

Atomic parameters for the first and second coordination spheres of the cation are given in Table 1, selected bond distances and angles in Table 2.* A perspective view of the cation with atomic numbering is shown in Fig. 1.

Related literature. The hexakis(imidazole)Fe^{II} coordination sphere is also found in [Fe(C₄H₆N₂)₆][Fe₂(CO)₈] and [Fe(C₅H₈N₂)₆][Fe(CO)₄]₂ (Seel & Lehnert, 1980), and in 3Fe(C₃H₃N₂)₂·2C₃H₄N₂ (Lehnert & Seel, 1978). [Fe(C₄H₆N₂)₆](BPh₄)₂·CH₂Cl₂ is apparently unique in containing *only* this coordination sphere. 3Fe(C₃H₃N₂)₂·2C₃H₄N₂ contains both bridging imidazolato and terminal imidazole ligands.

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* A complete list of bond lengths and angles, anisotropic thermal parameters, H-atom parameters, selected least-squares planes and interplane angles, and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51475 (27 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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The Structure of [VCl₃(C₄H₈O)₂(OH)].C₄H₈O

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Abstract. Trichloro(hydroxo)bis(tetrahydrofuran)vanadium–tetrahydrofuran, C₁₂H₂₅Cl₃O₄V, *M_r* = 390.66, triclinic, *P* $\bar{1}$, *a* = 8.191 (15), *b* = 9.038 (5), *c* = 13.816 (12) Å, α = 91.35 (6), β = 106.39 (10), γ = 110.52 (1)°, *V* = 910 (3) Å³, *Z* = 2, *D_x* = 1.42 Mg m⁻³, Mo *K* α radiation, λ = 0.71073 Å, μ = 0.980 mm⁻¹, *F*(000) = 406, *T* = 295 K, *R* = 0.088, *wR* = 0.093 for 2272 unique reflections [*I*₀ ≥ 2.5σ(*I*)]. The V atom is octahedrally coordinated by the O atoms of two *trans* C₄H₈O (thf) ligands [V–O 2.018 (5), 2.043 (5) Å], three Cl atoms [V–Cl 2.322 (2), 2.347 (2) and 2.384 (2) Å] and one OH [V–O 2.046 (6) Å]. The OH ligand is hydrogen bonded to the O of a lattice thf. Both coordinated thf molecules are rotationally disordered about their V–O axes.

Experimental. [VCl₃(thf)₂(OH)].thf crystals were isolated as a byproduct of the preparation of (η-CH₃-

C₅H₄)VCl₂(O) from VCl₂(thf)₂ (Sutin, 1988; Köhler & Prössdorf, 1977; Bottomley & Sutin, 1987). Light orange crystals were obtained. One of these, of dimensions 0.28 × 0.28 × 0.35 mm, was mounted on an Enraf–Nonius CAD-4 diffractometer. Lattice constants were obtained by accurate centering of 25 reflections in the range 15 < 2θ < 30°. Intensities were measured using the θ/2θ scan mode, to a 2θ_{max} of 50° (*h*_{max} 9, *k*_{max} 10, *l*_{max} 15). Three standard reflections were monitored every hour. There was no significant change in their intensity. The intensities of 3193 unique reflections were measured, of which 2272 were judged as observed by the criteria that *I* > 2.5σ(*I*). An empirical absorption correction (*DIFABS*; Walker & Stuart, 1983) was made; relative transmission factors ranged from 0.63 to 1.44. The structure was solved using direct methods (*MULTAN80*; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) and

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Table 1. Atomic coordinates of the non-hydrogen atoms with e.s.d.'s in parentheses

