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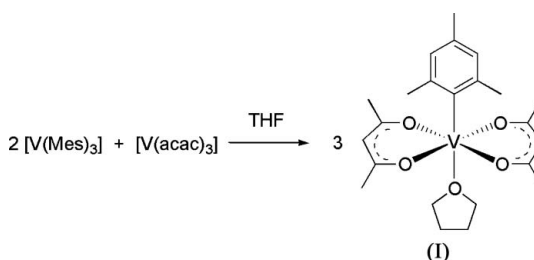
Key indicators

Single-crystal X-ray study
 $T = 293\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$
Disorder in main residue
 R factor = 0.046
 wR factor = 0.132
Data-to-parameter ratio = 19.2For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.*trans*-Bis(acetylacetonato- $\kappa^2\text{O},\text{O}'$)mesityl-
(tetrahydrofuran- κO)vanadium(III)In the title compound, $[\text{V}(\text{C}_9\text{H}_{11})(\text{C}_5\text{H}_7\text{O}_2)_2(\text{C}_4\text{H}_8\text{O})]$, the V atom is octahedrally coordinated by two chelating acetylacetonate (acac) ligands in *trans* positions, one tetrahydrofuran (THF) ligand and one σ -bonded mesityl group.

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Comment

The synthesis of V^{III} and V^{IV} compounds with σ -bonded alkyl or aryl ligands is generally difficult due to the ease of ligand exchange, disproportionation or β -hydride elimination (Imamoto & Gridnev, 2003). Nevertheless, these classes of coordination compounds have generated considerable interest because of their relevance to vanadium-catalysed oxidation and polymerization reactions as well as in the context of model compounds for haloperoxidase activity (Jeong *et al.*, 2005, 2004; Smith & Pecoraro, 2002; Hagen *et al.*, 2001; Gerlach & Arnold, 1997; Crans & Schelble, 1990; Priebisch & Rehder, 1990; Rehder, 1991).The synthesis of $[\text{VMes}(\text{acac})_2(\text{THF})]$ (Mes is mesityl, acac is acetylacetonate and THF is tetrahydrofuran) may be performed either by the reaction of $[\text{VMes}_3]$ with Hacac or by ligand exchange from $[\text{VMes}_3]$ and $[\text{V}(\text{acac})_3]$ (see scheme) (Kreisel & Seidel, 1986). Crystals of the title compound, (I), suitable for X-ray diffraction studies were obtained from a THF solution at low temperature.Fig. 1 shows the molecular structure of the title compound. The V atom is octahedrally surrounded by the two acac ligands, the coordinated THF molecule and the σ -bonded mesityl group. To the best of our knowledge, this is the first structurally characterized mononuclear vanadium compound with at least one σ -bonded alkyl or aryl group and an additional *N*- or *O*-donor set showing an octahedral coordination. The V^{III} compounds investigated so far are either tetrahedral in the case of $[\text{VAr}_3\text{L}]$, $[\text{VAr}_3\text{X}]^+$ or $[\text{VAr}_2\text{XL}]$ derivatives ($L = \text{THF}$: Gambarotta *et al.*, 1984; Vivanco *et al.*, 1993; $X = \text{Cl}$, $L = \text{THF}$: Gibson *et al.*, 1996; $X = \text{RO}$: Rost *et al.*, 1998; Thiele *et al.*, 2002) or square-pyramidal. The latter coordination mode is observed for chelating tetradentate ligands with an *N*- or *N,O*-donor set (Ciurli *et al.*, 1986; Solari *et al.*, 1992; Rosset *et al.*,

