Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

The *exo* and *endo* isomers of [*N*,*N*′-bis(salicylidene)-1,2-diphenyl-(*RS*,*SR*)-1,2-ethanediyldiaminato]oxovanadium(IV)

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Received 9 March 2000
Accepted 20 March 2000

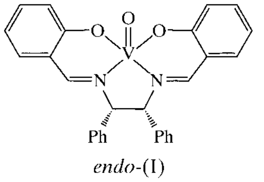
Data validation number: IUC0000085

The title complexes, *exo*- and *endo*-[$VO(C_{28}H_{22}N_2O_2)$], show monomeric structures with a distorted square-pyramidal coordination. The two phenyl groups on the five-membered *N*,*N*′-chelate ring are both on the same side as the oxo ligand for the *exo* isomer and on opposite sides for the *endo* isomer.

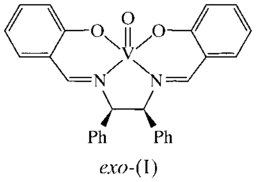
Comment

The crystal structures of the monomeric and polymeric forms of [VO{sal-(*RR*)-stien}] were reported by Nakajima *et al.* (1996). The crystal structures and the thermal isomerization of [VO(3-EtOsal-*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).

The title complex, [VO(sal-*meso*-stien)], (I), did not show thermal isomerization in the solid state below 463 K. The *endo* isomer was more reactive in dehydrogenation upon heating in air than the *exo* isomer.



endo-(I)



exo-(I)

Experimental

The *exo*-[VO(sal-*meso*-stien)] isomer was prepared selectively by the reaction of vanadium(IV) oxide sulfate (0.25 g, 1 mmol) with the Schiff base ligand H_2sal-*meso*-stien (0.42 g, 1 mmol) in methanol containing pyridine (yield 74%). The *endo* isomer was prepared separately by the reaction of vanadium(IV) oxide sulfate (0.25 g, 1 mmol) and H_2sal-*meso*-stien (0.42 g, 1 mmol) in methanol without pyridine (yield 86%). The *endo*-[VO(sal-*meso*-stien)] isomer was eluted more slowly than the *exo* isomer by high-performance liquid reverse-phase chromatography using CH_3CN/H_2O (1:1 *v*/*v*) as eluent.

The *exo* isomer

Crystal data

[$VO(C_{28}H_{22}N_2O_2)$]
$M_r = 485.43$
Monoclinic, $P2_1/n$
$a = 13.278$ (2) Å
$b = 11.340$ (3) Å
$c = 15.940$ (2) Å
$\beta = 101.12$ (1)°
$V = 2355.1$ (7) Å^3
$Z = 4$

$D_x = 1.369$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 14.9$–$15.0°$
$\mu = 0.454$ mm^{-1}
$T = 297$ (1) K
Prismatic, green
0.7 × 0.2 × 0.2 mm

Data collection

Rigaku AFC-7*R* diffractometer
θ–2θ scans
Absorption correction: by integration (Coppens *et al.*, 1965)
$T_{\min} = 0.881$, $T_{\max} = 0.915$
5646 measured reflections
5417 independent reflections
3380 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.016$
$\theta_{\max} = 27.5°$
$h = -17 \rightarrow 0$
$k = 0 \rightarrow 14$
$l = -20 \rightarrow 20$
3 standard reflections every 150 reflections intensity decay: none

Refinement

Refinement on F^2
$R(F) = 0.047$
$wR(F^2) = 0.133$
$S = 1.03$
5417 reflections
307 parameters
H-atom parameters not refined

$w = 1/[\sigma^2(F_o^2) + (0.0505P)^2 + 1.8247P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.81$ e Å^{-3}
$\Delta\rho_{\min} = -0.33$ e Å^{-3}

The *endo* isomer

Crystal data

[$VO(C_{28}H_{22}N_2O_2)$]
$M_r = 485.43$
Orthorhombic, *Pbca*
$a = 17.671$ (6) Å
$b = 21.635$ (7) Å
$c = 12.565$ (4) Å
$V = 4803$ (2) Å^3
$Z = 8$
$D_x = 1.342$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 13.4$–$14.9°$
$\mu = 0.445$ mm^{-1}
$T = 297$ (1) K
Prismatic, green
0.7 × 0.5 × 0.2 mm

Data collection

Rigaku AFC-7*R* diffractometer
θ–2θ scans
Absorption correction: by integration (Coppens *et al.*, 1965)
$T_{\min} = 0.787$, $T_{\max} = 0.912$
5807 measured reflections
5516 independent reflections
4206 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.024$
$\theta_{\max} = 27.5°$
$h = -8 \rightarrow 22$
$k = -10 \rightarrow 28$
$l = -6 \rightarrow 16$
3 standard reflections every 150 reflections intensity decay: none

Refinement

Refinement on F^2
$R(F) = 0.055$
$wR(F^2) = 0.193$
$S = 1.04$
5516 reflections
307 parameters
H-atom parameters not refined

$w = 1/[\sigma^2(F_o^2) + (0.0776P)^2 + 11.0899P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.0001$
$\Delta\rho_{\max} = 0.75$ e Å^{-3}
$\Delta\rho_{\min} = -0.83$ e Å^{-3}

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

For both compounds, 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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Acta Cryst. (2000). C**56**, e188–e189 Gakuse Hoshina *et al.* • *exo*- and *endo*-[VO($C_{28}H_{22}N_2O_2$)] **e189**