| | x | y | z | B _{iso} * (Å ²) |
|--------|------------|-------------|-------------|--------------------------------------|
| V | 0.4259 (2) | 0.0801 (1) | 0.1997 (1) | 2.95 (6) |
| Cl(1) | 0.3117 (3) | -0.1384 (2) | 0.0675 (1) | 3.99 (10) |
| Cl(2) | 0.5647 (3) | 0.3064 (3) | 0.3235 (2) | 4.59 (11) |
| Cl(3) | 0.1743 (3) | -0.0276 (3) | 0.2576 (2) | 4.27 (11) |
| O(3) | 0.6482 (7) | 0.1700 (6) | 0.1487 (3) | 3.8 (3) |
| H(3) | 0.777 | 0.212 | 0.199 | |
| O(11) | 0.5700 (7) | -0.0324 (6) | 0.2945 (3) | 3.9 (3) |
| C(12) | 0.6899 (7) | -0.1026 (6) | 0.2705 (3) | 3.2 (7) |
| C(13) | 0.7813 (7) | -0.1478 (6) | 0.3681 (3) | 5.4 (11) |
| C(14) | 0.6649 (7) | -0.1482 (6) | 0.4350 (3) | 5.2 (10) |
| C(15) | 0.6038 (7) | -0.0125 (6) | 0.4055 (3) | 2.0 (5) |
| C(12A) | 0.6299 (7) | -0.1549 (6) | 0.2636 (3) | 4.0 (8) |
| C(13A) | 0.7204 (7) | -0.2082 (6) | 0.3583 (3) | 5.3 (11) |
| C(14A) | 0.7263 (7) | -0.0987 (6) | 0.4439 (3) | 7.7 (15) |
| C(15A) | 0.5481 (7) | -0.0685 (6) | 0.3970 (3) | 6.3 (13) |
| O(21) | 0.2979 (7) | 0.1978 (6) | 0.1031 (3) | 4.1 (3) |
| C(22) | 0.2193 (7) | 0.2955 (6) | 0.1431 (3) | 3.5 (7) |
| C(23) | 0.2208 (7) | 0.4242 (6) | 0.0760 (3) | 4.5 (9) |
| C(24) | 0.2036 (7) | 0.3442 (6) | -0.0249 (3) | 4.4 (9) |
| C(25) | 0.3123 (7) | 0.2396 (6) | 0.0051 (3) | 5.4 (11) |
| C(22A) | 0.2573 (7) | 0.1693 (6) | -0.0057 (3) | 4.6 (9) |
| C(23A) | 0.2531 (7) | 0.3227 (6) | -0.0442 (3) | 5.5 (11) |
| C(24A) | 0.1872 (7) | 0.3905 (6) | 0.0312 (3) | 4.6 (9) |
| C(25A) | 0.1610 (7) | 0.2647 (6) | 0.1010 (3) | 6.0 (12) |
| O(41) | 1.0256 (8) | 0.6315 (7) | 0.7417 (4) | 4.4 (3) |
| C(42) | 0.946 (1) | 0.681 (1) | 0.6445 (7) | 5.0 (5) |
| C(43) | 0.802 (1) | 0.537 (1) | 0.5854 (7) | 6.2 (6) |
| C(44) | 0.881 (2) | 0.410 (1) | 0.6168 (9) | 7.8 (8) |
| C(45) | 0.990 (1) | 0.466 (1) | 0.7241 (8) | 5.8 (5) |

* B_{iso} is the mean of the principal axes of the thermal ellipsoid.

Table 2. Important interatomic distances (Å) and angles (°) with e.s.d.'s in parentheses

| | | | |
|---------------|-----------|---------------|-----------|
| V(1)-Cl(1) | 2.384 (2) | V(1)-O(11) | 2.043 (5) |
| V(1)-Cl(2) | 2.347 (2) | V(1)-O(21) | 2.018 (5) |
| V(1)-Cl(3) | 2.322 (3) | O(3)-H(3) | 1.01 |
| V(1)-O(3) | 2.046 (6) | O(41)-H(3) | 1.70 |
| Cl(1)-V-O(3) | 86.9 (2) | Cl(2)-V-Cl(3) | 93.8 (1) |
| Cl(1)-V-O(11) | 90.4 (1) | Cl(3)-V-O(3) | 178.7 (2) |
| Cl(1)-V-O(21) | 89.6 (1) | Cl(3)-V-O(11) | 92.2 (2) |
| Cl(1)-V-Cl(2) | 174.0 (1) | Cl(3)-V-O(21) | 91.5 (2) |
| Cl(1)-V-Cl(3) | 92.2 (1) | O(3)-V-O(11) | 86.8 (2) |
| Cl(2)-V-O(3) | 87.0 (1) | O(3)-V-O(21) | 89.5 (2) |
| Cl(2)-V-O(11) | 89.4 (1) | O(11)-V-O(21) | 176.3 (2) |
| Cl(2)-V-O(21) | 90.2 (1) | | |

