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exo-[*N*,*N'*-Bis(3-*tert*-butylsalicylidene)-1,2-diphenyl-(*RS*,*SR*)-1,2-ethanediyldiaminato]oxovanadium(IV)

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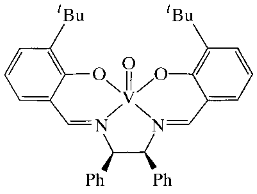
The title mononuclear oxovanadium(IV) complex, [VO($C_{36}H_{38}N_2O_2$)], has a distorted square-pyramidal coordination. The complex was shown to be the *exo* isomer.

Comment

The crystal structure and the thermal isomerization of [VO(3-EtO-sal-*meso*-stien)] have been investigated by the authors (Hoshina *et al.*, 1999), together with the thermal dehydrogenation in the solid state (Hoshina *et al.*, 1998, 2000).

In the crystals of [VO(3-^{t}Bu,5-Me-salen)], (I), the complex has an asymmetric distortion with a ^{t}Bu—C···C—^{t}Bu torsion angle of 25.1° (Cornman *et al.*, 1997). Distortion of the title compound in the crystal is moderate and the C9—C8···C37—C38 torsion angle is 10.1 (3)°°.



(1)

Experimental

To a hot methanol solution (50 ml) of vanadium(IV) oxide sulfate (0.25 g, 1 mmol) were added H_2(3-^{t}Bu-sal-*meso*-stien) (0.53 g, 1 mmol) and pyridine (2 ml), and the mixture was stirred for 1 h at 333 K. The resulting green precipitate was collected by filtration and washed with ether (yield 80%). Found: C 72.34, H 6.52, N 4.72%; calculated for $C_{36}H_{38}N_2O_3V$: C 72.35, H 6.41, N 4.69%. An analysis of the product by high-performance liquid reverse-phase chromatography suggested that only one of two possible geometrical isomers had been obtained. Crystals of the title compound were grown from an acetonitrile solution.

Crystal data

[VO($C_{36}H_{38}N_2O_2$)]
M_r = 597.65
Monoclinic, $C2/c$
a = 32.529 (4) Å
b = 11.877 (5) Å
c = 18.909 (7) Å
β = 121.01 (1)°
V = 6261 (3) Å^3
Z = 8

D_x = 1.268 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 14.8–15.0°
μ = 0.354 mm^{-1}
T = 297 (1) K
Prismatic, green
0.5 × 0.3 × 0.1 mm

Data collection

Rigaku AFC-7*R* diffractometer
θ–2θ scans
Absorption correction: by integration (Coppens *et al.*, 1965)
$T_{\min}$ = 0.866, $T_{\max}$ = 0.979
7427 measured reflections
7203 independent reflections
3823 reflections with $I > 2\sigma(I)$

$R_{\rm int}$ = 0.045
$\theta_{\max}$ = 27.5°
$h = -42 \rightarrow 42$
$k = 0 \rightarrow 15$
$l = -25 \rightarrow 0$
3 standard reflections
every 150 reflections
intensity decay: none

Refinement

Refinement on F^2
$R(F)$ = 0.061
$wR(F^2)$ = 0.219
S = 1.18
7203 reflections
379 parameters

H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.1616P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max}$ = 0.0001
$\Delta\rho_{\max}$ = 0.81 e Å^{-3}
$\Delta\rho_{\min}$ = −0.73 e Å^{-3}

Table 1
Selected geometric parameters (Å).

V1—O2	1.601 (3)	V1—N5	2.077 (5)
V1—O3	1.917 (3)	V1—N6	2.063 (3)
V1—O4	1.918 (4)		

All H-atom positional parameters were calculated geometrically, and fixed with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(parent atom).

Data collection: *MSC*/*AFC Diffractometer Control Software* (Molecular Structure Corporation, 1993); cell refinement: *MSC*/*AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1999); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); software used to prepare material for publication: *TEXSAN*.

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