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Pentaaquaoxovanadium(IV) bis(sulfonatosalicylic acid) dihydrate

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Key indicators

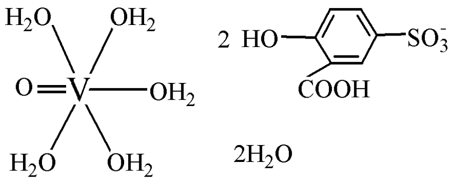
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.033
wR factor = 0.072
Data-to-parameter ratio = 10.5

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

In the title compound, $[VO(H_2O)_5](C_7H_5O_6S)_2 \cdot 2H_2O$, the V^{IV} atom is six-coordinated by O atoms (one vanadyl neighbour and five water molecules) in a distorted octahedral configuration. The complex also contains two uncoordinated sulfosalicylate anions and two uncoordinated water molecules. Numerous O—H···O hydrogen bonds result in the formation of a three-dimensional network.

Comment

Fig. 1 of the title compound, (I), shows that the V^{IV} atom is six-coordinated, in a distorted octahedral configuration, by one vanadyl O atom and five coordinated water O atoms (Table 1). The complex also contains two sulfosalicylate anions and two uncoordinated water molecules.



(I)

The V1—O13 vanadyl bond length of 1.576 (2) Å is typical for a V=O bond (Rath *et al.*, 1997). The O13—V1—O14, O13—V1—O18, O13—V1—O17 and O13—V1—O16 angles are 97.05 (8), 95.67 (9), 95.82 (9) and 101.16 (8)°, respectively, and the O13—V1—O15 angle is 176.44 (8)°; thus, the V-atom coordination is distorted octahedral. The V atom is displaced from the mean plane of its equatorial O-atom neighbours (O14, O16, O17 and O18) by 0.2617 (8) Å (Li *et al.*, 2004).

Many different O—H···O hydrogen bonds are present in (I) (Table 2), involving all types of O atoms in the structure, resulting in a three-dimensional network.

Experimental

Vanadyl sulfate monohydrate (2 mmol, 451 mg) was dissolved in water (10 ml), then sulfosalicylic acid (4 mmol, 1017 mg) in water (10 ml) was added slowly to the solution. The mixture was stirred and heated to reflux for 4 h. Finally, it was cooled to room temperature, filtered and the resulting blue solution held at room temperature for 10 d, whereupon blue crystals suitable for X-ray diffraction were obtained. Analysis calculated for $C_{14}H_{24}O_{20}S_2V$: C 26.78, H 3.83%; found: C 26.97, H 3.63%.

Crystal data

$[VO(H_2O)_5](C_7H_5O_6S)_2 \cdot 2H_2O$
$M_r = 627.39$
Monoclinic, $P2_1/c$
$a = 12.873$ (11) Å
$b = 6.777$ (6) Å
$c = 27.28$ (2) Å
$\beta = 94.393$ (12)°
$V = 2373$ (3) Å^3
$Z = 4$

$D_x = 1.756$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2823 reflections
$\theta = 5.0$–26.3°
$\mu = 0.69$ mm^{-1}
$T = 298$ (2) K
Block, blue
0.22 × 0.19 × 0.11 mm

Data collection

Bruker SMART CCD area detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
$T_{\min} = 0.863$, $T_{\max} = 0.928$
11246 measured reflections

4087 independent reflections
2842 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.031$
$\theta_{\max} = 25.0°$
$h = -11 \rightarrow 15$
$k = -7 \rightarrow 8$
$l = -32 \rightarrow 31$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.072$
$S = 0.90$
4087 reflections
390 parameters

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0294P)^2]$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.37$ e Å^{-3}
$\Delta\rho_{\min} = -0.26$ e Å^{-3}

Table 1
Selected bond distances (Å).

