$0 \le k \le 20$, $-19 \le l \le 19$; $\omega/2\theta$ scan mode with $\Delta\omega = (1\cdot0 + 0\cdot14\tan\theta)°$] were corrected for Lp. No correction for absorption was applied in view of a < 6% intensity variation of the 360° $\psi$ scan of the close-to-axial reflection $2\bar{4}0$. The structure was solved with standard heavy-atom methods and refined with blocked full-matrix least-squares techniques on $F$ (*SHELX76*; Sheldrick, 1976). H atoms were included on calculated positions (C—H = 1·08 Å) with one overall isotropic thermal parameter [$U = 0\cdot137$ (3) Å$^2$]. Convergence was reached at $R = 0\cdot0638$, $wR = 0\cdot0644$ [$w = 1$, 3204 reflections with $I > 2\cdot5\sigma(I)$, 369 variables, $\langle\Delta/\sigma\rangle = 0\cdot1$, $S = 1\cdot88$]. A final difference synthesis showed no residual density higher than 0·44 e Å$^{-3}$. Scattering factors from Cromer & Mann (1969); anomalous-dispersion factors from Cromer & Liberman (1970). Geometrical calculations and illustrations by programs of the *EUCLID* package (Spek, 1982). Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1.* Fig. 1 shows the atom-numbering scheme and geometry of the molecule. Bond lengths and angles and torsion angles are listed in Table 2.

* Lists of anisotropic thermal parameters, H-atom positions, complete lists of bond distances and angles and observed and calculated structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43762 (29 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

**Related literature.** For the preparation of the compound and a discussion of the results see van der Knaap *et al.* (1983). For a related $\eta^2$-phosphaalkene nickel complex see Cowley, Jones, Stewart, Stuart, Atwood, Hunting & Zhang (1983).

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# Structure of Vanadyl Diformate Monohydrate

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**Abstract.** Aquadiformatooxovanadium(IV), $[VO(HCOO)_2(H_2O)]$, $M_r = 175\cdot0$, orthorhombic, *Pcca*, $a = 8\cdot395$ (2), $b = 7\cdot433$ (1), $c = 8\cdot510$ (1) Å, $V = 531\cdot0$ Å$^3$, $Z = 4$, $D_m = 2\cdot2$, $D_x = 2\cdot19$ g cm$^{-3}$, $\lambda$(Mo $K\bar{\alpha}$) = 0·71073 Å, $\mu = 19\cdot3$ cm$^{-1}$, $F(000) = 348$, room temperature, $R = 0\cdot047$ for 680 unique observed reflections. Distorted $VO(H_2O)(HCOO)_{4/2}$ octahedra with the V atom and *trans* oxo and aqua O atoms on the twofold rotation axes parallel to [010] are linked into polymeric layers perpendicular to that direction by the bidentate formato ligands. The layers are connected in pairs by weak, bifurcated hydrogen bonds between aqua and formato O atoms.

**Experimental.** Blue crystals obtained by hydrothermal synthesis from vanadium(V) oxide and formic acid at 423 K. Crystal 0·2 × 0·2 × 0·1 mm, $D_m$ by flotation, Syntex $P2_1$ diffractometer with graphite monochromator. Lattice constants from setting angles of 15 reflections with $25 < 2\theta < 27°$, intensities by $\omega$ scan with scan rates of 0·7–58·6° min$^{-1}$. Three standard reflections every 100 reflections; only small, random variations. No correction for absorption. 1171 unique reflections measured with $(\sin\theta)/\lambda$ up to 0·807 Å$^{-1}$ and $0 \le h \le 13$, $0 \le k \le 11$, $0 \le l \le 13$; 680 observed reflections with $I > 1\cdot96\sigma(I)$.

Heavy-atom method, full-matrix least-squares refinement based on $F$ magnitudes; observed reflections only, weighted according to $w = [\sigma^2(F) + (0\cdot025|F_o|)^2]^{-1}$. H atoms located by the difference Fourier method and included in the final refinement of 51 parameters (one scale factor and all variable coordinates and thermal

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parameters, isotropic for H and anisotropic for non-H atoms). $R = 0{\cdot}047$, $wR = 0{\cdot}049$, $S = 1{\cdot}173$. All $\Delta/\sigma$ in final cycle 0·00; $\Delta\rho_{min}$ and $\Delta\rho_{max}$ in final difference Fourier synthesis −0·73 and +0·58 e Å$^{-3}$, respectively. Atomic scattering factors for the neutral atoms from Cromer & Waber (1974), with corrections for anomalous dispersion of the V atom from Cromer & Liberman (1974). All calculations performed with the Syntex (1976) *EXTL* program system on a Data General Eclipse S/200 computer.

