$a = 10.670\,(1)$ Å
$b = 11.752\,(1)$ Å
$c = 12.099\,(1)$ Å
$\alpha = 83.93\,(1)^\circ$
$\beta = 72.21\,(1)^\circ$
$\gamma = 74.02\,(1)^\circ$
$V = 1388.4\,(2)$ Å^3
$Z = 2$
$D_x = 1.471$ Mg m^{-3}
D_m not measured

$\mu = 0.841$ mm^{-1}
$T = 293\,(2)$ K
Prismatic
$0.5 \times 0.5 \times 0.3$ mm
Green

Data collection

Siemens *P*4/PC diffractometer
$\theta/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.733$, $T_{\max} = 0.777$
5723 measured reflections
4867 independent reflections
3572 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.024$
$\theta_{\max} = 25^\circ$
$h = -1 \rightarrow 12$
$k = -13 \rightarrow 13$
$l = -13 \rightarrow 14$
3 standard reflections every 97 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.144$
$S = 1.019$
4867 reflections
393 parameters
H atoms constrained
$w = 1/[\sigma^2(F_o^2) + (0.0734P)^2 + 1.0087P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.017$
$\Delta\rho_{\max} = 0.53$ e Å^{-3}
$\Delta\rho_{\min} = -0.50$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

Cu1—O1	1.907 (3)	Cu1—N2	1.998 (3)
Cu1—O2	1.939 (3)	Cu1—O1*W*	2.306 (4)
Cu1—N1	1.994 (3)		
O1—Cu1—O2	93.8 (1)	N1—Cu1—N2	82.0 (1)
O1—Cu1—N1	93.8 (1)	O1—Cu1—O1*W*	98.5 (1)
O2—Cu1—N1	171.8 (1)	O2—Cu1—O1*W*	88.8 (2)
O1—Cu1—N2	169.6 (1)	N1—Cu1—O1*W*	93.4 (1)
O2—Cu1—N2	90.0 (1)	N2—Cu1—O1*W*	91.3 (1)
O1—C13—C18—C19	−2.4 (6)	O2—C19—C18—C13	4.2 (7)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H1*W*···O4	0.97	1.87	2.770 (8)	154
O1*W*—H1*W*···N3	0.97	2.58	3.462 (7)	151
O1*W*—H2*W*···O2*W*	0.94	1.94	2.879 (7)	172
O2*W*—H3*W*···O5$^{\rm i}$	1.01	2.10	3.033 (9)	154
O2*W*—H3*W*···O3$^{\rm i}$	1.01	2.12	2.987 (8)	144
O2*W*—H3*W*···N3$^{\rm i}$	1.01	2.44	3.415 (9)	163
O2*W*—H4*W*···O3	1.00	1.95	2.880 (9)	153

Symmetry code: (i) $1 - x, -y, 1 - z$.

All H atoms attached to C atoms were refined using a riding model, while water H atoms were placed in fixed positions with refined $U_{\rm iso}$ values.

Data collection: *SHELXTL/PC* (Siemens, 1994). Cell refinement: *SHELXTL/PC*. Data reduction: *SHELXTL/PC*. Program(s) used to solve structure: *SHELX97* (Sheldrick, 1997). Program(s) used to refine structure: *SHELX97*. Molecular graphics: *ZORTEP* (Zsolnai, 1997). Software used to prepare material for publication: *CIFTAB* in *SHELX97*.

We would like to acknowledge Sylvain Bernès from USAI–Facultad de Química for collection of the data.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1122). Services for accessing these data are described at the back of the journal.

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Tetrakis(μ-phenylacetato-*O*:*O*′)bis[(quinoline-*N*)cobalt(II)]

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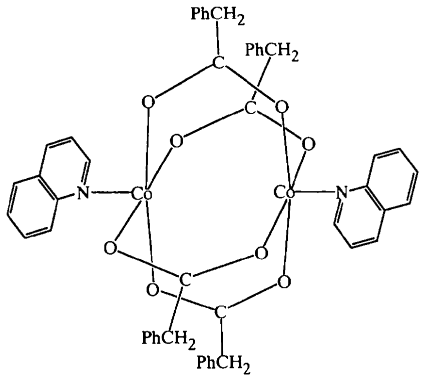
Abstract

The centrosymmetric title molecule, $[\mathrm{Co_2(C_8H_7O_2)_4(C_9H_7N)_2}]$, contains two Co$^{\rm II}$ centres bridged by four phenylacetate ligands in *syn–syn* bridging modes.