V1—O13	1.576 (2)	V1—O14	2.036 (2)
V1—O17	2.028 (2)	V1—O18	2.049 (2)
V1—O16	2.035 (2)	V1—O15	2.171 (2)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O9	0.82	1.93	2.705 (3)	157
O6—H6···O2	0.82	1.91	2.630 (3)	146
O7—H7···O3	0.82	1.94	2.716 (3)	157
O12—H12···O8	0.82	1.91	2.633 (3)	146
O14—H15···O10$^{\rm i}$	0.896 (10)	1.758 (10)	2.653 (3)	177 (3)
O14—H16···O9$^{\rm ii}$	0.897 (10)	1.840 (13)	2.720 (3)	166 (3)
O15—H17···O20	0.898 (10)	2.034 (15)	2.836 (3)	148 (2)
O15—H18···O19	0.893 (10)	1.926 (12)	2.787 (3)	161 (3)
O16—H19···O5$^{\rm iii}$	0.897 (10)	1.813 (14)	2.661 (3)	157 (2)
O16—H20···O19$^{\rm iii}$	0.905 (10)	1.815 (12)	2.710 (3)	170 (3)
O17—H21···O3	0.881 (10)	1.812 (11)	2.687 (3)	171 (3)
O17—H22···O4$^{\rm iv}$	0.894 (10)	1.735 (11)	2.625 (3)	173 (4)
O18—H23···O20	0.893 (10)	1.773 (11)	2.655 (3)	169 (2)
O18—H24···O11$^{\rm v}$	0.895 (10)	1.821 (10)	2.716 (3)	177 (3)
O19—H25···O5$^{\rm vi}$	0.891 (10)	1.927 (12)	2.812 (3)	172 (3)
O19—H26···O2$^{\rm ii}$	0.894 (10)	1.955 (15)	2.807 (3)	159 (3)
O20—H27···O8	0.896 (10)	1.959 (15)	2.828 (4)	163 (3)
O20—H28···O11$^{\rm vii}$	0.894 (10)	2.069 (16)	2.924 (3)	160 (3)

Symmetry codes: (i) $x, \frac{3}{2} - y, \frac{1}{2} + z$; (ii) $x, \frac{1}{2} - y, \frac{1}{2} + z$; (iii) $1 - x, 1 - y, 1 - z$; (iv) $x, 1 + y, z$; (v) $-x, \frac{1}{2} + y, \frac{1}{2} - z$; (vi) $1 - x, -y, 1 - z$; (vii) $-x, y - \frac{1}{2}, \frac{1}{2} - z$.

Water H atoms were found in difference maps and the O-H distances were restrained to 0.91 Å. All other H atoms were placed in geometrically calculated positions (O—H = 0.82, C—H = 0.93 Å) and allowed to ride on their respective parent atoms, with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$ or $1.5U_{\rm eq}({\rm O})$.

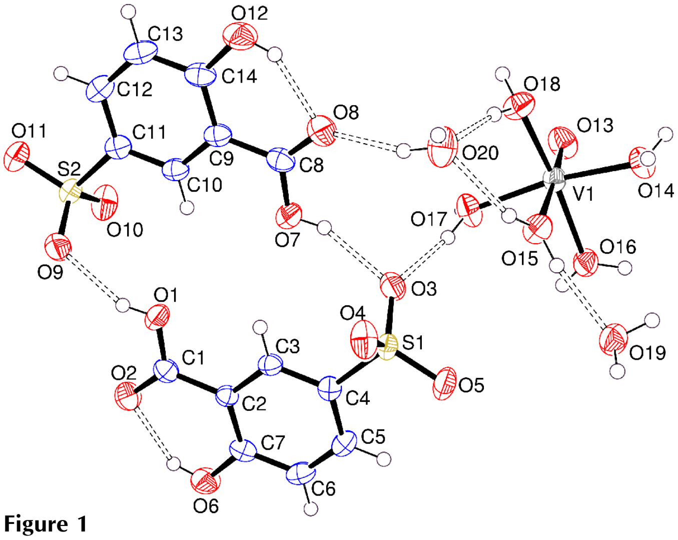


Figure 1
The structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. Hydrogen bonds are indicated by dashed lines.

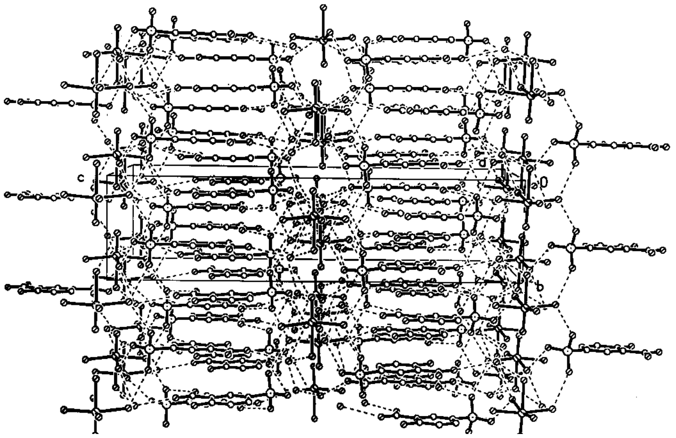


Figure 2
The crystal packing of (I), with the hydrogen-bond interactions shown as dashed lines.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT-Plus* (Bruker, 1999); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

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