

Zandbergen, 1991) describing a new homologous series $\text{La}_{4+4n}\text{Cu}_{8+2n}\text{O}_{14+8n}$. The $n = 2$ member of this series is $\text{La}_2\text{Cu}_2\text{O}_5$. As described by Cava *et al.*, this phase is structurally unrelated to $\text{La}_2\text{Cu}_2\text{O}_5$ described herein, and contains copper in four- and sixfold coordination. Close examination of the X-ray powder patterns of the two phases revealed no obvious similarities. Thus, as with LaCuO_3 , $\text{La}_2\text{Cu}_2\text{O}_5$ adopts more than one structural type.

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Structure of Hexakis(3-methylpyridinium) Decavanadate Bis(3-methylpyridinium perchlorate) Dihydrate

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Abstract. $[\text{V}_{10}\text{O}_{28}(\text{C}_6\text{H}_7\text{NH})_6].2(\text{C}_6\text{H}_7\text{NH}).2(\text{ClO}_4).2\text{H}_2\text{O}$, $M_r = 1945.4$, monoclinic, $P2_1/c$, $a = 12.580$ (2), $b = 26.262$ (5), $c = 11.340$ (4) Å, $\beta = 108.11$ (2)°, $V = 3561$ (2) Å³, $Z = 2$, $D_m = 1.8$ (1) (by flotation in $\text{CHBr}_3\text{-CCl}_4$), $D_x = 1.81$ Mg m⁻³, $\lambda(\text{Mo K}\alpha) = 0.71069$ Å, $\mu = 1.38$ mm⁻¹, $F(000) = 1960$, $T = 298$ K, $R = 0.025$ for 1789 observed reflections. The formula unit consists of the decavanadate anion, two perchlorate anions, eight 3-methylpyridinium cations and two water molecules and is centrosymmetric. The polyanion is similar to other known decavanadates. The perchlorate ions are disordered.

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Introduction. The structures of several decavanadates have been described previously: $\text{K}_2\text{Zn}_2(\text{V}_{10}\text{O}_{28}).16\text{H}_2\text{O}$ (Evans, 1966), $\text{Ca}_3(\text{V}_{10}\text{O}_{28}).17\text{H}_2\text{O}$ (Swallow, Ahmed & Barnes, 1966), $\text{Nd}_2\text{V}_{10}\text{O}_{28}.28\text{H}_2\text{O}$ (Saf'yanov & Belov, 1976), $\text{Y}_2\text{V}_{10}\text{O}_{28}.24\text{H}_2\text{O}$ (Saf'yanov, Kuz'min & Belov, 1978a), $\text{La}_2\text{V}_{10}\text{O}_{28}.20\text{H}_2\text{O}$ (Saf'yanov, Kuz'min & Belov, 1978b), $\text{Na}_6\text{V}_{10}\text{O}_{28}.18\text{H}_2\text{O}$ (Durif, Averbuch-Pouchot & Guitel, 1980), $(\text{H}_9\text{C}_7\text{NH})_4\text{H}_2\text{V}_{10}\text{O}_{28}$ (Debaeremaeker, Arrieta & Amigó, 1982), $(\text{H}_3\text{NCH}_2\text{CH}_2\text{-NH})_3\text{V}_{10}\text{O}_{28}.6\text{H}_2\text{O}$ (Shao, Wang, Zhang & Tang, 1983), $\text{Er}_2\text{V}_{10}\text{O}_{28}.25\text{H}_2\text{O}$ (Rivero, Rigotti, Punte & Navaza, 1984), $(\text{C}_7\text{H}_{13}\text{N}_2)_4\text{H}_2\text{V}_{10}\text{O}_{28}.6\text{H}_2\text{O}$ (Shao, Wang, Zhang & Tang, 1984), $(\text{C}_{10}\text{H}_{14}\text{N}_5\text{O}_4)_4\text{H}_2\text{V}_{10}\text{O}_{28}$

