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Tris(*N*,*N*-dimethylethane-1,2-diammonium) decavanadate(V) pentahydrate

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The asymmetric unit of the title compound, $(C_4H_{14}N_2)_3[V_{10}O_{28}]\cdot 5H_2O$, is composed of half of a centrosymmetric $[V_{10}O_{28}]^{6-}$ polyanion, one and a half doubly protonated *N*,*N*-dimethylethane-1,2-diammonium cations (H_2dmen^{2+}) and two and a half solvent water molecules. Disorder is observed in one of the H_2dmen^{2+} cations and also in the water solvent molecules. Hydrogen bonds assemble the compound to form a three-dimensional supramolecular structure.

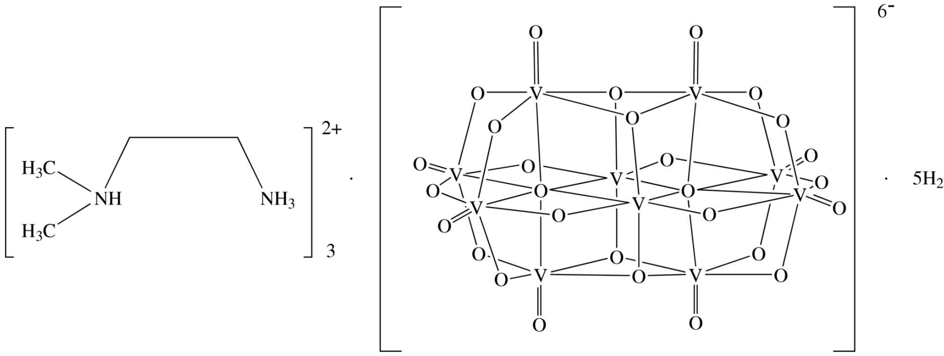
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.009 Å
H-atom completeness 89%
Disorder in solvent or counterion
R factor = 0.039
wR factor = 0.117
Data-to-parameter ratio = 11.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

Among the vanadate oligomers, decavanadate has attracted considerable attention due to its biochemical importance (Aureliano & Gândara, 2005; Tiago *et al.*, 2004). It is considered the major protein-bound species and has also been reported to exhibit high affinity for phosphotransferases (Boyd *et al.*, 1985) and Ca^{2+}-ATPase (Csermely *et al.*, 1985). In order to provide more examples of decavanadate compounds and to understand better the effect of cations on the structures of this kind of compound, we have synthesized and characterized the title new compound, (I).



(I)

The asymmetric unit of compound (I) is composed of half of a centrosymmetric $[V_{10}O_{28}]^{6-}$ polyanion, one and a half doubly protonated *N*,*N*-dimethylethane-1,2-diammonium cations (H_2dmen^{2+}) and two and a half solvent water molecules (Fig. 1). Selected geometric parameters are listed in Table 1. The decavanadate polyanion exhibits the usual structure consisting of a framework of ten V^V atoms in the sites of two almost regular octahedra (Correia *et al.*, 2004; Crans *et al.*, 1994; Evans, 1966).

There are different types of disorder present for the water molecules and H_2dmen^{2+} cations. Water molecule O2*W* is positionally disordered over two positions related by inversion, with site occupation factors of 0.5. A similar disordered

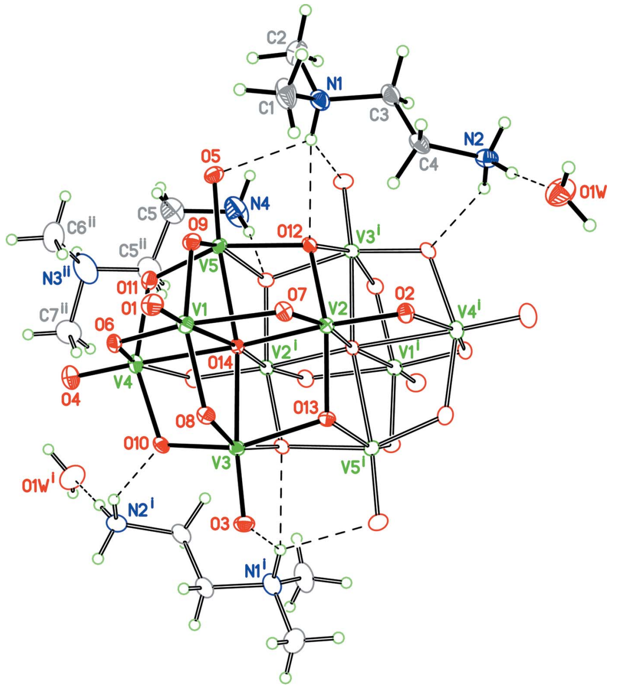


Figure 1

The structure of the ions of (I), with the atomic numbering scheme. Shaded octant ellipsoids (for the asymmetric unit) and outlines (for the symmetry-generated atoms) are drawn at the 30% probability level. Only one disordered component of H_2dmen^{2+} cations is shown. The disordered water molecules O2*W*, O3*W* and O4*W* have been omitted for clarity. Dashed lines indicate hydrogen bonds. [Symmetry codes: (i) $1 - x, 1 - y, 1 - z$; (ii) $1 - x, 1 - y, -z$.]

