ω scans
Absorption correction:
empirical (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.562$, $T_{max} = 0.862$
16 882 measured reflections
6373 independent reflections
$R_{int} = 0.038$
$\theta_{max} = 23.26°$
$h = -16 \rightarrow 15$
$k = 0 \rightarrow 14$
$l = 0 \rightarrow 27$
Intensity variation: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.135$
$S = 1.011$
6373 reflections
384 parameters
H atoms not refined
$w = 1/[\sigma^2(F_o^2) + (0.0512P)^2 + 11.5194P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.817$ e Å^{-3}
$\Delta\rho_{min} = -0.622$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Mo1—C6	1.932 (7)	Mo2—C2	2.009 (8)
Mo1—C4	1.995 (8)	Mo2—C3	2.013 (8)
Mo1—C5	1.998 (7)	Mo2—O7	2.234 (4)
Mo1—O8	2.215 (4)	Mo2—S2	2.460 (2)
Mo1—S1	2.477 (2)	Mo2—S1	2.464 (2)
Mo1—S2	2.482 (2)	O7—C31	1.257 (7)
Mo1—Mo2	2.9071 (8)	O8—C31	1.260 (7)
Mo2—C1	1.936 (8)		
S1—Mo1—S2	107.36 (6)	Mo2—S1—Mo1	72.08 (5)
O8—Mo1—Mo2	81.13 (11)	Mo2—S2—Mo1	72.06 (5)
S2—Mo2—S1	108.45 (6)	O7—C31—O8	125.1 (6)
O7—Mo2—Mo1	81.45 (11)		

Data were collected over a hemisphere of reciprocal space by a combination of three sets of exposures. Each set had a different φ angle for the crystal and each exposure of 10 s covered 0.3° in ω. The crystal-to-detector distance was 4.95 cm. Coverage of the unique set was over 99% complete to at least 23° in θ. Crystal decay was monitored by measurement of duplicate reflections. H atoms were all located theoretically and were not refined. The lengths of some C—C bonds in the cation are shorter than is reasonable; this is due to high thermal motion and disorder.

Data collection: *SMART* (Siemens, 1996). Cell refinement: *SMART* and *SAINT* (Siemens, 1994*a*). Data reduction: *XPREP* in *SHELXTL* (Siemens, 1994*b*). Program(s) used to solve structure: *SHELXTL*. Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: Siemens *SHELXTL*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: TA1216). Services for accessing these data are described at the back of the journal.

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trans-Diaquabis(quinoline-2-carboxylato-*N*,*O*)cobalt(II)–water–ethanol (1/2/2)

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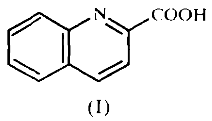
Abstract

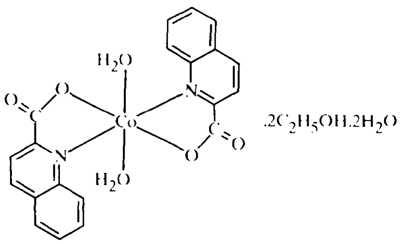
The title compound, $[Co(C_{10}H_6NO_2)_2(H_2O)_2]\cdot 2H_2O\cdot 2C_2H_5OH$, contains a six-coordinate Co^{II} ion at a center of symmetry. The Co^{II} ion displays distorted octahedral coordination geometry defined by the two quinoline N atoms, two O atoms of the carboxylate groups and two O atoms of the water molecules. All of the corresponding pairs of ligand atoms lie in *trans* positions with respect to each other. Molecules are linked together by an intermolecular hydrogen-bonding network involving the uncoordinated water and ethanol molecules.

Comment

2-Quinolinecarboxylic acid, (I), is an intermediate tryptophan metabolite and is known to chelate transition metal ions (Martell & Smith, 1974). Crystal structures of complexes of 2-quinolinecarboxylic acid have been determined for several metal ions, including Cu^{II} (Haendler, 1986), Mn^{II} (Haendler, 1996; Okabe & Koizumi, 1997) and Fe^{II} (Okabe & Makino, 1998). We have carried out the structural analysis of the Co^{II} complex, (II), and report the results here.