O₂₈.11H₂O (Caparelli, Goodgame, Hayman & Skapski, 1986), [(C₆H₅)₄P]₃H₃V₁₀O₂₈.4CH₃CN (Day, Klemperer & Maltbie, 1987), (C₆H₇NH)₃H₃V₁₀O₂₈.H₂O (Santiago, Arnaiz, Lorente, Arrieta & Germain, 1988), (C₇H₉NH)₃H₃V₁₀O₂₈.H₂O (Arrieta, Arnaiz, Lorente, Santiago & Germain, 1988) and (C₅H₅NH)₄(V₁₀O₂₈H₂), (C₆H₇NH)₄(V₁₀O₂₈H₂).2H₂O, (C₇H₉NH)₄(V₁₀O₂₈H₂) and (C₆H₇NH)₆(V₁₀O₂₈) (Arrieta, 1992). As part of a general study of the crystal chemistry of the polyanions of organic bases (Arrieta, Gili & Lorente, 1984; Arrieta, Gili, Lorente & Germain, 1985; Arrieta, 1992, and references therein), we have solved the structure of the title compound.

Experimental. Orange transparent prismatic crystals have been synthesized from acidic aqueous media following the method described by Arrieta, Gili & Lorente (1984) and Arrieta, Gili, Lorente & Germain (1985), at pH ~ 6, (V) ≤ 0.14 M, acidifying with perchloric acid and using the respective organic base. The chemical analysis for V, C, H and N gave the following calculated (experimental) results: V₂O₅ 46.62 (46.52), C 29.70 (29.27), H 3.53 (3.68), N 5.77% (5.74%). Vanadium was determined as V₂O₅ after calcination at 823 K for 2 h. The intensity data were collected from a crystal of approximate size 0.08 × 0.08 × 0.25 mm. The cell parameters were determined by least-squares methods from the setting angles of 25 reflections (18 ≤ 2θ ≤ 30°). 6251 independent reflections were measured with ω-2θ scans [$\Delta 2\theta = (2.5 + 0.5 \tan \theta)^\circ$] up to 2θ = 50° on an Enraf-Nonius CAD-4 diffractometer with graphite-monochromated Mo Kα radiation. One standard reflection (402) was measured every 100 reflections; a linear decay correction was made to allow for an overall 16% decrease in diffracting power caused by decomposition. Lorentz and polarization corrections were applied. Only 1789 reflections [$I \geq 2.5\sigma(I)$] were considered observed and included in the refinement. The index range was *h* - 14 to 14, *k* 0 to 31, *l* 0 to 13. The structure was solved with the PC version of MULTAN88 (Debaerdemaeker, Germain, Main, Tate & Woolfson, 1988) in a standard run. The *E* map showed five V and twelve O atoms of the decavanadate anion. All remaining non-H atoms were found in five subsequent Δρ calculations. The refinement was carried out by least-squares methods using SHELX76 (Sheldrick, 1976). The function Σ(|*F*_o| - |*F*_c|)² with unit weights was minimized. At convergence with isotropic displacement parameters an absorption correction was applied empirically with DIFABS (Walker & Stuart, 1983); min. and max. corrections were 0.640 and 0.814. Anisotropic displacement parameters were used, except for the disordered O atoms of the perchlorate anion and the H atoms. The disordered O atoms of the perchlorate

Table 1. Atomic coordinates (× 10⁴), equivalent isotropic displacement parameters (Å²) and occupancy factors for disordered atoms with e.s.d.'s in parentheses