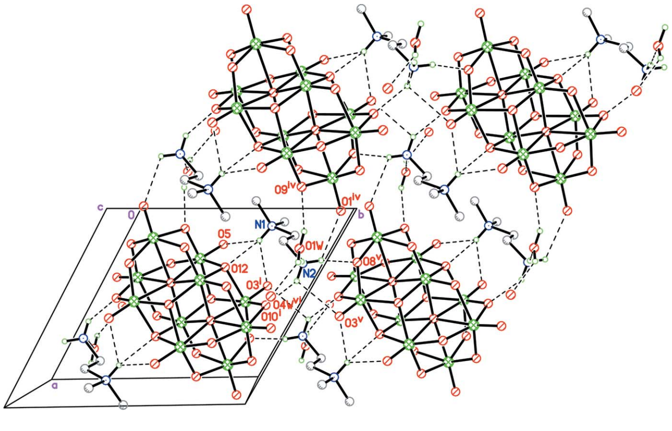


Figure 2

The two-dimensional hydrogen-bonded (dashed lines) network of (I), viewed along the *c* axis. Molecules O2*W* and O3*W* have been omitted for clarity. [Symmetry codes: (i) $1 - x, 1 - y, 1 - z$; (iv) $-x, 1 - y, 1 - z$; (v) $x, 1 + y, z$; (vi) $x, y, 1 + z$.] Only one disorder component of the cation is shown.

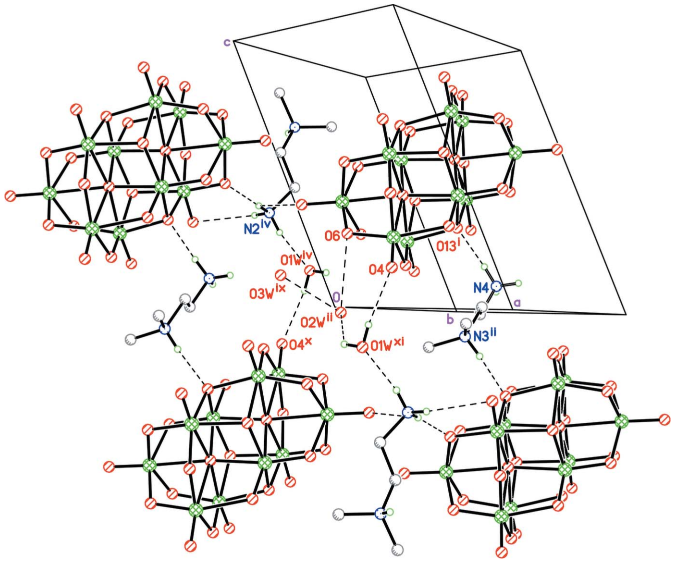


Figure 3

The interlayer hydrogen bonds (dashed lines) involving the H_2dmen^{2+} cations, and molecules O2*W* and O3*W*. [Symmetry codes: (i) $1 - x, 1 - y, 1 - z$; (ii) $1 - x, 1 - y, -z$; (iv) $-x, 1 - y, 1 - z$; (ix) $x - 1, y - 1, z$; (x) $-x, -y, -z$; (xi) $x, y - 1, z - 1$.] Only one disorder component each of the cation, O2*W* and O3*W* is shown.

phenomenon is observed for molecules O3*W* and O4*W*, each of them having 0.5 occupancy. The H_2dmen^{2+} cation is orientationally disordered, with the dimethylammonium group (N3) and the primary ammonium group (N4) being equally disordered over two orientations. All H_2dmen^{2+} cations adopt an anti conformation, with torsion angles N1—C3—C4—N2 = 152.9 (4) and N3—C5—C5^{ii}—N4^{ii} = 180.0 [symmetry code: (ii) 1 − x, 1 − y, −z], and with N···N separations of 3.729 (6) Å (N1···N2) and 3.739 (9) Å (N3···N4^{ii}), respectively.

A three-dimensional supramolecular hydrogen-bonding network is observed in the crystal structure of (I); details are given in Table 2. All the H_2dmen^{2+} cations, and water molecules O1*W* and O2*W*, are involved in hydrogen bonds with either terminal or bridging O atoms in the $[V_{10}O_{28}]^{6-}$ anion, while molecules O3*W* and O4*W* form hydrogen bonds with molecules O2*W* and O1*W* (Figs. 2 and 3).