Square-pyramidal five-coordination of each cobalt is completed by a quinoline N atom. The $Co^{II}\cdots Co^{II}$ distance is 2.813 (1) Å.

Comment

It has been recognized that Co^{II} is reluctant to form binuclear carboxylate-bridged complexes (Mehrotra & Bohra, 1983). While there are many examples of Rh^{II} and Cu^{II} binuclear species, it is mononuclear complexes of cobalt that predominate. The major factor encouraging binuclear complex formation with cobalt is the presence of steric crowding around the metal centres, achieved by the coordination of sterically demanding carboxylate groups and 2-substituted heterocyclic amines, in order to inhibit the formation of bis(amine) adducts. Hitherto, several dimeric Co^{II} carboxylates have been reported, but few of them have been structurally characterized by X-ray diffraction. Here, we report the structure of the title binuclear Co complex, $[Co_2(O_2CCH_2Ph)_4(C_9H_7N)_2]$, (I).



(I)

The title molecule is a centrosymmetric dimer with four bidentate phenylacetate groups as bridging ligands between two Co^{II} centres, to each of which a quinoline group is also coordinated, as shown in Fig. 1. The structure is similar to $[Co_2(O_2CCMe_3)_4(C_9H_7N)_2]$ (Cui *et al.*, 1999) and $[Co_2(O_2CPh)_4(C_9H_7)_2]$ (Catterick *et al.*, 1977). The coordination geometry about each Co atom is distorted square pyramidal, with four O atoms from four different carboxylate ligands forming the basal plane and a quinoline N atom occupying the axial position, where the most distorted angle is 103.44 (9)° for $O4^i$—Co—N [symmetry code: (i) $1-x$, $1-y$, $1-z$]. The interatomic distances of Co—O [2.010 (2)–2.047 (2) Å], Co—N [2.084 (2) Å] and Co···Co [2.813 (1) Å] all agree well with the related values recorded for the structures of the analogous pivalate (Cui *et al.*, 1999) and benzoate (Catterick *et al.*, 1977). The Co···Co distance is much longer than the value of 2.36 Å found for the multiple Cr—Cr bond in $[Cr_2(O_2CCH_3)_4(H_2O)_2]$ (Cotton *et al.*, 1971); it is also longer than the Cu···Cu distances in binuclear copper carboxylates (2.58–2.747 Å; Catterick & Thornton, 1977), but is much shorter than the V···V separation of 3.704 Å found for cyclopentadienylvanadium(II) trifluoroacetate (Larin *et al.*, 1971). The molecular geometries within the quinoline and phenylacetate ligands are unexceptional. The quinoline plane almost exactly bisects the O1—Co—O3 angle. The dihedral angles between the mean planes through the C13–C18 and C23–C28 benzene rings and the quinoline plane are 16.81 (17) and 74.87 (8)°, respectively. The dihedral angle between these two benzene rings is 88.34 (10)°, indicating that they are almost orthogonal to each other.

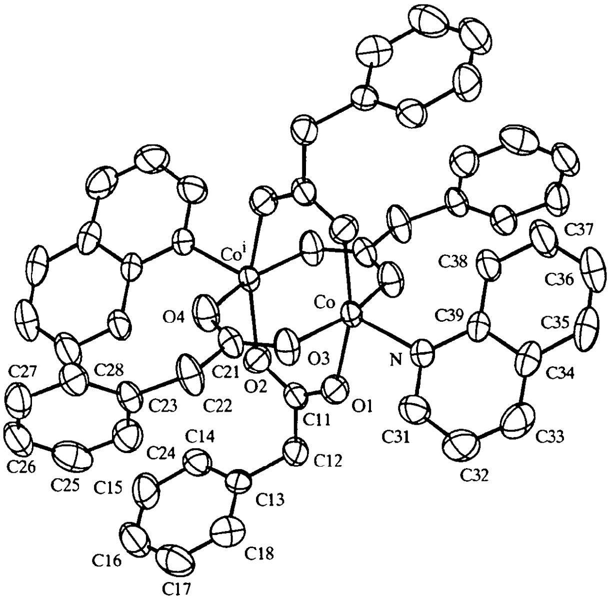


Fig. 1. A view of the molecular structure of (I) showing 50% probability displacement ellipsoids and the atom-numbering scheme. H atoms have been omitted for clarity [symmetry code: (i) $1-x$, $1-y$, $1-z$].