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> _{eq}	Occupancy factor
V(1)	6217 (1)	278 (1)	575 (1)	2.04	
V(2)	5337 (1)	1135 (1)	-1345 (1)	2.55	
V(3)	3039 (1)	608 (1)	-2426 (1)	2.56	
V(4)	3928 (1)	860 (1)	388 (1)	2.36	
V(5)	4824 (1)	-8 (1)	2340 (1)	2.29	
O(1)	5374 (4)	578 (2)	1586 (4)	2.21	
O(2)	2762 (4)	894 (2)	-1027 (5)	2.84	
O(3)	6447 (4)	-150 (2)	-716 (5)	2.38	
O(4)	7327 (4)	70 (2)	1744 (5)	2.61	
O(5)	4328 (4)	284 (2)	3295 (5)	3.36	
O(6)	4761 (4)	1339 (2)	-102 (5)	2.85	
O(7)	5901 (5)	1639 (2)	-1649 (5)	3.85	
O(8)	1882 (4)	715 (2)	-3496 (5)	3.87	
O(9)	3502 (5)	1162 (2)	1379 (5)	3.49	
O(10)	6195 (4)	-183 (2)	3241 (5)	2.91	
O(11)	6598 (4)	831 (2)	75 (5)	2.64	
O(12)	3925 (4)	1142 (2)	-2542 (5)	2.96	
O(13)	4642 (4)	371 (2)	-830 (5)	2.36	
O(14)	4205 (4)	-646 (2)	2311 (5)	2.92	
N(1)	1736 (6)	-50 (3)	1239 (7)	3.13	
H(1)	2634 (102)	2 (44)	9031 (106)	12.85	
C(12)	1482 (7)	238 (4)	2104 (9)	4.01	
C(13)	553 (8)	127 (4)	2447 (9)	4.18	
C(14)	-112 (8)	-269 (5)	1887 (10)	4.63	
C(15)	150 (7)	-569 (4)	976 (10)	4.34	
C(16)	1085 (7)	-444 (4)	658 (9)	3.44	
C(17)	273 (10)	443 (5)	3453 (10)	7.62	
N(2)	6266 (7)	1443 (3)	2705 (8)	4.16	
H(2)	5683 (99)	1062 (47)	2229 (108)	12.85	
C(22)	5834 (9)	1616 (4)	3564 (10)	4.22	
C(23)	6268 (10)	2053 (4)	4253 (9)	4.65	
C(24)	7166 (10)	2284 (4)	4037 (11)	5.27	
C(25)	7642 (10)	2104 (4)	3140 (11)	5.54	
C(26)	7159 (10)	1672 (4)	2485 (10)	5.04	
C(27)	5740 (11)	2243 (5)	5232 (10)	6.98	
N(3)	7089 (6)	867 (3)	-3628 (7)	3.26	
H(3)	6594 (98)	742 (45)	-2843 (110)	12.85	
C(32)	7899 (7)	1209 (4)	-3129 (8)	3.38	
C(33)	8744 (8)	1293 (3)	-3645 (9)	3.32	
C(34)	8709 (8)	1013 (4)	-4701 (9)	3.90	
C(35)	7859 (8)	666 (4)	-5211 (8)	4.14	
C(36)	7059 (7)	591 (4)	-4630 (8)	3.47	
C(37)	9702 (9)	1659 (4)	-3047 (10)	5.81	
N(4)	2779 (9)	2990 (4)	3440 (10)	5.44	
H(4)	2617 (102)	3445 (5)	33248 (109)	12.85	
C(42)	2185 (10)	2756 (5)	4118 (10)	5.64	
C(43)	2057 (10)	2223 (5)	3965 (12)	6.08	
C(44)	2571 (10)	1979 (4)	3146 (11)	5.24	
C(45)	3158 (10)	2237 (5)	2566 (12)	5.74	
C(46)	3259 (10)	2761 (5)	2703 (11)	5.79	
C(47)	1399 (12)	1941 (6)	4617 (13)	9.36	
O(W1)	4065 (7)	1089 (3)	4905 (6)	8.01	
Cl(1)	9667 (2)	1764 (1)	832 (3)	5.25	
O(1A)	8725 (15)	3347 (7)	4768 (20)	4.98	0.52
O(1B)	9168 (30)	3174 (12)	4486 (26)	4.98	0.25
O(1C)	8584 (24)	3464 (12)	5337 (40)	4.98	0.24
O(2A)	10740 (25)	3353 (20)	5599 (54)	4.98	0.31
O(2B)	9777 (67)	3689 (14)	6681 (37)	4.98	0.22
O(2C)	10352 (49)	3671 (14)	6681 (37)	4.98	0.22
O(2D)	9176 (50)	1311 (13)	1449 (40)	4.98	0.20
O(2E)	10724 (31)	3515 (24)	6014 (58)	4.98	0.29
O(3A)	9772 (35)	2959 (28)	6966 (59)	4.98	0.25
O(3B)	9163 (34)	2883 (11)	6596 (29)	4.98	0.26
O(3C)	9747 (39)	2783 (31)	6529 (87)	4.98	0.26
O(3D)	9570 (58)	1697 (40)	2138 (65)	4.98	0.09
O(3E)	9573 (98)	2396 (54)	935 (148)	4.98	0.07
O(4A)	10468 (37)	2912 (21)	5535 (51)	4.98	0.19
O(4B)	10371 (75)	2706 (32)	6195 (85)	4.98	0.10
O(4C)	9796 (29)	2241 (13)	65 (33)	4.98	0.21
O(4D)	0234 (94)	3171 (42)	4863 (111)	4.98	0.08

