$S = 2.39$
4973 reflections
414 parameters
H-atom parameters not refined
$w = 4F_o^2/\sigma^2(F_o)$

Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)*

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Co1	0.6795 (1)	0.419	0.12506 (9)	1.78 (2)
S11	0.7665 (2)	0.4343 (2)	−0.0252 (2)	2.61 (4)
S12	0.6007 (2)	0.4065 (2)	0.2637 (2)	2.57 (4)
O11	0.8306 (6)	0.3764 (6)	0.2185 (5)	2.6 (1)
O12	1.0148 (6)	0.4419 (8)	0.2762 (6)	3.4 (2)
O13	0.6277 (6)	0.2690 (5)	0.0886 (6)	2.6 (1)
O14	0.4603 (7)	0.1713 (6)	0.0364 (7)	3.5 (2)
O15	0.6330 (8)	0.3042 (8)	0.3260 (7)	4.1 (2)
O16	0.6145 (8)	0.5078 (8)	0.3275 (7)	4.3 (2)
N11	0.7382 (7)	0.5694 (7)	0.1586 (7)	2.5 (2)
N12	0.5220 (7)	0.4519 (6)	0.0343 (6)	2.1 (1)
C11	0.9111 (9)	0.4527 (9)	0.2243 (7)	2.5 (2)
C12	0.8682 (8)	0.5582 (8)	0.1595 (8)	2.3 (2)
C13	0.8759 (9)	0.5431 (9)	0.0391 (8)	2.6 (2)
C14	0.834 (1)	0.648 (1)	−0.0277 (9)	3.5 (2)
C15	1.0035 (9)	0.513 (1)	0.0365 (9)	3.6 (2)
C16	0.848 (1)	0.302 (1)	−0.017 (1)	4.4 (2)
C17	0.5113 (9)	0.2594 (8)	0.0607 (8)	2.4 (2)
C18	0.4471 (7)	0.3677 (8)	0.0691 (6)	1.9 (1)
C19	0.4405 (8)	0.3912 (9)	0.1868 (8)	2.7 (2)
C110	0.382 (1)	0.298 (1)	0.237 (1)	4.5 (3)
C111	0.379 (1)	0.508 (1)	0.191 (1)	4.9 (3)
Co2	0.84196 (9)	0.8668 (1)	0.37470 (8)	1.38 (2)
S21	0.7117 (2)	0.8585 (2)	0.4905 (2)	1.94 (3)
S22	0.9694 (2)	0.8909 (2)	0.2766 (2)	1.72 (3)
O21	0.7185 (6)	0.8136 (6)	0.2546 (5)	2.3 (1)
O22	0.5304 (6)	0.8494 (8)	0.1706 (6)	3.4 (2)
O23	0.9033 (5)	0.7206 (5)	0.4116 (5)	2.0 (1)
O24	1.0691 (6)	0.6377 (6)	0.5066 (7)	3.2 (2)
O25	0.9655 (6)	0.8056 (7)	0.1931 (6)	3.0 (1)
O26	0.9686 (6)	1.0053 (6)	0.2377 (6)	2.5 (1)
N21	0.7663 (6)	1.0102 (6)	0.3288 (6)	1.9 (1)
N22	0.9738 (5)	0.9107 (6)	0.4974 (5)	1.6 (1)
C21	0.6252 (7)	0.8710 (9)	0.2350 (7)	2.2 (2)
C22	0.6372 (8)	0.9813 (9)	0.3024 (8)	2.3 (2)
C23	0.6045 (8)	0.9604 (9)	0.4116 (8)	2.4 (2)
C24	0.627 (1)	1.067 (1)	0.4821 (9)	3.7 (2)
C25	0.4741 (8)	0.925 (1)	0.392 (1)	4.2 (3)
C26	0.642 (1)	0.7232 (9)	0.462 (1)	4.1 (2)
C27	1.0126 (8)	0.7216 (8)	0.4712 (7)	2.1 (2)
C28	1.0699 (7)	0.8362 (7)	0.4870 (7)	1.8 (1)
C29	1.1118 (6)	0.8709 (8)	0.3875 (7)	1.9 (1)
C210	1.1889 (9)	0.7844 (9)	0.3505 (9)	2.9 (2)
C211	1.1775 (8)	0.9833 (8)	0.407 (1)	2.9 (2)
O1*W*	0.7320 (7)	0.0582 (7)	0.0899 (7)	3.6 (2)
O2*W*	0.100 (1)	0.221 (1)	0.292 (1)	8.3 (4)

