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Linkage isomeric oxamate chelates: *rac*-bis(ethane-1,2-diamine)-oxamatocobalt(III) bis(trifluoromethanesulfonate) dihydrate and $\Lambda(+)_{578}$-bis(ethane-1,2-diamine)-[oxamato(2−)]cobalt(III) trifluoromethanesulfonate

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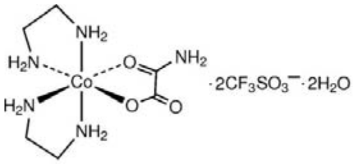
The structures of *rac*-bis(ethane-1,2-diamine)(oxamato-κ^2O^1,O^2)cobalt(III) bis(trifluoromethanesulfonate) dihydrate, $[Co(C_2H_2NO_3)(C_2H_8N_2)_2](CF_3SO_3)_2\cdot 2H_2O$, (I), and $\Lambda(+)_{578}$-bis(ethane-1,2-diamine)[oxamato(2−)-κ^2N,O^1]cobalt(III) trifluoromethanesulfonate, $[Co(C_2HNO_3)(C_2H_8N_2)_2]CF_3SO_3$, (II), are compared. Together, the two complexes constitute the first pair of linkage isomers of bidentate oxamate available for structural comparison.

Comment

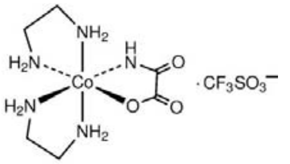
The production of multidimensional magnetic materials through self-assembly of paramagnetic metal ions with small anionic ligands has to a large degree relied on oxalate as the bridging ligand (Pilkington & Decurtins, 2004; Kou *et al.*, 2008, and references therein). However, anionic forms of derivatives such as oxamide ($H_2NCOCONH_2$), dithiooxamide ($H_2NCSCSNH_2$), oxamic acid (H_2NCOCO_2H) and their derivatives are also employed in bridging capacities due to the structural and electronic variation offered by such species (Verdaguer *et al.*, 1985; Kahn, 2000; Cangussu *et al.*, 2008).

The unsymmetrical nature of oxamic acid, whether singly or doubly deprotonated, gives rise to different coordination modes for the oxamate entity in both bridging and nonbridging roles (Novosad *et al.*, 2000; Rodríguez-Martín *et al.*, 2001). For bidentate oxamate in mononuclear systems, both the *O*,*O*′- and *N*,*O*-coordination modes are possible. However, essentially all reported crystal structures of mononuclear oxamate complexes have the ligand *O*,*O*′-bound at a labile metal centre (Braibanti *et al.*, 1971; Pellinghelli *et al.*, 1972; Skoulika *et al.*, 1988*a*,*b*; Veltsistas *et al.*, 1995, 1999; Papadimitriou *et al.*, 1998; Novosad *et al.*, 2000; Rodríguez-Martín *et al.*, 2001; Wang *et al.*, 2003). The sole example of a robust metal centre is the cobalt(III) complex *p*-$[Co(O_2CCONH_2\text{-}O,O')(tren)]Cl_2\cdot 2H_2O$ [tren is tris(2-aminoethyl)amine; Chun *et al.*, 1999].

The two cobalt(III) complexes of this study constitute oxamate linkage isomers covering both coordination modes. However, the two complexes do differ with respect to the level of deprotonation of the oxamate ligand. Thus, the *O*,*O*′-isomer, *rac*-$[Co(O_2CCONH_2\text{-}O,O')(en)_2](O_3SCF_3)_2\cdot 2H_2O$, (I), comprises the oxamate monoanion, whereas the *N*,*O*-isomer, $\Lambda(+)_{578}$-$[Co(O_2CCONH\text{-}N,O)(en)_2](O_3SCF_3)$, (II), comprises the dianion, with the oxamate also deprotonated at the N atom, as expected for an *N*-bound amide (Sigel & Martin, 1982). Reprotonation of (II), presumably at the amide *exo*-O atom, only occurs in strong acids, consistent with a pK_a value of 0.7 for the protonated form, as determined spectrophotometrically for the chloride salt (Grøndahl, Hammershøi *et al.*, 1995).



