

$\omega/2\theta$ scans
 Absorption correction:
 empirical via ψ scans
 (MOLÉN; Fair, 1990)
 $T_{\min} = 0.833$, $T_{\max} = 0.857$
 2092 measured reflections
 2018 independent reflections

$R_{\text{int}} = 0.037$
 $\theta_{\max} = 26.0^\circ$
 $h = 0 \rightarrow 13$
 $k = 0 \rightarrow 14$
 $l = 0 \rightarrow 18$
 3 standard reflections
 frequency: 120 min
 intensity decay: 0.26%

Refinement

Refinement on F^2
 $R = 0.029$
 $wR = 0.034$
 $S = 0.74$
 1350 reflections
 154 parameters
 H atoms: see below
 Weighting scheme: see below

$(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.561 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.291 \text{ e } \text{Å}^{-3}$
 Extinction correction: none
 Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. Selected geometric parameters (Å , $^\circ$)

Mo—M1	2.392 (7)	Mo—C9	2.462 (6)
Mo—M2	2.385 (9)	Mo—C10	2.496 (6)
Mo—C1	2.030 (7)	O1—C1	1.151 (9)
Mo—C2	1.952 (6)	O2—C2	1.176 (8)
Mo—C3	1.945 (6)	O3—C3	1.166 (8)
Mo—C4	2.032 (6)	O4—C4	1.156 (7)
Mo—C5	2.476 (6)	C5—C6	1.360 (8)
Mo—C6	2.497 (6)	C9—C10	1.348 (8)
C1—Mo—M1	94.9 (3)	C3—Mo—M2	94.2 (3)
C1—Mo—M2	96.8 (2)	C3—Mo—C4	86.1 (3)
C1—Mo—C2	84.3 (3)	C3—Mo—C5	160.6 (2)
C1—Mo—C3	84.4 (3)	C3—Mo—C6	162.0 (2)
C1—Mo—C4	165.4 (2)	C3—Mo—C9	92.8 (2)
C1—Mo—C5	79.0 (2)	C3—Mo—C10	95.2 (2)
C1—Mo—C6	110.7 (2)	C4—Mo—M1	96.4 (3)
C1—Mo—C9	112.5 (2)	C4—Mo—M2	94.9 (2)
C1—Mo—C10	81.4 (2)	C4—Mo—C5	112.1 (2)
C2—Mo—M1	97.7 (3)	C4—Mo—C6	80.8 (2)
C2—Mo—M2	173.7 (3)	C4—Mo—C9	79.0 (2)
C2—Mo—C3	92.1 (3)	C4—Mo—C10	110.6 (2)
C2—Mo—C4	84.9 (3)	C5—Mo—C6	31.8 (2)
C2—Mo—C5	96.0 (2)	C5—Mo—C9	84.6 (2)
C2—Mo—C6	98.9 (2)	C5—Mo—C10	72.7 (2)
C2—Mo—C9	162.9 (2)	C6—Mo—C9	72.7 (2)
C2—Mo—C10	163.2 (2)	C6—Mo—C10	78.1 (2)
C3—Mo—M1	170.0 (3)	C9—Mo—C10	31.6 (2)

For the weighting scheme, if $F \geq 90.55$, then $w = 90.55/F^2$, otherwise $w = 1$, or $w = 0$ if $F^2 < 3\sigma(F^2)$. All non-H atoms were refined with anisotropic displacement parameters. H atoms were placed geometrically 0.95 Å from their parent C atoms. For all H atoms, a riding model was used with $B_{\text{iso}}(\text{H}) = 1.3B_{\text{eq}}(\text{C})$.

Data collection: *CAD-4 Express* (Enraf–Nonius, 1993). Data reduction: *MOLÉN* (Fair, 1990). Program(s) used to solve structure: *SIMPEL* in *MOLÉN*. Program(s) used to refine structure: *LSFM* in *MOLÉN*. Molecular graphics: *ORTEPII* (Johnson, 1976) in *MOLÉN*. Software used to prepare material for publication: *MOLÉN*.