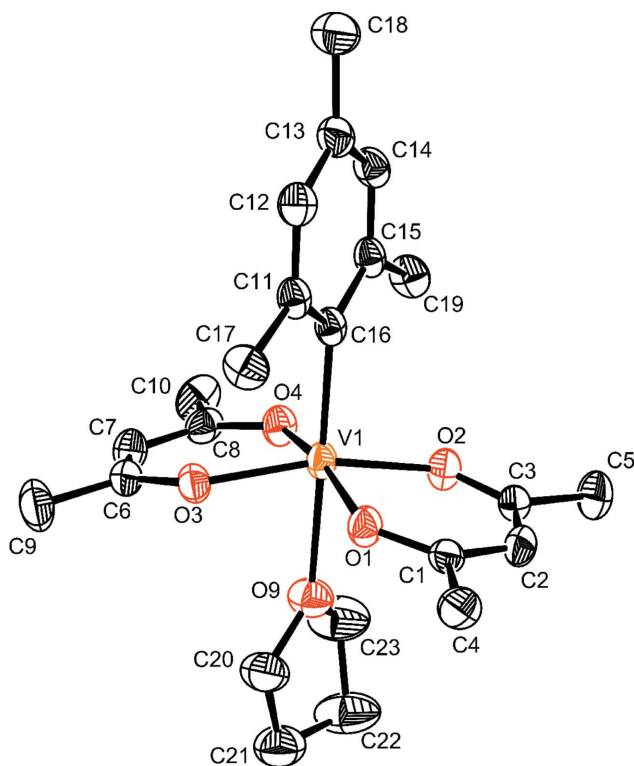


Figure 1
The molecular structure of the title compound. Displacement parameters are drawn at the 30% probability level. H atoms have been omitted for clarity. Only one disorder component is shown.

1990; Vivanco *et al.*, 1993). There is only one dinuclear V^{III} compound in which the vanadium atoms are octahedrally coordinated by a benzyl group and an acetylacetonate ligand with one of the O atoms bridging two metal centres (Gambarotta *et al.*, 1985).

The metal–oxygen bond lengths in (I) show expected values. The V–C bond length of 2.131 (2) Å is also in the typical range observed for mesityl vanadium(III) complexes (see cited literature). The mesityl–V–THF arrangement is almost perfectly linear [C16–V1–O9 = 178.93 (7)°]. The bond angles involving the acac ligands show slight deviations from perfect octahedral geometry due to the steric pressure of the chelating coordination mode of the ligands. Atom V1 is situated 0.2313 (9) Å above the O1/O2/O3/O4 plane. Nevertheless, this deviation is much smaller than those observed in the square-pyramidal V^{III} compounds mentioned above (Ciurli *et al.*, 1986; Solari *et al.*, 1992; Rosset *et al.*, 1990; Vivanco *et al.*, 1993). In these compounds, the tetradentate ligands show a saddle-like conformation, thus preventing the coordination of a sixth ligand. In some of the crystal structures, the presence of THF in the crystal structure is reported but without any binding interaction with the V atoms (Ciurli *et al.*, 1986; Solari *et al.*, 1992).

Experimental

The synthesis of the title compound, together with the corresponding spectroscopic and analytical data, has been published elsewhere

(Kreisel & Seidel, 1986). Crystals of the title compound were isolated from a THF solution after standing at 243 K for several months.

Crystal data

[V(C₉H₁₁)(C₅H₇O₂)₂(C₄H₈O)]
 $M_r = 440.43$
 Triclinic, $P\bar{1}$
 $a = 10.469$ (2) Å
 $b = 10.981$ (2) Å
 $c = 11.811$ (2) Å
 $\alpha = 65.77$ (3)°
 $\beta = 72.77$ (3)°
 $\gamma = 83.20$ (3)°
 $V = 1182.6$ (4) Å³

$Z = 2$
 $D_x = 1.237$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 25 reflections
 $\theta = 18.0$ – 25.2 °
 $\mu = 0.45$ mm⁻¹
 $T = 293$ (2) K
 Block, purple
 $0.2 \times 0.08 \times 0.08$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer
 $\omega/2\theta$ scans
 Absorption correction: ψ scan (North *et al.*, 1968)
 $T_{\min} = 0.823$, $T_{\max} = 0.922$
 5625 measured reflections
 5363 independent reflections
 4116 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.014$
 $\theta_{\text{max}} = 27.4$ °
 $h = -12 \rightarrow 13$
 $k = -12 \rightarrow 14$
 $l = 0 \rightarrow 15$
 3 standard reflections
 frequency: 60 min
 intensity decay: 1.8%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.046$
 $wR(F^2) = 0.132$
 $S = 0.86$
 5363 reflections
 279 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0822P)^2 + 0.5624P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.008$
 $\Delta\rho_{\text{max}} = 0.35$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.33$ e Å⁻³

All H atoms were positioned geometrically and refined as riding on their parent C atoms, with aromatic C–H = 0.93 Å, methylene C–H = 0.97 Å and methyl C–H = 0.96 Å and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{CH}_2, \text{CH})$ or $1.5U_{\text{eq}}(\text{CH}_3)$. One of the C atoms of the THF ligand (C22) is statistically disordered; the site-occupancy factors refined to 0.64 (3) and 0.36 (3), respectively.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *SET4* (de Boer *et al.*, 1984); data reduction: *MolEN* (Enraf–Nonius, 1990); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996) and *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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