(I)



(II)

To the best of our knowledge, compound (II) represents the first structurally unambiguous example of an *N*,*O*-linkage isomer of oxamate in a mononuclear complex. Together, complexes (I) and (II) constitute the first pair of linkage isomers of bidentate oxamate available for structural comparison (Tables 1 and 3). The two isomers differ significantly with respect to the bond distances of their oxamate amide segments. These segments are both polarized by coordination to the metal, but to different degrees in each linkage isomer. A valid comparison of these differences should take unbound oxamate as the point of reference. Aakeröy *et al.* (1996) reported the structures of six independent oxamate salts in which the oxamate anion is not coordinated. The C—O and C—N bond distances of the oxamate amide groups of these structures are all very similar (±0.01 Å), averaging 1.236 and 1.320 Å, respectively. Comparing these values with those from the chelated compounds, it transpires that the *O*-bound amide group of (I) has the C1—O1 bond [1.2657 (11) Å] longer by *ca* 0.03 Å and the C1—N1 bond [1.2979 (12) Å] shorter by *ca* 0.02 Å relative to the unbound anion. This is consistent with a relative shift of double-bond character from the C1—O1 bond towards the C1—N1 bond, induced by the metal. The same trend, albeit less pronounced, is evident in (II), with C1—O1 and C1—N1 bond lengths of 1.2578 (9) and 1.3139 (9) Å, respectively. In (II), the metal centre has formally replaced a H atom at the amidic N atom. The smaller charge-to-radius ratio of the cobalt(III) centre renders it less polarizing than a H atom (Dixon & Sargeson, 1993), and the relative effect of such a H^+/Co^{III} replacement would be to release electron density into the N—C bond, albeit on a minor scale, as judged from the small bond-length changes compared

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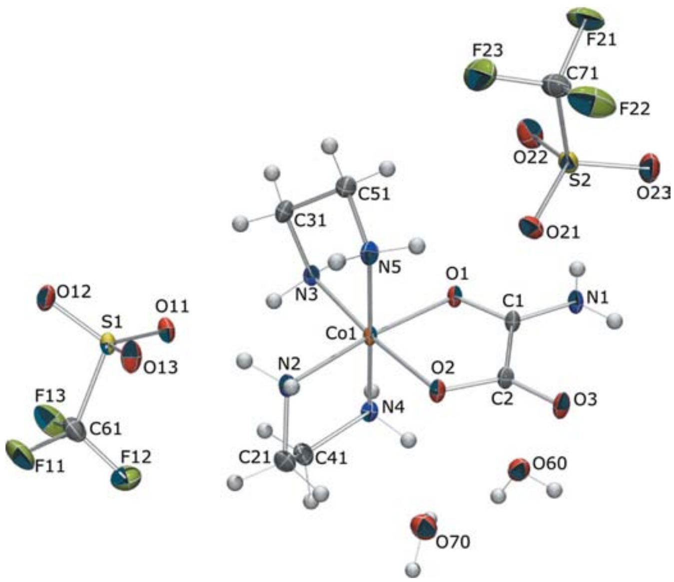

Figure 1
A view of the molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

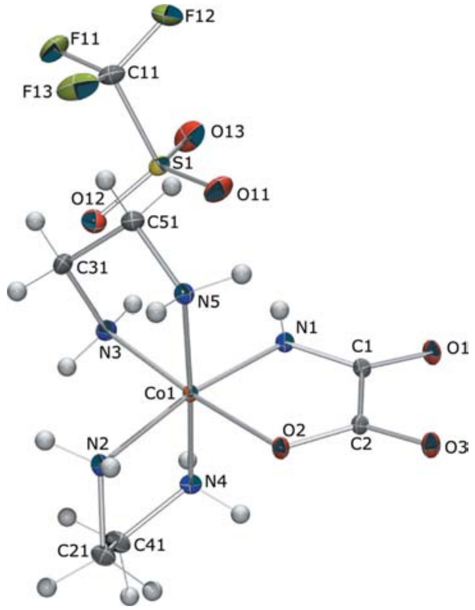

Figure 2
A view of the molecular structure of (II), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