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Lists of structure factors, anisotropic displacement parameters, atomic coordinates and complete geometry have been deposited with the IUCr (Reference: AB1394). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Diaquabis(*N,N*-diethylnicotinamide-*N*¹)bis-(2-hydroxybenzoato-*O*)cobalt(II)

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Abstract

The title monomeric centrosymmetric cobalt complex, $[\text{Co}(\text{C}_7\text{H}_5\text{O}_3)_2(\text{C}_{10}\text{H}_{14}\text{N}_2\text{O})_2(\text{H}_2\text{O})_2]$, contains two water molecules, two 4-hydroxybenzoate ligands and two diethylnicotinamide (DENA) ligands. All ligands are coordinated to the Co atom as monodentate ligands. The four nearest O atoms around the Co atom form a slightly distorted square-planar arrangement, with the distorted octahedral coordination completed by the pyridine N atoms of the DENA ligand at a distance of

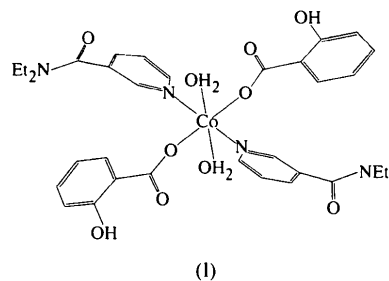
2.141 (5) Å. The water molecules are hydrogen bonded to the carboxyl O atoms [O···O 2.687 (5) Å].

Comment

The structural functions and coordination relationships of the arylcarboxylate ion in cobalt(II) complexes of benzoic acid derivatives change depending on the nature and position of the substituted groups in the phenyl ring, the nature of the additional ligand molecule or solvent and the medium of synthesis (Shnulin, Nadzhafov, Amiraslanov, Usubaliev & Mamedov, 1981; Adiwidjaja, Rossmannith & Kupperts, 1978; Amiraslanov, Mamedov, Movsumov, Musaev & Nadzhafov, 1979; Nadzhafov, Shnulin & Mamedov, 1981; Antsyshkina, Chiragov & Poray-Koshits, 1980). When pyridine and its derivatives are used instead of water molecules, the structure is completely different (Catterick, Hursthouse, New & Thornton, 1974). The crystal structure of [Co(CH₃CO₂)₂(DENA)₂(H₂O)₂], a diethylnicotinamide (DENA) complex of cobalt(II), has been reported (Mikelashvili, 1982). This complex is isostructural with similar Ni, Mn, Zn and Cd complexes (Sergienko, Shurkina, Khodashova, Poray-Koshits & Tsintsadze, 1980). We report here the structure of [Co(C₇H₅O₃)₂(DENA)₂(H₂O)₂], (I).

The structure of the title compound has been determined and compared with the results reported previously. The monomeric [Co(C₇H₅O₃)₂(DENA)₂(H₂O)₂] molecules have the typical structure of DENA complexes in which the metal atom has a *trans*-octahedral coordination (Sergienko *et al.*, 1980). The complex has a centre of symmetry with the benzoate ion and DENA ligand acting as monodentate ligands (Fig. 1).

Four symmetry-related O atoms form a slightly distorted square plane around the Co atom and the slightly distorted octahedral arrangement is completed by the pyridine N atoms of DENA ligands at a distance of 2.141 (5) Å. There are hydrogen bonds between water molecules and the carboxyl O3 atoms [O···O 2.687 (5) Å]. Similar hydrogen bonds are observed in both bis(*p*-aminobenzoate)tetraaquacobalt(II) [O···O 2.592 (3) Å] (Amiraslanov *et al.*, 1979) and bis(*p*-nitrobenzoate)tetraaquacobalt(II) dihydrate [O···O 2.59 Å] (Nadzhafov *et al.*, 1981). There is also an intramolecular O4···O3 hydrogen bond [1.716 (2) Å].