anion were introduced into the refinement with an overall isotropic displacement parameter and the occupancy factor of each atom was refined. The H atoms bonded to N were located and those bonded to C were introduced in calculated atomic positions and all were refined with an overall isotropic displacement

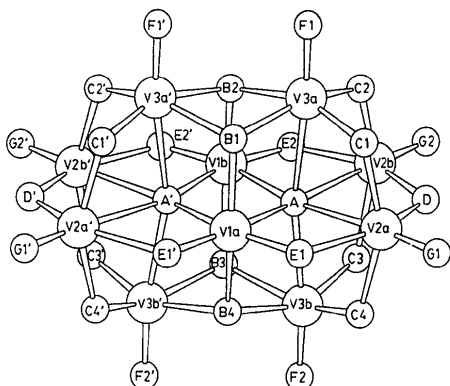


Fig. 1. Drawing of the decavanadate anion showing the different V [V(1)–V(3)] and O (Oa–Og) atom types.

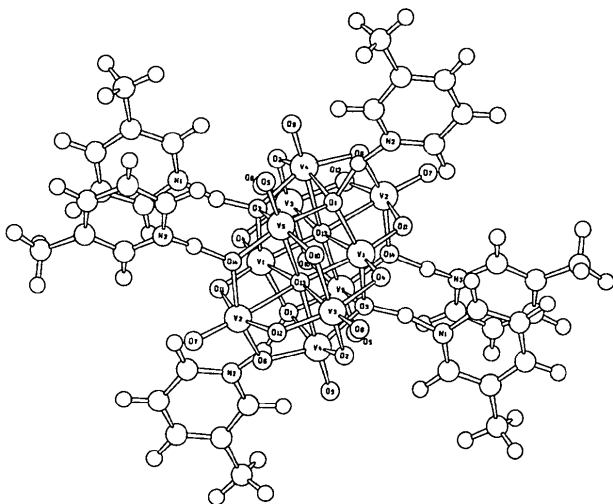


Fig. 2. View of the molecule with the atom-numbering scheme.

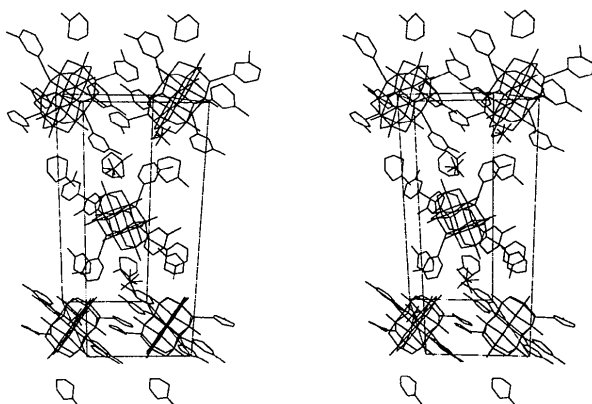


Fig. 3. Stereoscopic view of the molecular packing.

placement parameter. The refinement converged at $R = 0.025$, $wR = 0.025$ and $S = 2.40$, for 174 parameters. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV) and the anomalous-scattering factors from Cromer & Liberman (1970). The max. and min. electron densities in the final $\Delta\rho$ map were 0.20 and -0.23 e \AA^{-3} , respectively, and the max. Δ/σ was 0.028 [for $^x\text{O}(6)$]. Table 1* gives the final atomic parameters with their B_{eq} values (Willis & Pryor, 1975). Fig. 1 shows a perspective view of $V_{10}O_{28}^{6-}$ with the arrangement of different V [V(1)–V(3)] and O (Oa–Og) atom types according to Day, Klemperer & Maltbie (1987). Fig. 2 shows a perspective view of the anion with the atomic numbering. Fig. 3 shows a stereoscopic view of the unit cell. Molecular geometry calculations were performed using *PARST* (Nardelli, 1983). All drawings were made with *SCHAKAL* (Keller, 1988). The infrared spectrum was obtained with a 681 Perkin–Elmer IR spectrophotometer in the $4000\text{--}200 \text{ cm}^{-1}$ range (KBr pellets).