Experimental

A mixture of $Co(NO_3)_2\cdot 6H_2O$, $Mg(NO_3)_2\cdot 6H_2O$, phenylacetic acid and quinoline (molar ratio 2:1:6:2) in EtOH was refluxed for 10 h. The resulting solution was allowed to stand in air and green crystals of the title complex were deposited after a week.

Crystal data

$[Co_2(C_8H_7O_2)_4(C_9H_7N)_2]$
$M_r = 916.72$
Triclinic
$P\bar{1}$

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 24 reflections

$a = 8.410\,(2)$ Å
$b = 11.116\,(2)$ Å
$c = 12.122\,(2)$ Å
$\alpha = 95.24\,(3)^\circ$
$\beta = 108.29\,(3)^\circ$
$\gamma = 93.24\,(3)^\circ$
$V = 1067.1\,(4)$ Å^3
$Z = 1$
$D_x = 1.426$ Mg m^{-3}
D_m not measured

$\theta = 14$–15°
$\mu = 0.836$ mm^{-1}
$T = 293\,(2)$ K
Trigonal prism
$0.20 \times 0.15 \times 0.15$ mm
Green

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
4482 measured reflections
4181 independent reflections
3637 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.027$
$\theta_{\rm max} = 26^\circ$
$h = 0 \rightarrow 10$
$k = -13 \rightarrow 13$
$l = -14 \rightarrow 14$
3 standard reflections
frequency: 120 min
intensity variation: 1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.116$
$S = 1.084$
4181 reflections
280 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0676P)^2 + 0.2407P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.31$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.61$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

Co—O1	2.047 (2)	Co—O4$^{\rm i}$	2.010 (2)
Co—O2$^{\rm i}$	2.014 (2)	Co—N	2.084 (2)
Co—O3	2.045 (2)	Co···Co$^{\rm i}$	2.8130 (10)
O1—Co—N	94.00 (9)	O2$^{\rm i}$—Co—O3	89.85 (10)
O2$^{\rm i}$—Co—N	102.66 (9)	O3—Co—O1	85.52 (10)
O3—Co—N	93.06 (9)	O4$^{\rm i}$—Co—O1	88.74 (10)
O4$^{\rm i}$—Co—N	103.44 (9)	O4$^{\rm i}$—Co—O2$^{\rm i}$	90.97 (10)
O2$^{\rm i}$—Co—O1	162.93 (8)	O4$^{\rm i}$—Co—O3	162.87 (9)

Symmetry code: (i) $1 - x, 1 - y, 1 - z$.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC* (Sheldrick, 1994). Software used to prepare material for publication: *SHELXL*93.

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Charge-transfer salt of bis(*cis*-1,2-diphenylethene-1,2-dithiolato)palladate(1–) with cobaltocenium, and neutral bis(*cis*-1,2-diphenylethene-1,2-dithiolato)palladium

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Abstract

The crystal structure of the charge-transfer salt cobaltocenium bis(*cis*-1,2-diphenylethene-1,2-dithiolato)palladate(1−), $[\mathrm{Co(C_5H_5)_2}][\mathrm{Pd\{S_2C_2(C_6H_5)_2\}_2}]$, consists of linear $-D^+A^-D^+A^-D^+A^-$– chains. The $[\mathrm{Co(C_5H_5)_2}]^+$ cation adopts an approximately eclipsed D_{5h} configuration. The stereochemistry of the $[\mathrm{Pd\{S_2C_2(C_6H_5)_2\}_2}]^-$ anion is largely similar to that of the neutral molecule, bis(*cis*-1,2-diphenylethene-1,2-dithiolato)palladium, $[\mathrm{Pd\{S_2C_2(C_6H_5)_2\}_2}]$, the structure of which has also been determined; however, the Pd atom of the former has a distorted square-planar environment, while the $\mathrm{PdS_4}$ chromophore of the latter is strictly planar. Furthermore, the former has longer Pd—S and S—C distances, and shorter C—C distances (in the chelate rings) than the latter.