(I)



(II)

The molecular structure of the title complex is shown in Fig. 1. The Co^{II} complex is hexacoordinate, with the Co^{II} ion at a center of symmetry. The two 2-quinolinecarboxylate ligands chelate the metal through the N atom and one O atom to form the equatorial plane. Two water molecules complete the octahedron at the axial positions. The six-coordinate geometry found in this study is similar to that of the Mn^{II} (Haendler, 1996; Okabe & Koizumi, 1997) and Fe^{II} (Okabe & Makino, 1998) complexes, but is different to that of the pentacoordinate Cu^{II} complex (Haendler, 1986).

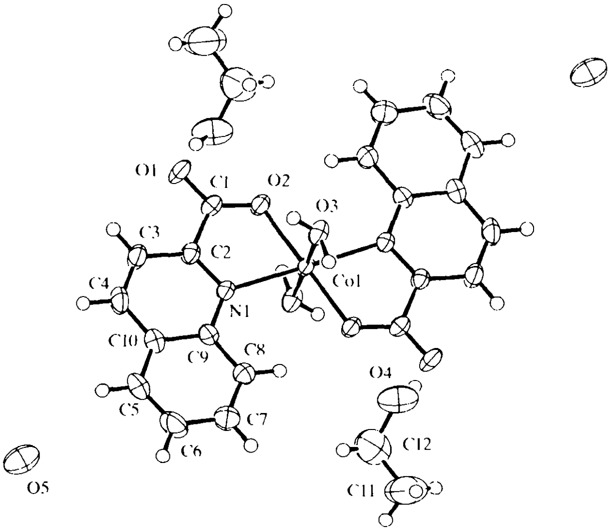


Fig. 1. *ORTEP*II (Johnson, 1976) drawing of the title compound. Ellipsoids for non-H atoms are shown at the 50% probability level.

The 2-quinolinecarboxylate ligand and the Co^{II} ion form a five-membered ring, which is also present in the Fe, Mn and Cu complexes. In this ring, the Co—O bonds [2.037 (2) Å] are shorter than those in both the Fe (2.087 Å; Okabe & Makino, 1998) and Mn complexes [2.125 (Haendler, 1996) and 2.131 Å (Okabe & Koizumi, 1997)], but longer than those in the Cu complex (1.954 and 1.962 Å; Haendler, 1996). The Co—N bonds [2.226 (2) Å] are also shorter than those in the Fe (2.270 Å; Okabe & Makino, 1998) and Mn complexes [2.324 (Haendler, 1996) and 2.315 Å (Okabe & Koizumi, 1997)], but longer than those in the Cu complex (2.014 and 2.012 Å; Haendler, 1986). The order of these bond lengths is Mn > Fe > Co > Cu. These differences may be explained by the difference in the ionic radii of the metal ions [Mn^{II} (high-spin state, six coordinate = *h*,6) 0.97 > Fe^{II} (*h*,6) 0.92 > Co^{II} (*h*,6) 0.89 > Cu^{II} (square planar) 0.71 Å; Cu^{II} (six coordinate) 0.87 Å (Shannon, 1976)]. The reverse of this order also coincides well with the well known Irving–Williams series which indicates the stability of the high-spin metal–chelate compound.

Two uncoordinated water molecules and two uncoordinated ethanol molecules are present in the title compound. Similar uncoordinated water and ethanol molecules are also found in the Mn (Haendler, 1996) and Fe complexes (Okabe & Makino, 1997). The molecules are linked together by a hydrogen-bonding network involving the chelate molecules, the uncoordinated water molecules and the ethanol molecules. Each uncoordinated water molecule is involved in three hydrogen bonds: two to O atoms of the uncoordinated ethanol molecules and one to the O atom of the coordinated water molecule. The O atoms of the ethanol molecules and the O atoms of the coordinated water molecules form hydrogen bonds with the uncoordinated O atoms of the carboxylate groups. In this hydrogen-bonding scheme, two uncoordinated water molecules, two uncoordinated ethanol molecules and two coordinated water molecules participate in an extended hydrogen-bonding network. The magnitude of the hydrogen-bond distances indicate that the hydrogen bonds are relatively strong.

Experimental

Yellow prismatic crystals of (II) were obtained at room temperature by the slow evaporation of a mixture of an ethanol solution (*ca* 90%) of 2-quinolinecarboxylic acid and cobalt(II) chloride hexahydrate (molar ratio 4:1).