Discussion. In order to investigate the H atoms of the polyanion we have used the empirical bond-length/bond-number calculation in an attempt to find valence-deficient O atoms, with the power function $s = (R/1.791)^{-5.1}$ (Brown, 1981), which relates the V–O distance R and the bond number s . In Table 2, we give the V–O distances and the results of applying the same calculation to the present anion to obtain the Σs values for the O atoms. The Σs values for the O atoms are all in the range 1.62 for O(14) (Oc1) to 2.01 for O(13) (Oa).

The distances given in Table 3 and the angles deposited for the organic cations are normal. The values of χ^2 [$\chi^2 = \Sigma(\Delta/\sigma)^2$, where Δ is the atomic deviation from the calculated mean plane and σ is the standard deviation of Δ] for the organic groups indicate that they are planar for the N(1)–C(16), N(2)–C(25), N(3)–C(35) and N(4)–C(45) rings, these values being 2.24, 1.95, 4.77 and 3.57, respectively. Table 3 also gives the distances and angles of the intermolecular hydrogen bonds between the polyanion and the organic cations. Two 3-methylpyridinium groups [N(1)–C(17) and N(2)–C(27)] are linked to the same O atom-type Ob of the polyanion, by hydrogen bonding. The other group, N(3)–C(37), is bonded by hydrogen bonding to an Oc atom-type. The shortest intermolecular distance between the

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, least-squares planes, interlayer distances, infrared data and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55915 (53 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB1007]

Table 2. Bond lengths (Å) with *e.s.d.*'s of 0.006 Å, and bond numbers, *s*, of the decavanadate anion

V1a	Ob4	Oe1'	Oe1	Oa	Ob1	Oa'	Σ <i>s</i> (V)
V(1)	O(1)	O(4)	O(11)	O(13)	O(3) ⁱ	O(13) ⁱ	
Å	1.940	1.690	1.682	2.136	1.937	2.086	
<i>s</i>	0.67	1.35	1.38	0.41	0.67	0.46	4.93
Σ <i>s</i> (O)	1.77	1.85	1.90	2.01	1.74	2.01	
V2a	Oc4	Og1	Oe1	Od	Oa1	Oc1	
V(2)	O(6)	O(7)	O(11)	O(12)	O(13)	O(14)	
Å	1.854	1.589	2.039	1.862	2.335	1.891	
<i>s</i>	0.84	1.84	0.52	0.82	0.26	0.76	5.03
Σ <i>s</i> (O)	1.73	1.84	1.90	1.75	2.01	1.62	
V2a'	Oe1'	Og1'	Oc4'	Od	Oc1'	Oa'	
V(3)	O(4)	O(8)	O(10)	O(2) ⁱ	O(12) ⁱ	O(13) ⁱ	
Å	2.051	1.604	1.894	1.883	1.816	2.336	
<i>s</i>	0.50	1.75	0.75	0.77	0.93	0.26	4.97
Σ <i>s</i> (O)	1.85	1.75	1.81	1.72	1.75	2.01	
V3b	Ob4	Oc3	Ob3	Oc4	Of2	Oa	
V(4)	O(1)	O(2)	O(3)	O(6)	O(9)	O(13)	
Å	2.032	1.809	1.986	1.831	1.598	2.266	
<i>s</i>	0.53	0.95	0.59	0.89	1.79	0.30	5.05
Σ <i>s</i> (O)	1.77	1.72	1.74	1.73	1.79	2.01	
V3b'	Ob4	Ob3	Of2'	Oc4'	Oa'	Oc3'	
V(5)	O(1)	O(3)	O(5)	O(10)	O(13) ⁱ	O(14) ⁱ	
Å	1.997	2.071	1.604	1.770	2.238	1.843	
<i>s</i>	0.57	0.48	1.75	1.06	0.32	0.86	5.05
Σ <i>s</i> (O)	1.77	1.74	1.75	1.81	2.01	1.62	

