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Low-temperature oxobis(2,2,6,6-tetramethyl-3,5-heptanedionato)vanadium(IV)

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Key indicators

Single-crystal X-ray study
T = 149 K
Mean σ(C–C) = 0.010 Å
Disorder in main residue
R factor = 0.074
wR factor = 0.187
Data-to-parameter ratio = 16.0

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

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The coordination geometry around the V atom in the title complex, $[VO(C_{11}H_{19}O_2)_2]$, is square pyramidal with the basal plane defined by four O atoms from two chelating β-diketonate ligands and the axial position occupied by an oxo group.

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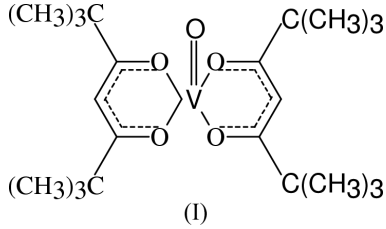
Comment

As part of an ongoing study of precursors for metal-organic chemical vapour deposition (MOCVD) of thin films (Patnaik *et al.*, 1996, Urs *et al.*, 2000), the structure of a vanadium-containing precursor was analyzed to gain an insight into the coordination geometry around the V atom. The coordination geometry in the title compound, (I) (Fig. 1), is square pyramidal with the V atom lying 0.565 (1) Å out of the least-squares plane defined by the four O atoms (O1–O4) derived from two chelating β-diketonate ligands.



(I)

The basal V—O bond lengths vary from 1.953 (4) to 1.972 (4) Å and the apical V═O5 distance is 1.589 (5) Å. The

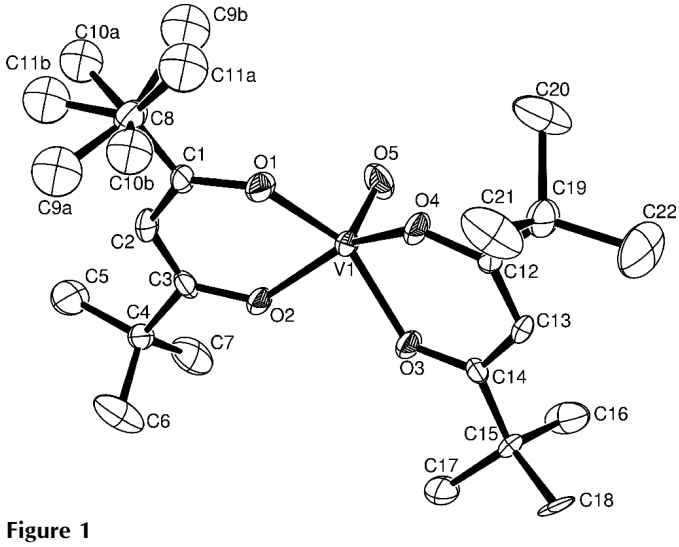


Figure 1
View of (I) showing the disordered *tert*-butyl group. Displacement ellipsoids are drawn at 50% probability level (Johnson, 1965). H atoms have been omitted for clarity and disordered C atoms labelled with *a* have 0.580 (7) site occupancy.

angle between the least-squares planes through the two five-membered chelate rings is 16.2 (2)° (Nardelli, 1995). The conformation of each ring is half chair, with the V atom significantly out of the plane defined by the remaining atoms. The bite O···O distances are shorter than that observed in vanadyl bis(acetylacetonate) (Shuter *et al.*, 1995).

Experimental

About 0.45 g (2.5 mmol) of vanadium pentoxide was reduced to $V(O)^{2+}$ by heating with concentrated sulfuric acid and ethanol using standard procedures, and filtered. The solution then was added to ice-cold dipivaloylmethane and stirred for 3 h. This solution was then neutralized with ethanolic KOH until the pH was 7. The green precipitate was suction filtered, washed with 5 ml portions of ice cold water and recrystallized in chloroform to yield green crystals of the title complex.

Crystal data

$[VO(C_{11}H_{19}O_2)_2]$
M_r = 433.46
Monoclinic, $P2_1$
a = 9.4515 (2) Å
b = 10.7953 (3) Å
c = 12.7494 (4) Å
β = 109.025 (1)°
V = 1229.79 (6) Å^3
Z = 2
D_x = 1.171 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 8–15°
μ = 0.43 mm^{-1}
T = 149 (2) K
Prism, green
0.50 × 0.40 × 0.40 mm

Data collection

Siemens $P4$ diffractometer
ω scans
7897 measured reflections
4217 independent reflections
2751 reflections with $I > 2\sigma(I)$
R_{int} = 0.093
θ_{max} = 26.5°
$h = -11 \rightarrow 11$
$k = -9 \rightarrow 13$
$l = -15 \rightarrow 14$
3 standard reflections
frequency: 120 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.074
$wR(F^2)$ = 0.187
S = 0.92
4217 reflections
263 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0898P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.95 e Å^{-3}
$\Delta\rho_{min}$ = −1.01 e Å^{-3}
Absolute structure: Flack (1983)
Flack parameter = 0.03 (5)

Table 1
Selected geometric parameters (Å, °).

V1—O5	1.588 (5)	V1—O2	1.965 (4)
V1—O1	1.952 (4)	V1—O3	1.972 (4)
V1—O4	1.957 (3)		
O5—V1—O1	107.5 (2)	O4—V1—O2	147.4 (2)
O5—V1—O4	106.2 (2)	O5—V1—O3	106.6 (2)
O1—V1—O4	83.59 (16)	O1—V1—O3	145.88 (18)
O5—V1—O2	106.4 (2)	O4—V1—O3	85.92 (18)
O1—V1—O2	86.81 (16)	O2—V1—O3	84.78 (17)

The tertiary butyl group attached to the C8 atom is disordered with two distinct positions with partial occupancies of 0.58 and 0.42 (from refinement) for each of the C9, C10 and C11 atoms. These atoms were refined with isotropic displacement parameters and H atoms were fixed. Positional parameters of all the other H atoms were calculated geometrically and fixed with $U(H) = 1.2U_{eq}$(parent atom).

Data collection: *XSCANS* (Fait, 1991); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Johnson, 1965; Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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