Experimental

A solution of oxovanadium(IV) oxalate dihydrate (0.191 g, 1 mmol), prepared according to the literature procedure of Sathyanarayana & Patel (1965), in 95% methanol (25 ml) was added in portions to a solution of *N*,*N*-dimethylethane-1,2-diamine (0.088 g, 1 mmol) in methanol (5 ml). The mixture was stirred continuously at 333 K for 10 h and a brown precipitate formed. The precipitate was filtered off, washed sequentially with methanol and diethyl ether several times, and then dissolved in water. Orange crystals of compound (I) (58% yield based on vanadium) suitable for X-ray analysis were obtained from this aqueous solution by slow evaporation at room temperature. Elemental analysis, calculated: C 10.94, H 3.98, N 6.38%; found: C 10.85, H 3.90, N 6.49%.

Crystal data

$(C_4H_{14}N_2)_3[V_{10}O_{28}]\cdot 5H_2O$
$M_r = 1317.99$
Triclinic, $P\bar{1}$
$a = 10.362$ (7) Å
$b = 10.469$ (7) Å
$c = 10.935$ (7) Å
$\alpha = 96.827$ (11)°
$\beta = 106.257$ (10)°
$\gamma = 115.898$ (10)°

$V = 983.7$ (11) Å^3
$Z = 1$
$D_x = 2.215$ Mg m^{-3}
Mo *Kα* radiation
$\mu = 2.37$ mm^{-1}
$T = 298$ (2) K
Block, orange
0.35 × 0.33 × 0.26 mm

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Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{\min} = 0.461$, $T_{\max} = 0.543$
5074 measured reflections
3474 independent reflections
2590 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.019$
$\theta_{\max} = 25.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.117$
$S = 1.04$
3474 reflections
301 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0687P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.61$ e Å^{-3}
$\Delta\rho_{\min} = -0.56$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

V1—O1	1.595 (3)	V4—O4	1.615 (3)
V3—O3	1.616 (3)	V5—O5	1.617 (3)
N1—C3—C4—N2	152.9 (4)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O5	0.91	2.38	3.010 (5)	127
N1—H1···O12	0.91	2.24	3.054 (5)	148
N1—H1···O3$^{\rm i}$	0.91	2.33	2.975 (5)	128
N2—H2*B*···O1*W*	0.89	1.99	2.862 (5)	168
N2—H2*C*···O1$^{\rm ii}$	0.89	2.57	2.997 (5)	110
N2—H2*C*···O8$^{\rm iii}$	0.89	1.98	2.838 (5)	162
N2—H2*A*···O10$^{\rm i}$	0.89	2.07	2.855 (5)	147
N2—H2*A*···O3$^{\rm iii}$	0.89	2.59	3.052 (5)	114
N3—H3···O13$^{\rm i}$	0.91	1.86	2.756 (5)	167
N4—H4*E*···O13$^{\rm i}$	0.89	1.87	2.756 (5)	172
O1*W*—H1*W*···O9$^{\rm ii}$	0.86	2.13	2.74 (1)	128.5
O1*W*—H2*W*···O4*W*$^{\rm iv}$	0.86	1.97	2.69 (1)	140.6
O1*W*—H1*W*···O2*W*$^{\rm v}$	0.86	2.44	3.09 (1)	132.2
O1*W*—H2*W*···O4$^{\rm vi}$	0.86	2.43	3.15 (1)	141.0
O2*W*···O6$^{\rm vii}$			3.02 (2)	
O2*W*···O3*W*$^{\rm viii}$			2.78 (4)	

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $-x, -y+1, -z+1$; (iii) $x, y+1, z$; (iv) $x, y, z+1$; (v) $-x+1, -y+2, -z+1$; (vi) $x, y+1, z+1$; (vii) $-x+1, -y+1, -z$; (viii) $-x+2, -y+2, -z$.

Water molecules O2*W*, O3*W* and O4*W* were treated as positionally disordered, with a constrained site occupancy of 0.5. The H_2dmen^{2+} cations containing atoms N3 and N4 were treated as orientationally disordered. The positions of the H atoms on atoms O2*W*, O3*W* and O4*W* were not determined, but the H atoms of O1*W* were located in a difference Fourier map. The remaining H atoms were placed in calculated positions, with C—H = 0.97 (methylene) or 0.96 Å (methyl) and N—H = 0.89 (primary ammonium) or 0.91 Å (tertiary ammonium). All H atoms were treated as riding, with fixed $U_{\rm iso}$(H) = 0.08 Å^2 (for water molecules) or with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}$(methylene C and tertiary ammonium N) or $1.5U_{\rm eq}$(methyl C and primary ammonium N).

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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