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[*N*,*N*′-Bis(3-carboxysalicylidene)-1,2-diphenyl-(*RR*,*SS*)-1,2-ethanediyldiaminato-*O*,*N*,*N*′,*O*′](methanol-*O*)oxovanadium(IV) and [*N*,*N*′-bis(3-carboxysalicylidene)-2,3-dimethyl-2,3-butanediyldiaminato-*O*,*N*,*N*′,*O*′]oxovanadium(IV) monohydrate

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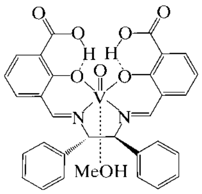
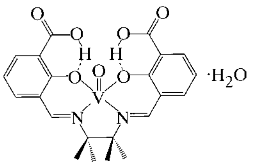
The two title mononuclear oxovanadium (IV) complexes, $[VO(C_{30}H_{22}N_2O_6)(CH_3OH)]$ and $[VO(C_{22}H_{22}N_2O_6)]\cdot H_2O$, respectively, have distorted square-pyramidal coordination and the 3-carboxy groups form intramolecular hydrogen bonds with the coordinated salicyl O atoms. In (I), methanol coordinates to the vanadium atom *trans* with respect to the oxo ligand.

Comment

The structures of dinuclear metal complexes containing the 3-COOH-salen ligand have been reported for homonuclear Cu–Cu (Galy *et al.*, 1979; Gawron *et al.*, 1986) and Ni–Ni (Morgenstern-Badarau *et al.*, 1982) complexes, as well as for heteronuclear Cu–Mg (Beale *et al.*, 1979), Cu–V (Kahn *et al.*, 1982), Cu–Fe (Journaux *et al.*, 1983), Cu–Co (Mikuriya *et al.*, 1978; Galy *et al.*, 1980) and Cu–Ni (Morgenstern-Badarau *et al.*, 1982) complexes, where the inside N_2O_2 coordination site is occupied by the Cu^{II} atom.



(I)



(II)

The structures of mononuclear metal complexes with 3-COOH-salen were also reported for Fe, which occupies an O_4 coordination site (Zarembowitch *et al.*, 1982), and for Co which occupies N_2O_2 coordination site (Charpin *et al.*, 1988).

The structures of [VO{H_2(fsa)$_2$-*rac*-stien}(MeOH)], (I), and [VO{H_2(fsa)$_2$tmen}].H_2O, (II), are reported here [H_4(fsa)$_2$-*rac*-stien is *N*,*N*′-bis(3-carboxysalicylidene)-1,2-diphenyl-1,2-ethanediyldiamine and H_4(fsa)$_2$tmen is *N*,*N*′-bis(3-carboxysalicylidene)-2,3-dimethyl-2,3-butanediyldiamine].

Experimental

3-Aldehydesalicylic acid was prepared according to the literature method of Duff & Bills (1932). H_4(fsa)$_2$-*rac*-stien was prepared by the reaction of 3-aldehydesalicylic acid (2 mmol) with 1,2-(±)-diphenylethylenediamine (1 mmol) in ethanol (yield 94%). The oxovanadium(IV) complex was prepared by the reaction of H_4(fsa)$_2$-*rac*-stien (0.5 mmol) with vanadyl sulfate (0.5 mmol) in methanol containing pyridine (yield 92%). Crystals of (I) were grown from a DMSO solution. For the preparation of (II), H_4(fsa)$_2$tmen was prepared by the reaction of 3-aldehydesalicylic acid (2 mmol) with 1,1,2,2-tetramethyl-1,2-ethylenediamine (1 mmol) in ethanol (yield 88%). The oxovanadium(IV) complex was prepared by the reaction of H_4(fsa)$_2$tmen (0.5 mmol) with vanadyl sulfate (0.5 mmol) in methanol containing pyridine (yield 83%). Crystals of (II) were grown from an acetone solution.

Compound (I)

Crystal data

$[VO(C_{30}H_{22}N_2O_6)(CH_3OH)]$
$M_r = 605.50$
Monoclinic, $P2_1/c$
$a = 9.681$ (2) Å
$b = 18.474$ (2) Å
$c = 15.786$ (2) Å
$\beta = 95.27$ (1)°
$V = 2811.1$ (7) Å^3
$Z = 4$
$D_x = 1.431$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 24 reflections
$\theta = 12.8$–$14.8°$
$\mu = 0.408$ mm^{-1}
$T = 298$ (1) K
Prismatic, red
0.4 × 0.3 × 0.1 mm

Data collection

Rigaku AFC-7*R* diffractometer
θ–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.901$, $T_{max} = 0.960$
8471 measured reflections
8202 independent reflections
4703 reflections with $I > 2\sigma(I)$
$R_{int} = 0.014$
$\theta_{max} = 30°$
$h = 0 \rightarrow 13$
$k = 0 \rightarrow 25$
$l = -22 \rightarrow 22$
3 standard reflections every 150 reflections intensity decay: none

Refinement

Refinement on F^2
$R(F) = 0.056$
$wR(F^2) = 0.167$
$S = 1.309$
8202 reflections
397 parameters
H atoms: see text
$w = 1/[\sigma^2(F_o^2) + (0.05P)^2 + 1P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.50$ e Å^{-3}
$\Delta\rho_{min} = -0.44$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °) for (I).