The atomic parameters are listed in Table 1,* interatomic distances and angles in Table 2. Fig. 1 shows the coordinating ligands around the V atom; Fig. 2 is a stereo plot of the crystal structure.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43722 (5 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic coordinates and (equivalent) isotropic thermal parameters with e.s.d.'s in parentheses*

| | $x$ | $y$ | $z$ | $B_{eq}$†($B_H$)(Å$^2$) |
|---|---|---|---|---|
| V | 0·0 | 0·2271 (1) | 0·25 | 1·38 (1) |
| O(1) | 0·0 | 0·0138 (5) | 0·25 | 3·1 (1) |
| O(2) | 0·0 | 0·5245 (5) | 0·25 | 3·4 (1) |
| O(3) | −0·2891 (2) | 0·2725 (3) | −0·1433 (3) | 2·11 (5) |
| O(4) | −0·1072 (2) | 0·2664 (3) | 0·0402 (3) | 2·44 (6) |
| C | −0·2444 (4) | 0·2342 (4) | −0·0075 (4) | 1·76 (5) |
| H(1) | −0·324 (5) | 0·181 (5) | 0·067 (5) | 3·5 (9) |
| H(2) | 0·072 (7) | 0·598 (7) | 0·191 (7) | 9 (2) |

† $B_{eq} = \frac{1}{3}(B_{11}a^{*2}a^2 + B_{12}a^*b^*ab\cos\gamma + \cdots)$.

Table 2. *Interatomic distances* (Å) *and angles* (°)

| | | | |
|---|---|---|---|
| V–O(1) | 1·586 (4) | O(1)–V–O(2) | 180·0 |
| V–O(2) | 2·210 (4) | O(1)–V–O(3$^{ii}$) | 99·6 (1) |
| V–O(3$^{ii}$) | 2·019 (2) | O(1)–V–O(4) | 98·3 (1) |
| V–O(4) | 2·021 (2) | O(2)–V–O(3$^{ii}$) | 80·4 (1) |
| C–O(3) | 1·248 (4) | O(2)–V–O(4) | 81·7 (1) |
| C–O(4) | 1·245 (4) | O(4)–V–O(3$^{ii}$) | 89·0 (1) |
| C–H(1) | 1·00 (4) | O(4)–V–O(3$^{iii}$) | 88·2 (1) |
| O(2)–H(2) | 0·96 (6) | O(3)–C–O(4) | 122·4 (3) |
| O(2)···O(3$^{iv}$) | 2·998 (2) | O(3)–C–H(1) | 118 (2) |
| O(2)···O(4$^{iv}$) | 3·054 (3) | O(4)–C–H(1) | 118 (2) |
| H(2)···O(3$^{iv}$) | 2·10 (6) | H(2)–O(2)–H(2$^{i}$) | 110 (5) |
| H(2)···O(4$^{iv}$) | 2·23 (4) | O(2)–H(2)···O(3$^{iv}$) | 155 (4) |
| | | O(2)–H(2)···O(4$^{iv}$) | 144 (5) |

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

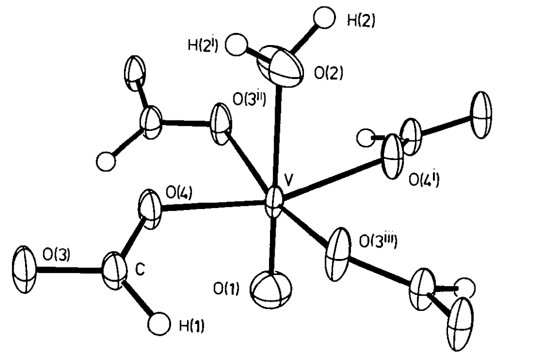


Fig. 1. The distorted $VO(H_2O)(HCOO)_{4/2}$ octahedron with 50% probability ellipsoids (size of H atoms arbitrary; *ORTEP*II, Johnson, 1976).

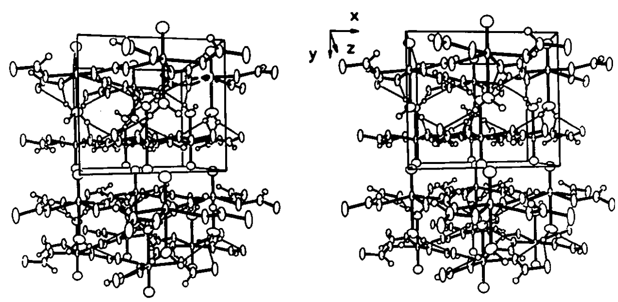


Fig. 2. Stereo plot of the crystal structure with polymeric layers and hydrogen bonding.

**Related literature.** Seifert (1962); Satapathy, Parmar & Sahoo (1963); Kalinnikov, Zelentsov, Volkov & Shostakovskii (1964).


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