Symmetry code: (i) 1 - *x*, -*y*, -*z*.Table 3. Bond distances (Å) of the 3-methylpyridinium cations and hydrogen-bond geometry (Å, °) with *e.s.d.*'s in parentheses

N(1)—C(12)	1.35 (1)	N(3)—C(32)	1.34 (1)	
N(1)—C(16)	1.36 (1)	N(3)—C(36)	1.34 (1)	
C(12)—C(13)	1.37 (2)	C(32)—C(33)	1.38 (2)	
C(13)—C(14)	1.36 (2)	C(33)—C(34)	1.39 (1)	
C(13)—C(17)	1.54 (2)	C(33)—C(37)	1.53 (1)	
C(14)—C(15)	1.42 (2)	C(34)—C(35)	1.39 (1)	
C(15)—C(16)	1.37 (2)	C(35)—C(36)	1.38 (2)	
N(2)—C(22)	1.33 (2)	N(4)—C(42)	1.37 (2)	
N(2)—C(26)	1.36 (2)	N(4)—C(46)	1.32 (2)	
C(22)—C(23)	1.40 (1)	C(42)—C(43)	1.41 (2)	
C(23)—C(24)	1.37 (2)	C(43)—C(44)	1.44 (2)	
C(23)—C(27)	1.54 (2)	C(43)—C(47)	1.47 (2)	
C(24)—C(25)	1.40 (2)	C(44)—C(45)	1.32 (2)	
C(25)—C(26)	1.38 (2)	C(45)—C(46)	1.38 (2)	
D—H...X	D—H	D...X	H...X	D—H...X
N(1)—H(1)...O(3)	1.3 (1)	2.59 (1)	1.3 (1)	167 (10)
N(2)—H(2)...O(1)	1.3 (1)	2.66 (1)	1.5 (1)	159 (11)
N(3)—H(3)...O(14)	1.3 (1)	2.59 (1)	1.4 (1)	162 (11)

fourth group and the polyanion is O(2)(Oc3)—N(4) (*x*, $\frac{1}{2} - y$, $z - \frac{1}{2}$) 2.99 (1) Å. This could indicate that the group is associated with the disordered perchlorate anion. The intermolecular hydrogen-bonding scheme for this compound contrasts with another hexakis centrosymmetric structure (Arrieta, 1992) where two 4-methylpyridinium groups are linked to the same oxygen, atom-type Oc2, and the other group to an Ob4 atom-type.

In order to investigate the different intermolecular bonding schemes we have compared the present

polyanion with the V₁₀O₂₈⁶⁻ polyanion of Na₆(V₁₀O₂₈).18H₂O (Durif, Averbuch-Pouchot & Guitel, 1980) following the work done by Day, Klemperer & Maltbie (1987).

The differences between the distances of the individual bonds with respect to the averaged values within the V₁₀O₂₈⁶⁻ polyanion for the V₄O₄ rings are given in Fig. 4. While these differences are in the range 0.083 to -0.078 Å for hexakis(4-methylpyridinium) decavanadate (Arrieta, 1992), for the present structure they are in the range 0.06 to -0.06 Å, possibly indicating that the H atoms involved in the intermolecular bonding are not linked to the inorganic anion. In both cases the influence of the hydrogen bond is unable to produce a full *trans* bond length alternation similar to that found by Day, Klemperer & Maltbie (1987, and references therein) and by Arrieta (1992) with similar compounds.