Crystal data

$[Co(C_{10}H_6NO_2)_2(H_2O)_2]\cdot 2H_2O\cdot 2C_2H_6O$
$M_r = 567.452$
Triclinic
$P\bar{1}$
$a = 9.027\ (1)$ Å
$b = 11.095\ (3)$ Å
$c = 7.275\ (2)$ Å
$\alpha = 106.23\ (2)^\circ$
$\beta = 108.02\ (2)^\circ$
$\gamma = 78.09\ (2)^\circ$
$V = 659.5\ (2)$ Å^3
$Z = 1$
$D_x = 1.429$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 21.1\text{–}23.9^\circ$
$\mu = 0.702$ mm^{-1}
$T = 296$ K
Prism
$0.3 \times 0.2 \times 0.1$ mm
Yellow

Data collection

Rigaku AFC-5*R* diffractometer

$R_{int} = 0.019$
$\theta_{max} = 27.5^\circ$

ω–2θ scans
Absorption correction: none
3226 measured reflections
3035 independent reflections
2521 reflections with $I > \sigma(I)$

$h = 0 \rightarrow 11$
$k = -13 \rightarrow 14$
$l = -9 \rightarrow 8$
3 standard reflections every 150 reflections intensity decay: −1.4%

Refinement

Refinement on F^2
$R(F) = 0.063$
$wR(F^2) = 0.097$
$S = 1.500$
2521 reflections
181 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 4F_o^2/\sigma^2(F_o^2)$
$(\Delta/\sigma)_{\max} = 0.010$
$\Delta\rho_{\max} = 0.52$ e Å^{-3}
$\Delta\rho_{\min} = -0.44$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. *Selected geometric parameters (Å, °)*

Co1—O2	2.037 (2)	C3—C4	1.352 (4)
Co1—O3	2.122 (2)	C4—C10	1.404 (4)
Co1—N1	2.226 (2)	C5—C6	1.361 (5)
O1—C1	1.246 (3)	C5—C10	1.412 (4)
O2—C1	1.264 (3)	C6—C7	1.401 (5)
O4—C12	1.411 (5)	C7—C8	1.370 (4)
N1—C2	1.326 (3)	C8—C9	1.405 (4)
N1—C9	1.372 (4)	C9—C10	1.421 (4)
C1—C2	1.509 (4)	C11—C12	1.430 (6)
C2—C3	1.409 (4)		
O2—Co1—O3	91.10 (8)	O1—C1—O2	124.3 (2)
O2—Co1—N1	77.23 (7)	O1—C1—C2	118.6 (2)
O3—Co1—N1	89.51 (8)	O2—C1—C2	117.1 (2)
Co1—O2—C1	118.9 (2)	N1—C2—C1	115.9 (2)
Co1—N1—C2	110.8 (2)	N1—C2—C3	123.6 (2)
Co1—N1—C9	131.0 (2)	C1—C2—C3	120.5 (2)
C2—N1—C9	118.2 (2)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H16···O1^{i}	0.91 (5)	1.80 (5)	2.701 (4)	166 (4)
O3—H9···O1^{ii}	0.80 (4)	1.95 (4)	2.746 (3)	173 (4)
O3—H10···O5^{iii}	1.09 (4)	1.63 (4)	2.717 (3)	174 (3)
O5···O4^{iv}			2.757 (4)	
O5···O4^{v}			2.893 (4)	

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

H atoms were placed in calculated positions and included in the structure-factor calculations, with the exception of those involved in hydrogen bonding, which were located in a difference Fourier map and were refined isotropically.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985) and *DIRDIF* (Beurskens, 1984). Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *ORTEP*II (Johnson, 1976).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: DA1034). Services for accessing these data are described at the back of the journal.

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Bis[2-hydroxy-3-(1*H*-imidazol-4-yl)-propionato]cobalt(II)

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Abstract

In the title compound, $[Co(C_6H_7N_2O_3)_2]$, the Co^{II} ion has a slightly distorted octahedral coordination geometry. 2-Hydroxy-3-(1*H*-imidazol-4-yl)propanoic acid acts as a bidentate ligand. Two carboxylate O atoms and two hydroxyl O atoms are coordinated *cis* to each other and form the equatorial plane, and two imidazole N atoms are coordinated in axial positions. The molecules are held together by an intermolecular hydrogen-bonding network involving the carboxylate, imino and hydroxyl groups.

Comment

2-Hydroxy-3-(1*H*-imidazol-4-yl)propanoic acid, (I), also known as imidazolelactic acid, is one of the final well known products of L-histidine catabolism. Patients with liver cirrhosis or histidinemia have high urinary concentrations of (I) (Dubovsky & Dubovska, 1965; Murray *et al.*, 1993). It also has an inhibitory action