The cell

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