with the unbound anion. The Co1—N1(amide) bond of 1.9039 (7) Å is the shortest such bond in (II), with the other Co1—N(primary amine) distances ranging from 1.9444 (7) to 1.9699 (6) Å. The structure of the closely related 2-thiooxamate complex, $[Co(O_2CCSNH\text{-}N,O)(en)_2](O_3SCF_3)\cdot H_2O$ (Grøndahl, Hammershøi & Larsen, 1995) shows the same features, with thioamide Co—N and C—N bond distances of 1.896 (2) and 1.310 (4) Å, respectively, which closely match those of (II). The Co1—N1 distance in (II) is significantly shorter than the corresponding distance of 1.960 (2) Å in the *N*-formyloxamate complex, $\Lambda(+)_{578}$-$[Co(O_2CCONCHO\text{-}N,O)(en)_2]ClO_4$ (Grøndahl, Hammershøi *et al.*, 1995). Thus, the introduction of a formyl group has a dramatic effect on the Co—N(amide) distance. A parallel trend holds for 2-iminoacetate *versus* 2-methyliminoacetate as ligand. Thus, the introduction of a methyl group at the imine N atom increases the Co—N(imine) distance from 1.904 (4) Å in $[Co(O_2C\text{-}C{=}NH)(en)_2]Br_2\cdot H_2O$ to 1.951 (2) Å in $[Co(O_2CC{=}NCH_3)(en)_2]S_2O_6\cdot 1.5H_2O$ (Bendahl *et al.*, 2002). The oxamate and oxamate-to-metal bond parameters of (I) are almost identical to those of the corresponding tren complex, *p*-$[Co(O_2CCONH_2\text{-}O,O')(tren)]Cl_2\cdot 2H_2O$.

The study of Chun *et al.* (1999) was initiated against the background that certain racemic halide salts of the $[Co(ox)(en)_2]^+$ cation (ox is oxalate) were known to display conglomerate crystallization (spontaneous resolution) from aqueous solution (Yamanari *et al.*, 1973). Therefore, it was of interest to detect whether this property is retained if the oxalate ligand is replaced by structurally related ligands, *e.g.* oxamate. Indeed, the racemic chloride salt of (II), *rac*-$[Co(O_2CCONH\text{-}N,O)(en)_2]Cl\cdot H_2O$, has been demonstrated to display conglomerate crystallization from a comparison of the X-ray powder diffraction pattern of the racemate with that of one enantiomer, $\Lambda(+)_{578}$-$[Co(O_2CCONH\text{-}N,O)(en)_2]Cl\cdot H_2O$ (Grøndahl, Hammershøi *et al.*, 1995). These patterns are identical, implying that the racemate must crystallize in the same noncentrosymmetric space group as the enantiomer (Galsbøl *et al.*, 1978).

The structures of both (I) and (III) are characterized by extensive three-dimensional hydrogen-bonding networks; for details, see Tables 2 and 4.

Experimental

The *O*,*O*′-isomer, (I), was synthesized here anew. Earlier attempts at producing this complex from *trans*-$[CoCl_2(en)_2]Cl$ by reaction with sodium oxamate in water only resulted in the oxalate complex, $[Co(ox)(en)_2]Cl\cdot 4H_2O$ (Chun *et al.*, 1999), evidently due to adverse hydrolysis of oxamate under the aqueous conditions. This difficulty was circumvented here by allowing ethyl oxamate in acetone to replace the coordinated trifluoromethanesulfonate ions in *cis*-$[Co(O_3SCF_3)_2(en)_2](O_3SCF_3)$ (Barfod *et al.*, 2005). During aqueous work-up, the ester part of ethyl oxamate, once coordinated, readily hydrolyzes (Browne *et al.*, 2000). Thus, for the synthesis of (I), a solution of *cis*-$[Co(O_3SCF_3)_2(en)_2](O_3SCF_3)$ (4.82 g, 10.0 mmol) and ethyl oxamate (2.34 g, 20.0 mmol) in acetone (20 ml) was stirred at 313–318 K for 0.5 h. The solvent was then removed by rotary evaporation, the resulting residue taken up in water (15 ml) and the solution extracted with diethyl ether (2 × 20 ml). The aqueous phase was concentrated almost to dryness and the resulting residue redissolved in water (*ca* 5 ml). Gradual addition of LiCl (1.7 g) and EtOH (10 ml), followed by cooling in ice, deposited the chloride salt, *rac*-$[Co(O_2CCONH_2)(en)_2]Cl_2\cdot 2H_2O$, as red crystals (1.81 g). These were recrystallized from hot water (5.5 ml) and EtOH (10 ml) (1.64 g, 44%). Large ruby-coloured crystals of the trifluoromethanesulfonate salt, (I), suitable for X-ray crystallography were grown from an aqueous solution of the chloride salt to which was added excess $NaO_3SCF_3\cdot H_2O$. The *N*,*O*-isomer, (II), was obtained from the known