V1—O2	1.584 (3)	V1—O41	2.368 (3)
V1—O3	1.969 (2)	V1—N9	2.045 (3)
V1—O4	1.948 (2)	V1—N10	2.048 (2)
O2—V1—O41	178.0 (1)	O41—V1—N9	80.35 (10)
O3—V1—O41	78.59 (10)	O41—V1—N10	79.71 (10)
O4—V1—O41	80.10 (9)	V1—O41—C42	126.4 (3)

Table 2
Hydrogen-bonding geometry (Å, °) for (I).

D—H···A	D—H	H···A	D···A	D—H···A
O6—H6···O3	0.97 (4)	1.56 (4)	2.476 (3)	156 (3)
O8—H8···O4	0.98 (3)	1.63 (3)	2.514 (3)	148 (3)
O41—H41···O5^{i}	0.96 (4)	1.78 (2)	2.732 (3)	172 (3)

Symmetry code: (i) $x, \frac{1}{2} - y, z - \frac{1}{2}$.

Compound (II)

Crystal data

$[VO(C_{22}H_{22}N_2O_6)]\cdot H_2O$
$M_r = 495.37$
Triclinic, $P\overline{1}$
$a = 10.508$ (3) Å
$b = 11.728$ (2) Å
$c = 9.726$ (2) Å
$\alpha = 107.66$ (2)°
$\beta = 96.21$ (2)°
$\gamma = 100.45$ (2)°
$V = 1106.1$ (5) Å^3
$Z = 2$
$D_x = 1.487$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 14.9$–15.0°
$\mu = 0.500$ mm^{-1}
$T = 298$ (1) K
Prismatic, green
0.5 × 0.4 × 0.3 mm

Data collection

Rigaku AFC-7R diffractometer
θ–2θ scans
Absorption correction: by integration (Coppens *et al.*, 1965)
$T_{\min} = 0.860$, $T_{\max} = 0.915$
6772 measured reflections
6445 independent reflections
5528 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.085$
$\theta_{\max} = 30°$
$h = 0 \rightarrow 14$
$k = -16 \rightarrow 16$
$l = -13 \rightarrow 13$
3 standard reflections every 150 reflections intensity decay: none

Refinement

Refinement on F^2
$R(F) = 0.039$
$wR(F^2) = 0.116$
$S = 1.053$
6445 reflections
394 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0742P)^2 + 0.2356P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.70$ e Å^{-3}
$\Delta\rho_{\min} = -0.64$ e Å^{-3}

Table 3
Selected geometric parameters (Å) for (II).

V1—O2	1.588 (2)	V1—N9	2.061 (1)
V1—O3	1.952 (1)	V1—N10	2.047 (1)
V1—O4	1.950 (1)		

Table 4
Hydrogen-bonding geometry (Å, °) for (II).

D—H···A	D—H	H···A	D···A	D—H···A
O6—H6···O3	0.77 (4)	1.87 (4)	2.569 (2)	152 (3)
O8—H8···O4	0.91 (4)	1.68 (4)	2.535 (2)	154 (3)
O33—H33A···O5^{i}	0.76 (4)	2.11 (4)	2.886 (3)	167 (3)
O33—H33B···O7^{ii}	0.81 (4)	2.08 (4)	2.888 (3)	175 (4)

Symmetry codes: (i) $x - 1, y, z$; (ii) $2 - x, -y, 1 - z$.

In (I), there is a positional disorder of the C19 and C26 atoms of the five-membered chelate ring moiety. The site-occupancy factors of the C19—C26 and C19*—C26* pairs were estimated to be 81 and 19%, respectively. Namely, the *SS*- and *RR*- complexes share the same site with occupancy factors of 81 and 19%, respectively (and *vice versa* in the centrosymmetric crystal structure). The disorder in the phenyl groups was not taken into account. This caused artificial bond angles, for example, C19*—C20—C21 of 145.4 (6)°. The hydroxyl H atoms were located in difference syntheses and their positional parameters were refined. All the other H-atom positional parameters were calculated geometrically and fixed with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}$(parent atom). The O—H distances are in the range 0.96 (2)–0.98 (3) Å. In (II), all the H atoms were refined isotropically. The O—H and C—H distances are in the ranges 0.76 (4)–0.91 (4) and 0.83 (3)–1.04 (3) Å, respectively. The relatively large $R_{\rm int}$ value of 0.085 might be due to absorption by a thin glass rod.

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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