Table 2. *Selected geometric parameters (Å, °)*

Co1—S11	2.376 (3)	Co2—S21	2.366 (3)
Co1—S12	2.180 (3)	Co2—S22	2.185 (2)
Co1—O11	1.913 (6)	Co2—O21	1.906 (6)
Co1—O13	1.918 (6)	Co2—O23	1.912 (6)
Co1—N11	1.948 (8)	Co2—N21	1.956 (7)
Co1—N12	1.934 (7)	Co2—N22	1.945 (6)
S11—Co1—S12	179.2 (1)	S21—Co2—S22	174.0 (1)
S11—Co1—N11	84.2 (3)	S21—Co2—N21	84.7 (3)
S12—Co1—O11	90.1 (2)	S22—Co2—O21	94.6 (2)
S12—Co1—N12	87.3 (3)	S22—Co2—N22	84.3 (2)
O11—Co1—N11	84.2 (3)	O21—Co2—N21	82.8 (3)
O11—Co1—N12	175.9 (3)	O21—Co2—N22	175.8 (3)
O13—Co1—N11	177.2 (4)	O23—Co2—N21	174.8 (3)
O13—Co1—N12	82.1 (3)	O23—Co2—N22	83.7 (3)

All non-H atoms were located by direct methods and difference Fourier synthesis. The structure was refined on F by full-matrix least-squares techniques. When the refinements were carried out by use of the set of the enantiomeric parameters of the R(S) configuration the residual values converged to $R = 0.073$ and $wR = 0.086$, respectively. This fact indicated that the R(S) configuration is probably the correct choice.

All calculations were performed using *MolEN* (Fair, 1990) on a VAX computer. Molecular graphics were obtained with *ORTEP*II (Johnson, 1976).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PT1040). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Tetraaquabis(3,5-dinitrobenzoato-*O*)-cobalt(II) Tetrahydrate

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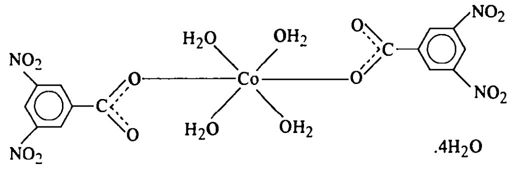
Abstract

In the title compound, $[Co(C_7H_3N_2O_6)_2(H_2O)_4].4H_2O$, the coordination polyhedron around the Co atom is a slightly distorted octahedron involving one carboxy O atom from each 3,5-dinitrobenzoato ligand and the O atoms of four water molecules. The mean Co—O_{water}

distance is 2.052 (2) Å, while the mean Co—$O_{benzoato}$ distance is significantly longer at 2.102 (11) Å. There are four water molecules of crystallization per asymmetric unit. Intra- as well as intermolecular hydrogen bonds exist in the structure.

Comment

3,5-Dinitrobenzoic acid is used for the preparation of amoxycillin and flucloxacillin (Amin, El-Ansary & Issa, 1994). The structures of two polymorphs of 3,5-dinitrobenzoic acid have been reported (Prince, Fronczek & Gandour, 1991). The complexes of 3,5-dinitrobenzoate with Zn^{II}, Cd^{II} and Hg^{II} have been synthesized and IR investigations of these complexes showed that the carboxylate ions coordinate to the central metal atom ion in a bidentate or bridging fashion (Odunola, 1993). We report herein the synthesis and structure of a cobalt complex of 3,5-dinitrobenzoic acid, (I).



(I)

The coordination polyhedron around the Co atom is a slightly distorted octahedron (Fig. 1). Four water O atoms constitute the basal plane of the octahedron, while two O atoms from two different carboxy groups occupy apical positions and complete the sixfold coordination. The dihedral angle between the benzene rings of the ligands is 16.3 (2)°. As can be seen from the torsion angles given in Table 2, neither the carboxy nor the nitro groups are coplanar with their respective benzene rings. The long axis of the molecule is parallel to the crystallographic *c* axis and the *a* axis is nearly normal to the benzene rings. The water molecules are involved in both intra- and intermolecular hydrogen bonds (Table 3).

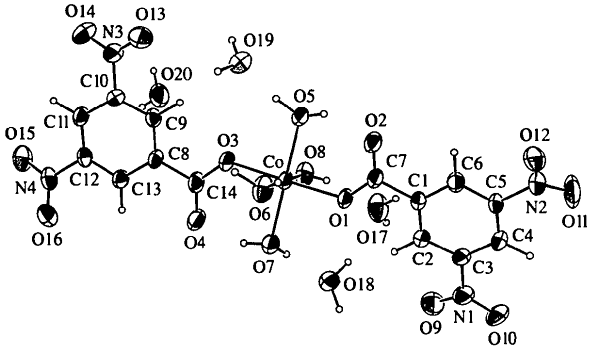


Fig. 1. A view of the title compound with the atom-numbering scheme. The displacement ellipsoids are drawn at the 50% probability level and H atoms are drawn as small circles of arbitrary radii.