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

Co1—O1	1.9150 (7)	Co1—N5	1.9575 (9)
Co1—O2	1.9208 (7)	C1—O1	1.2657 (11)
Co1—N2	1.9280 (8)	C1—N1	1.2979 (12)
Co1—N3	1.9389 (8)	C2—O2	1.2841 (11)
Co1—N4	1.9510 (9)	C2—O3	1.2268 (11)
O1—C1—C2—O2	0.78 (13)		

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

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
N1—H1*A*⋯O60^{i}	0.865 (13)	1.954 (13)	2.8181 (12)	176.3 (16)
N1—H1*B*⋯O3^{ii}	0.864 (13)	2.058 (14)	2.8281 (11)	148.0 (15)
N2—H2*A*⋯O13	0.92	2.05	2.8890 (12)	150
N2—H2*B*⋯O11^{iii}	0.92	1.98	2.8947 (12)	170
N3—H3*A*⋯O11	0.92	2.15	3.0225 (11)	158
N3—H3*B*⋯O21^{iv}	0.92	2.20	2.9966 (12)	145
N4—H4*A*⋯O60	0.92	2.20	3.0504 (12)	153
N4—H4*B*⋯O3^{iv}	0.92	2.13	3.0313 (12)	167
N5—H5*A*⋯O21	0.92	2.11	3.0301 (12)	175
N5—H5*B*⋯O12^{v}	0.92	2.14	2.9543 (12)	147
O60—H60*A*⋯O70	0.803 (14)	1.950 (15)	2.7067 (14)	156.6 (19)
O60—H60*B*⋯O23^{ii}	0.809 (14)	2.125 (15)	2.9280 (13)	171.6 (19)
O70—H70*A*⋯O22^{vi}	0.867 (15)	2.077 (16)	2.8984 (14)	158 (2)
O70—H70*B*⋯O23^{i}	0.878 (15)	2.143 (16)	2.9730 (14)	157.5 (19)

Symmetry codes: (i) $-x+1, -y+1, -z$; (ii) $-x+2, -y+1, -z$; (iii) $x+1, y, z$; (iv) $x-1, y, z$; (v) $-x+1, -y+1, -z+1$; (vi) $x, y+1, z$.

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

Co1—N1	1.9039 (7)	Co1—N5	1.9689 (7)
Co1—O2	1.9185 (6)	C1—O1	1.2578 (9)
Co1—N2	1.9699 (6)	C1—N1	1.3139 (9)
Co1—N3	1.9444 (7)	C2—O2	1.2896 (9)
Co1—N4	1.9558 (7)	C2—O3	1.2340 (9)
N1—C1—C2—O2	10.18 (9)		

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

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
N1—H1⋯O11	0.856 (15)	2.421 (15)	3.1733 (10)	147.1 (12)
N2—H2*A*⋯O1^{i}	0.92	2.19	3.0029 (9)	148
N2—H2*B*⋯O3^{ii}	0.92	2.03	2.9202 (9)	161
N3—H3*A*⋯O11	0.92	2.48	3.2955 (12)	148
N3—H3*A*⋯O12	0.92	2.53	3.2660 (11)	138
N3—H3*B*⋯O1^{iii}	0.92	2.52	3.1887 (10)	130
N3—H3*B*⋯O2^{ii}	0.92	2.53	3.2160 (9)	132
N4—H4*A*⋯O3^{iii}	0.92	2.07	2.9236 (9)	155
N4—H4*B*⋯O12^{iv}	0.92	2.22	3.1282 (11)	168
N5—H5*A*⋯O1^{i}	0.92	2.00	2.8809 (9)	160
N5—H5*B*⋯F12^{v}	0.92	2.51	3.4117 (11)	166