The bond lengths and angles in the DENA molecule and the phenyl ring of the monodentate benzoate anion are in good agreement with known values. The dihedral angle between the planar carboxyl group (C1, C2, O2, O3) and the planar phenyl ring (C2–C7) is 2.2 (6)°. The corresponding angles are 6.7 (9), 6.08, 7.12 and 5.10, and 1.3° in [Cu(C₆H₅COO)₂(C₁₀H₁₄N₂O)₂] (Hökelek, Gündüz & Necefoğlu, 1996), [Cu(PBN)₂(py)₂] (Usubaliev *et al.*, 1981), [Cu₂(C₆H₅COO)₄(py)₂] (Usubaliev *et al.*, 1980) and [Cu(PHB)₂.8(H₂O)] (Shnulin, Nadzhafov & Mamedov, 1981).

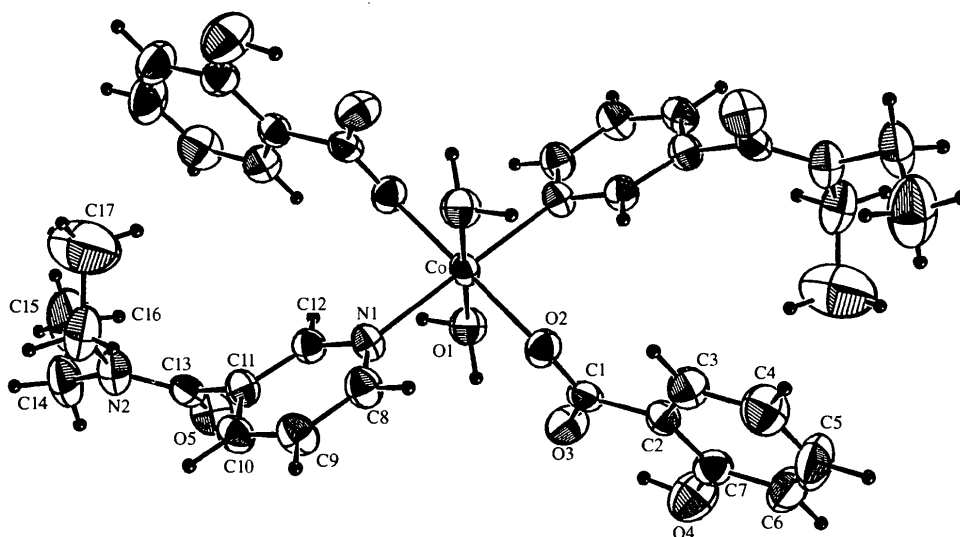


Fig. 1. An ORTEP (Johnson, 1976) drawing of the title molecule with the atom-numbering scheme. The displacement ellipsoids are drawn at the 50% probability level.

Experimental

The title compound was prepared from the reaction of 0.01 mol $[\text{Co}(\text{C}_6\text{H}_5\text{COO})_2] \cdot 3\text{H}_2\text{O}$ and 0.02 mol DENA in 100 ml ethanol. The mixture was filtered and set aside to crystallize at ambient temperature for a few weeks. Suitable pink crystals were obtained.

Crystal data

$[\text{Co}(\text{C}_7\text{H}_5\text{O}_3)_2(\text{C}_{10}\text{H}_{14}\text{N}_2\text{O})_2 \cdot (\text{H}_2\text{O})_2]$	Mo $K\alpha$ radiation
$M_r = 725.7$	$\lambda = 0.71073 \text{ \AA}$
Triclinic	Cell parameters from 25 reflections
$P\bar{1}$	$\theta = 10\text{--}18^\circ$
$a = 7.627 (1) \text{ \AA}$	$\mu = 0.545 \text{ mm}^{-1}$
$b = 9.054 (1) \text{ \AA}$	$T = 298 \text{ K}$
$c = 14.600 (1) \text{ \AA}$	Rod
$\alpha = 78.64 (1)^\circ$	$0.38 \times 0.16 \times 0.13 \text{ mm}$
$\beta = 84.91 (1)^\circ$	Pink
$\gamma = 63.67 (1)^\circ$	
$V = 885.9 (2) \text{ \AA}^3$	
$Z = 1$	
$D_x = 1.36 \text{ Mg m}^{-3}$	
D_m not measured	