Experimental

The title compound was synthesized by the reaction of hot aqueous solutions of $CoSO_4.7H_2O$ and sodium 3,5-dinitrobenzoate in a 1:2 stoichiometric ratio. The mixture was filtered and crystals were obtained after allowing the solution to stand for a few days at room temperature.

Crystal data

$[Co(C_7H_3N_2O_6)_2(H_2O)_4]$.-$4H_2O$
$M_r = 625.279$
Triclinic
$P\bar{1}$
$a = 7.204 (1)$ Å
$b = 11.752 (2)$ Å
$c = 15.057 (1)$ Å
$\alpha = 103.14 (1)°$
$\beta = 98.37 (1)°$
$\gamma = 92.45 (1)°$
$V = 1224.3 (6)$ Å^3
$Z = 2$
$D_x = 1.696$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 8.59–18.03°$
$\mu = 0.794$ mm^{-1}
$T = 295$ K
Prism
0.30 × 0.20 × 0.15 mm
Reddish

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scans (*MolEN*; Fair, 1990)
$T_{min} = 0.958$, $T_{max} = 0.999$
5198 measured reflections
4694 independent reflections

2944 observed reflections $[I > 3\sigma(I)]$
$R_{int} = 0.017$
$\theta_{max} = 26.32°$
$h = -8 \rightarrow 8$
$k = -14 \rightarrow 0$
$l = -18 \rightarrow 18$
3 standard reflections
frequency: 120 min
intensity decay: 1.6%

Refinement

Refinement on F
$R = 0.036$
$wR = 0.042$
$S = 1.27$
2765 reflections
352 parameters
$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{max} = 0.0007$

$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Co	0.23237 (6)	0.24792 (4)	0.49528 (3)	2.452 (8)
O1	0.1293 (3)	0.2282 (2)	0.6119 (1)	3.14 (5)
O2	0.0796 (4)	0.4090 (2)	0.6850 (2)	3.93 (6)
O3	0.3412 (3)	0.2698 (2)	0.3811 (1)	3.31 (5)
O4	0.3161 (4)	0.0903 (2)	0.2891 (2)	4.24 (6)
O5	0.3092 (3)	0.4250 (2)	0.5554 (2)	3.30 (5)
O6	−0.0359 (3)	0.2805 (2)	0.4334 (2)	3.78 (6)
O7	0.1520 (3)	0.0703 (2)	0.4355 (2)	3.33 (5)
O8	0.4996 (3)	0.2084 (2)	0.5542 (2)	3.63 (6)
O9	0.1365 (4)	−0.0847 (2)	0.7672 (2)	5.12 (7)
O10	0.0033 (4)	−0.0722 (2)	0.8888 (2)	5.04 (7)
O11	−0.0640 (5)	0.3305 (3)	1.0636 (2)	6.10 (8)

O12	−0.1819 (3)	0.4418 (2)	0.9778 (2)	4.15 (6)
O13	0.4319 (4)	0.6090 (2)	0.2541 (2)	5.15 (7)
O14	0.5732 (4)	0.5891 (2)	0.1353 (2)	4.91 (7)
O15	0.5474 (4)	0.1985 (3)	−0.0683 (2)	5.94 (8)
O16	0.3729 (4)	0.0638 (2)	−0.0381 (2)	4.23 (6)
O17	0.5145 (4)	0.1123 (2)	0.7099 (2)	5.31 (7)
O18	0.2777 (3)	−0.0700 (2)	0.5563 (2)	3.57 (6)
O19	0.2501 (3)	0.5834 (2)	0.4472 (2)	3.61 (6)
O20	0.9209 (5)	0.3471 (2)	0.2716 (2)	6.30 (9)
N1	0.0592 (4)	−0.0310 (3)	0.8284 (2)	3.79 (7)
N2	−0.1012 (4)	0.3563 (3)	0.9893 (2)	3.53 (7)
N3	0.4914 (4)	0.5491 (3)	0.1890 (2)	3.46 (7)
N4	0.4591 (4)	0.1596 (3)	−0.0171 (2)	3.33 (7)
C1	0.0433 (4)	0.2531 (3)	0.7585 (2)	2.32 (7)
C2	0.0637 (4)	0.1360 (3)	0.7563 (2)	2.45 (7)
C3	0.0309 (5)	0.0939 (3)	0.8318 (2)	2.59 (7)
C4	−0.0216 (5)	0.1630 (3)	0.9090 (2)	2.93 (7)
C5	−0.0433 (4)	0.2780 (3)	0.9084 (2)	2.57 (7)
C6	−0.0131 (4)	0.3262 (3)	0.8355 (2)	2.55 (7)
C7	0.0881 (5)	0.3017 (3)	0.6790 (2)	2.56 (7)
C8	0.3969 (4)	0.2527 (3)	0.2290 (2)	2.51 (7)
C9	0.4214 (4)	0.3736 (3)	0.2438 (2)	2.56 (7)
C10	0.4634 (5)	0.4211 (3)	0.1726 (2)	2.52 (7)
C11	0.4804 (5)	0.3546 (3)	0.0869 (2)	2.75 (7)
C12	0.4505 (5)	0.2348 (3)	0.0746 (2)	2.59 (7)
C13	0.4093 (5)	0.1820 (3)	0.1432 (2)	2.59 (7)
C14	0.3490 (5)	0.1989 (3)	0.3062 (2)	2.83 (7)