The structural effects of protonation and hydrogen bonding at the Ob and Oc atom-types can also be demonstrated by comparing the spacings between approximately planar layers of negatively charged and close-packed O atoms separated by layers of cationic vanadium centers. In our decavanadate anion there are two reference planes formed by six doubly-bridging, two triply-bridging and two six-coordinate O atoms [(Oc4', Ob4, Oc4, Od', Oa', Oa, Od, Oc2', Ob2, Oc2) and (Oc1', Ob1, Oc1, Od', Oa', Oa, Od, Oc3', Ob3, Oc3)] from which distances to other nearly parallel (in the 0.08–1.00° range) layers can be measured. Three types of atom lie in planes above and below the reference planes: two sets of four terminal O atoms [(Og2', Of2', Of2, Og2) and (Og1', Of1', Of1, Og1)], two sets of four doubly-bridging and one triply-bridging O atoms [(Oc3', Oe2', Ob3, Oe2, Oc3) and (Oc1', Oe', Ob1, Oe1, Oc1)] and two sets of five V atoms [(V2b', V3b', V1b, V3b, V2b) and (V2a', V3a', V1a, V3a, V2a)]. The results of our calculations are given in the deposit material. The interplanar spacings in the title compound are very similar to those found in the V₁₀O₂₈⁶⁻ polyanion of Na₆(V₁₀O₂₈).18H₂O (Durif, Averbuch-

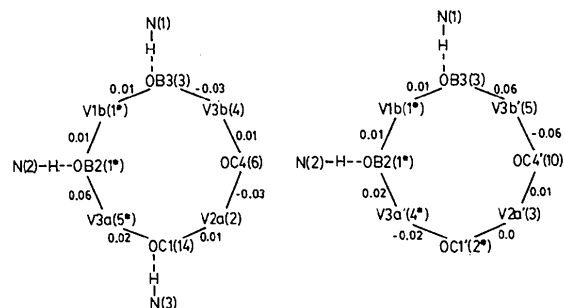


Fig. 4. The V₄O₄ rings of the title compound. Differences between the individual bond distance and the average values for the V₁₀O₂₈⁶⁻ anion are given. Starred atoms (*) are related by 1 - *x*, -*y*, -*z*.

Pouchot & Guitel, 1980) by Day, Klemperer & Maltbie (1987): 1.29, 2.15 and 2.22 Å, which is consistent with the intermolecular hydrogen-bonding scheme.

The water molecule also links two polyanions with distances O(5)(Of2')—O(W1) 2.88 (1) and O(W1)—O(12)(Od) ($x, y, 1+z$) 2.98 (1) Å, the angle O(5)—O(W1)—O(12) being 135 (1)°.

The IR spectrum of this compound is very similar to the triply-protonated decavanadate of the same organic base (Arrieta, Gili & Lorente, 1984), except for three bands at 1100 (very strong), 1045 and 940 cm^{-1} (weak). According to Nakamoto (1986) these bands correspond to the Cl—O stretching frequencies of the perchlorate anion in a lower symmetry than *Td* symmetry.

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Structure of Tetrakis(imidazole)copper(II) Diiodide

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Abstract. $[\text{Cu}(\text{C}_3\text{H}_4\text{N}_2)_4]\text{I}_2$, $M_r = 589.67$, orthorhombic, *Pna*2₁, $a = 14.6703$ (8), $b = 9.4136$ (5), $c = 13.4665$ (8) Å, $V = 1859.7$ Å³, $Z = 4$, $D_x = 2.11$ g cm⁻³, $\lambda(\text{Mo K}\alpha) = 0.71073$ Å, $\mu = 44.73$ cm⁻¹, $F(000) = 1116$, $T = 295$ K, $R = 0.27$, $wR = 0.039$, 1668 unique reflections. The structure consists of discrete $\text{Cu}(\text{imH})_4^{2+}$ square-planar cations, where im is imidazole, and I^- anions which weakly interact with Cu in the fifth and sixth tetragonal positions. The N atoms of the imidazole rings which are not involved in bonding to the copper are

arranged so that the point symmetry could contain a center of inversion. The six-coordinated species does not possess a center of inversion due to the different bonding distances of the two iodides and a moderate torsional twisting of one of the imidazole rings. The absolute configuration of the chiral complex has been determined using the η parameter [Rogers (1981). *Acta Cryst.* **A37**, 734–741].

Introduction. The tetragonal shape of this compound, with the copper–nitrogen and copper–iodide bond