Data collection

Enraf-Nonius CAD-4 diffractometer	$\theta_{\text{max}} = 25.0^\circ$
$\omega/2\theta$ scans	$h = 0 \rightarrow 9$
Absorption correction: none	$k = -8 \rightarrow 10$
3315 measured reflections	$l = -15 \rightarrow 16$
3105 independent reflections	3 standard reflections every 250 reflections
1599 reflections with $I > 3\sigma(I)$	frequency: 120 min
$R_{\text{int}} = 0.035$	intensity decay: 1%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(I) + (0.04F^2)^2/4F^2]$
$R = 0.050$	$(\Delta/\sigma)_{\text{max}} = 0.01$
$wR = 0.056$	$\Delta\rho_{\text{max}} = 0.49 \text{ e \AA}^{-3}$
$S = 1.56$	$\Delta\rho_{\text{min}} = -0.17 \text{ e \AA}^{-3}$
1599 reflections	Extinction correction: none
223 parameters	Scattering factors from <i>International Tables for X-ray Crystallography</i> (Vol. IV)
H atoms riding	

Table 1. Selected geometric parameters (\AA , $^\circ$)

Co—N1	2.141 (5)	O2—C1	1.251 (6)
Co—O1	2.119 (4)	O3—C1	1.254 (7)
Co—O2	2.078 (3)	O4—C7	1.328 (8)
N1—C8	1.345 (5)	O5—C13	1.235 (6)
N1—C12	1.326 (7)	C1—C2	1.500 (8)
N2—C13	1.337 (8)	C11—C13	1.492 (8)
N2—C14	1.471 (9)	C14—C15	1.492 (8)
N2—C16	1.468 (8)	C16—C17	1.47 (1)
N1—Co—O1	87.9 (2)	C1—C2—C7	120.7 (5)
N1—Co—O2	91.3 (1)	C3—C2—C7	118.8 (5)
O1—Co—O2	92.5 (1)	O4—C7—C2	121.8 (5)
Co—N1—C8	122.6 (4)	O4—C7—C6	117.5 (5)
Co—N1—C12	120.4 (3)	N1—C8—C9	122.4 (5)
Co—O2—C1	128.6 (3)	C10—C11—C12	117.2 (5)
C8—N1—C12	117.0 (5)	C10—C11—C13	123.9 (6)
C13—N2—C14	118.0 (5)	C12—C11—C13	118.4 (4)

C13—N2—C16	123.4 (5)	N1—C12—C11	124.7 (4)
C14—N2—C16	118.3 (5)	N2—C13—O5	121.7 (5)
O2—C1—O3	124.5 (5)	N2—C13—C11	120.2 (5)
O2—C1—C2	117.3 (5)	O5—C13—C11	118.1 (5)
O3—C1—C2	118.2 (5)	N2—C14—C15	114.5 (6)
C1—C2—C3	120.5 (5)	N2—C16—C17	114.2 (5)
C14—N2—C13—O5	2.6 (7)	O3—C1—C2—C3	-178.1 (5)
C14—N2—C13—C11	-175.2 (5)	O3—C1—C2—C7	1.0 (7)
C16—N2—C13—O5	-171.1 (5)	C10—C11—C13—N2	62.5 (7)
C16—N2—C13—C11	11.1 (8)	C10—C11—C13—O5	-115.4 (6)
O2—C1—C2—C3	3.0 (7)	C12—C11—C13—N2	-125.2 (5)
O2—C1—C2—C7	-177.9 (5)	C12—C11—C13—O5	56.9 (7)

H-atom positions were calculated geometrically, with $U_{\text{eq}}(\text{H}) = 1.3U_{\text{eq}}$ of the parent non-H atom. A riding model was used in the refinement.

Data collection: *MolEN* (Fair, 1990). Cell refinement: *MolEN*. Data reduction: *MolEN*. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *MolEN*. Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *MolEN*.

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Lists of structure factors, anisotropic displacement parameters, atomic coordinates and complete geometry have been deposited with the IUCr (Reference: AB1409). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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