Symmetry codes: (i) $x-1, y, z$; (ii) $-x, y+\frac{1}{2}, -z+1$; (iii) $-x+1, y+\frac{1}{2}, -z+1$; (iv) $-x+1, y-\frac{1}{2}, -z+1$; (v) $-x+1, y-\frac{1}{2}, -z+2$.

chloride salt (Grøndahl, Hammershøi *et al.*, 1995). A solution of $\Lambda(+)_{578}$-[Co(O$_2$CCONH-*N*,*O*)(en)$_2$]Cl·H$_2$O (0.25 g) and KO$_3$SCF$_3$ (0.50 g) in hot water (5 ml, 323 K) was cooled slowly to 298 K, depositing orange–red crystals (0.28 g). These were recrystallized from boiling water (15 ml) to produce crystals of (II) (0.10 g) of crystallographic quality.

Compound (I)

Crystal data

[Co(C$_2$H$_2$NO$_3$)(C$_2$H$_8$N$_2$)$_2$]-(CF$_3$SO$_3$)$_2$·2H$_2$O
$M_r = 601.36$
Triclinic, $P\overline{1}$
$a = 6.2621$ (9) Å
$b = 10.6826$ (7) Å
$c = 16.4111$ (6) Å
$\alpha = 92.214$ (5)°
$\beta = 94.350$ (5)°
$\gamma = 98.257$ (7)°
$V = 1081.95$ (18) Å^3
$Z = 2$
Mo *Kα* radiation
$\mu = 1.10$ mm^{-1}
$T = 122$ K
0.35 × 0.23 × 0.06 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Absorption correction: integration (Gaussian; Coppens, 1970)
$T_{\min} = 0.693$, $T_{\max} = 0.933$
68590 measured reflections
13446 independent reflections
10801 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.044$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.079$
$S = 1.09$
13446 reflections
316 parameters
6 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max} = 0.70$ e Å^{-3}
$\Delta\rho_{\min} = -0.70$ e Å^{-3}

Compound (II)

Crystal data

[Co(C$_2$HNO$_3$)(C$_2$H$_8$N$_2$)$_2$](CF$_3$SO$_3$)
$M_r = 415.25$
Monoclinic, $P2_1$
$a = 6.6398$ (4) Å
$b = 11.6663$ (8) Å
$c = 9.2846$ (10) Å
$\beta = 96.517$ (11)°
$V = 714.55$ (10) Å^3
$Z = 2$
Mo *Kα* radiation
$\mu = 1.42$ mm^{-1}
$T = 122$ K
0.20 × 0.13 × 0.06 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Absorption correction: integration (Gaussian; Coppens, 1970)
$T_{\min} = 0.714$, $T_{\max} = 0.919$
35436 measured reflections
8895 independent reflections
8611 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.033$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.019$
$wR(F^2) = 0.047$
$S = 1.06$
8895 reflections
211 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max} = 0.48$ e Å^{-3}
$\Delta\rho_{\min} = -0.75$ e Å^{-3}
Absolute structure: Flack (1983), with 4283 Friedel pairs
Flack parameter: 0.000 (4)

The space groups were determined from analysis of the systematically absent reflections. H atoms were found in a difference Fourier map and included in the refinement as constrained idealized H atoms riding on the parent atom, with C—H = 0.99 Å and N—H = 0.92 Å. In the final refinements of (I) and (II), the positions of the H atoms at the oxamate N atom were refined, with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(N), and in the case of (I) with an N—H distance restraint of 0.88 Å. The positions of the H atoms of the solvent water molecules in (I) were also refined, with a distance restraint (O—H = 0.82 Å) and with $U_{iso}(H) = 1.5U_{eq}(O)$.

For both compounds, data collection: *COLLECT* (Nonius, 1999); cell refinement: *COLLECT*; data reduction: *EVALCCD* (Duisenberg *et al.*, 2003); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: MX3011). Services for accessing these data are described at the back of the journal.

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