refined using *SHELX76* (Sheldrick, 1976) and *NRCVAX* (Gabe, Lee & Le Page, 1985). The function minimized was $\sum w(\Delta F)^2$, where $w = 1/[\sigma(F)^2 + 0.001F^2]$ and σ was obtained from counting statistics. All atoms except H were refined anisotropically; the C₄H₈O ligands were disordered, each occupying two positions having different angles of rotation about the V-O axis. The disorder was modelled using two positions of half-occupancy for the four C atoms and treating each [O($\frac{1}{2}$ C₈H₁₆)] as a rigid body pivoting about O (C-H = 0.96 Å). Full-matrix refinement of 211 parameters gave a final $R = 0.088$, $wR = 0.093$ and R for all reflections = 0.119. The high R value is a

result of the difficulties in dealing with the disorder. The largest Δ/σ was 0.175. The H atom connecting the OH ligand to the lattice thf was observed in a difference synthesis and included as a fixed contribution to F_c . A final difference synthesis had a maximum peak of 0.78 e Å⁻³ close to one of the disordered C atoms of a thf ligand, and a minimum hole of -0.77 e Å⁻³. Scattering factors for neutral atoms, corrected for the real and imaginary parts of the anomalous dispersion, were obtained from *International Tables for X-ray Crystallography* (1974). Positional parameters are listed in Table 1, selected bond lengths and angles in Table 2, and a diagram of the structure is shown in Fig. 1.*

Related literature. The structures of several vanadium complexes with thf ligands have been determined recently: [V₂(μ-Br)₃(Methf)₆]BPh₄ (Methf = 3-methyl-tetrahydrofuran) (Canich, Cotton, Roth & Duraj, 1988); VCl₃(thf)₃ (Cotton *et al.*, 1983); [V₂(μ-Cl)₃(thf)₆]₂Zn₂Cl₆ (Cotton, Duraj & Roth, 1985); [VCl₂(thf)₃]₂(μ-O) (Chandrasekhar & Bird, 1984) and V(thf)₂[ZnCl₃(PPh₃)₂] (Cotton, Duraj, Roth & Schmulbach, 1985).

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* Lists of H-atom positions, anisotropic thermal parameters and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51459 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

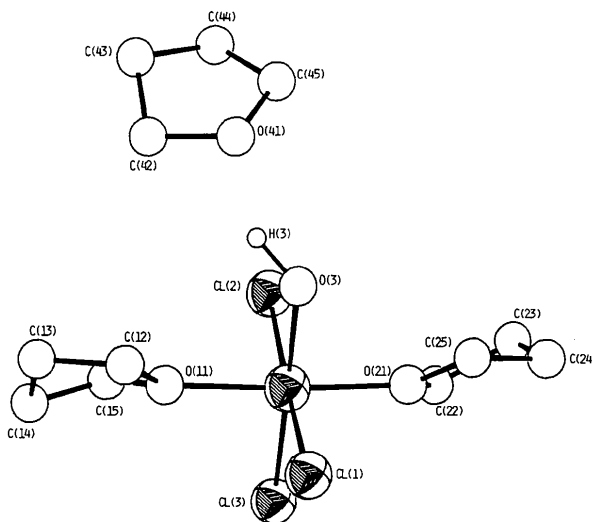


Fig. 1. Structure of [VCl₃(C₄H₈O)₂(OH)].C₄H₈O. Only one of each of the two disordered thf ligands is shown and the H atoms are omitted for clarity.