Table 2. *Selected geometric parameters (Å, °)*

Co—O1	2.059 (2)	Co—O8	2.116 (2)
Co—O3	2.052 (2)	O1—C7	1.253 (4)
Co—O5	2.087 (2)	O2—C7	1.248 (4)
Co—O6	2.110 (2)	O3—C14	1.250 (4)
Co—O7	2.096 (2)	O4—C14	1.248 (4)
O1—Co—O3	178.53 (8)	O5—Co—O7	179.3 (1)
O1—Co—O5	90.80 (9)	O5—Co—O8	88.82 (9)
O1—Co—O6	91.4 (1)	O6—Co—O7	86.09 (9)
O1—Co—O7	88.71 (9)	O6—Co—O8	177.74 (9)
O1—Co—O8	89.0 (1)	O7—Co—O8	91.69 (9)
O3—Co—O5	88.20 (9)	Co—O1—C7	131.6 (2)
O3—Co—O6	89.7 (1)	Co—O3—C14	130.8 (2)
O3—Co—O7	92.30 (9)	O1—C7—O2	126.2 (3)
O3—Co—O8	89.9 (1)	O3—C14—O4	126.2 (3)
O5—Co—O6	93.40 (9)		
O9—N1—C3—C2	11.1 (5)	O15—N4—C12—C11	22.8 (5)
O10—N1—C3—C4	12.0 (5)	O16—N4—C12—C13	23.7 (5)
O11—N2—C5—C4	25.4 (5)	C2—C1—C7—O1	4.9 (4)
O12—N2—C5—C6	25.5 (4)	C6—C1—C7—O2	2.3 (5)
O13—N3—C10—C11	161.8 (3)	C9—C8—C14—O3	−2.4 (5)
O14—N3—C10—C9	162.2 (3)	C13—C8—C14—O4	−2.0 (5)

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H···A	D—H	H···A	D···A	D—H···A
O5—H51···O2	0.80	2.06	2.767 (4)	146.6
O5—H52···O19	1.08	1.67	2.747 (4)	171.2
O6—H61···O19^{i}	0.91	1.92	2.808 (4)	165.1
O6—H62···O20^{ii}	1.03	1.72	2.704 (4)	157.6
O7—H71···O18	0.85	1.97	2.803 (3)	167.9
O7—H72···O4	0.97	1.78	2.699 (4)	156.5
O8—H81···O17	0.79	2.04	2.815 (4)	166.6
O8—H82···O18^{iii}	0.86	1.94	2.785 (4)	167.5
O17—H171···O4^{iii}	0.87	1.91	2.724 (4)	155.7
O18—H181···O17	0.94	2.12	3.022 (3)	161.5
O18—H182···O6^{iv}	1.04	2.02	3.010 (3)	158.6
O19—H192···O2^{i}	0.99	1.93	2.887 (4)	162.0
O20—H201···O2^{v}	0.88	1.98	2.792 (3)	152.4

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

H atoms bound to C atoms were placed geometrically at a distance of 1.05 Å, while water H atoms were taken from difference maps. For all H atoms, $U_{\rm iso} = 1.3U_{\rm eq}$ for the parent atom and a riding model was adopted.

Data collection: *CAD*-4 *EXPRESS Software* (Enraf–Nonius, 1993). Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *MolEN SIMPEL*. Program(s) used to refine structure: *MolEN LSFM*. Molecular graphics: *ORTEP* (Johnson, 1965) in *MolEN*. Software used to prepare material for publication: *MolEN*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BM1053). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1996). C**52**, 1394–1399

The Pseudo-Racemic Complex Bis[tris(2,2′-bipyridine)ruthenium(II)] Hexacyanocobaltate(III) Chloride Octahydrate, $[Ru(bpy)_3]_2[Co(CN)_6]Cl.8H_2O$

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Abstract

Crystals of $[Ru(C_{10}H_8N_2)_3]_2[Co(CN)_6]Cl.8H_2O$ ($C_{10}H_8N_2$ = bpy = 2,2′-bipyridine) which belong to the space group $C2$ are characterized by two 'pseudo-racemic' crystallographically independent $[Ru(bpy)_3]^{2+}$ cations. Three kinds of layers are found in the crys-

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