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1,1,3'-Trimethyl-3'-(trimethylsilyl)perhydroazetidino[1,2-c][1,3]oxazine-5-spiro-2'-oxiran-6-one, a Novel β -Lactam

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Abstract. $C_{13}H_{23}NO_3Si$, $M_r = 269.41$, triclinic, $P\bar{1}$, $a = 10.056$ (6), $b = 10.597$ (5), $c = 7.585$ (5) Å, $\alpha = 102.73$ (5), $\beta = 89.18$ (5), $\gamma = 102.01$ (4)°, $V = 770.7$ (8) Å³, $Z = 2$, $D_x = 1.16$ g cm⁻³, $\lambda(Mo K\alpha) = 0.71073$ Å, $\mu(Mo K\alpha) = 1.56$ cm⁻¹, $F(000) = 292$, $T = 295$ K. Final $R = 0.050$ for 1780 observed reflections. The X-ray structure shows the approximate planarity of the β -lactam ring, the chair conformation of the six-membered ring and the addition of perbenzoic acid oxygen from the least hindered side. The dihedral angle between the β -lactam plane and the three-membered ring is 88.4 (2)°.

Experimental. The title compound (I) was prepared by *m*-chloroperoxybenzoic acid oxidation of the corresponding alkene. Crystals of (I) are colorless rhombohedral plates; crystal dimensions 0.40 × 0.60 × 0.24 mm; unit-cell parameters obtained by least-squares refinement of 15 reflections in the range $10 < 2\theta < 25^\circ$, cell checked for higher symmetry alternatives using *XCELL* (Sheldrick, 1988), automatic Syntex P2₁ diffractometer, graphite-monochromated Mo K α radiation, $\theta/2\theta$ scan mode, variable scan

rate (3.0–14.7° min⁻¹, depending on intensity), 2287 measured reflections, 2026 independent reflections in the range $3 < 2\theta < 45^\circ$, $R_{int} = 0.005$, hkl range $h -10 \rightarrow 10$, $k -11 \rightarrow 11$, $l 0 \rightarrow 8$, 1780 observed reflections remeasured after every 100 reflections did not

Table 1. Fractional atomic coordinates with equivalent isotropic thermal parameters for the non-H atoms (*e.s.d.*'s in parentheses)

| | <i>x</i> | <i>y</i> | <i>z</i> | U_{eq} (Å ²)† |
|-------|------------|-------------|-------------|-----------------------------|
| Si | 0.2790 (1) | 0.2254 (1) | 0.3681 (1) | 0.0481 (2) |
| C(2) | 0.2570 (3) | 0.1319 (3) | 0.1233 (3) | 0.0481 (6) |
| C(3) | 0.2969 (2) | 0.0040 (2) | 0.0580 (3) | 0.0412 (5) |
| C(4) | 0.3201 (2) | -0.1042 (2) | 0.1517 (3) | 0.0400 (5) |
| C(5) | 0.2290 (3) | -0.1383 (3) | 0.3023 (3) | 0.0480 (6) |
| C(6) | 0.2056 (3) | -0.2851 (3) | 0.2902 (4) | 0.0577 (7) |
| O(7) | 0.1461 (2) | -0.3595 (2) | 0.1178 (3) | 0.0577 (5) |
| C(8) | 0.2246 (3) | -0.3392 (3) | -0.0370 (4) | 0.0511 (6) |
| N(9) | 0.2693 (2) | -0.1978 (2) | -0.0196 (3) | 0.0434 (5) |
| C(10) | 0.2437 (3) | -0.1065 (3) | -0.1066 (3) | 0.0511 (7) |
| C(11) | 0.1215 (3) | 0.1819 (3) | 0.4900 (4) | 0.0671 (8) |
| C(12) | 0.3093 (4) | 0.4041 (3) | 0.3615 (5) | 0.0733 (9) |
| C(13) | 0.4282 (3) | 0.1953 (3) | 0.4790 (5) | 0.0739 (9) |
| C(14) | 0.1607 (4) | 0.1691 (3) | 0.0005 (4) | 0.0712 (8) |
| O(15) | 0.3916 (2) | 0.1192 (2) | 0.0420 (3) | 0.0596 (5) |
| C(16) | 0.3490 (3) | -0.4022 (3) | -0.0453 (5) | 0.0745 (9) |
| C(17) | 0.1291 (3) | -0.4020 (3) | -0.1984 (4) | 0.0683 (9) |
| O(18) | 0.1987 (3) | -0.1126 (2) | -0.2578 (3) | 0.0804 (7) |

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† The standard deviations of the U_{eq} 's were calculated according to Schomaker & Marsh (1983).