Data collection

Siemens *P*3 diffractometer
ω scans
Absorption correction: empirical *via* ψ scans (*SHELXTL*5.2; Siemens, 1994)
$T_{min} = 0.64$, $T_{max} = 0.79$
845 measured reflections
836 independent reflections
577 observed reflections $[I > 2\sigma(I)]$
$\theta_{max} = 24.09°$
$h = 0 \rightarrow 14$
$k = 0 \rightarrow 14$
$l = 0 \rightarrow 14$
3 standard reflections monitored every 197 reflections intensity decay: <1%

Refinement

Refinement on F^2
$R(F) = 0.052$
$wR(F^2) = 0.155$
$S = 1.062$
835 reflections
78 parameters
H-atom parameters not refined and solvent H atoms not included
$w = 1/[\sigma^2(F_o^2) + (0.0637P)^2 + 7.9153P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = -0.001$
$\Delta\rho_{max} = 0.63$ e Å^{-3}
$\Delta\rho_{min} = -0.63$ e Å^{-3}
Extinction correction: *SHELXTL*5.2
Extinction coefficient: 0.0027 (8)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Mn	0.1950 (1)	0.1914 (1)	1/2	0.0280 (5)
O(1)	0.3310 (4)	0.2953 (4)	0.3404 (4)	0.0525 (14)
C(1)	0.2765 (4)	0.2550 (5)	0.4009 (5)	0.0353 (14)
O(2)	0.0195 (6)	0.3468 (6)	1/2	0.060 (2)
C(2)	0.0892 (8)	0.2896 (7)	1/2	0.041 (2)
C(3)	0.0195 (6)	0.0591 (6)	1/2	0.030 (2)
C(4)	0.0886 (5)	0.0862 (4)	0.4068 (4)	0.0316 (12)
C(5)	0.1937 (5)	0.0511 (4)	0.4045 (5)	0.0333 (14)
C(6)	0.2470 (7)	0.0309 (6)	1/2	0.038 (2)
Cl	0.0845 (3)	0.4155 (3)	1/4	0.1087 (14)
C(7)†	0.0405 (15)	0.5405 (15)	1/4	0.182 (22)

† Site occupancy = 0.50.

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

Mn—C(1)	1.802 (6)	O(2)—C(2)	1.131 (11)
Mn—C(2)	1.808 (10)	C(3)—C(4)	1.501 (7)
Mn—C(6)	2.114 (8)	C(3)—C(3^i)	1.559 (15)
Mn—C(5)	2.132 (6)	C(4)—C(5)	1.389 (8)
Mn—C(4)	2.214 (5)	C(5)—C(6)	1.402 (7)
O(1)—C(1)	1.143 (7)	Cl—C(7)	1.661 (12)
C(1)—Mn—C(1^{ii})	88.1 (4)	C(4^{ii})—C(3)—C(4)	103.4 (6)
C(1)—Mn—C(2)	96.6 (3)	C(4)—C(3)—C(3^i)	113.4 (5)
C(1)—Mn—C(6)	104.2 (3)	C(5)—C(4)—C(3)	119.4 (5)
C(2)—Mn—C(6)	150.8 (4)	C(4)—C(5)—C(6)	119.4 (6)
O(1)—C(1)—Mn	177.7 (5)	C(5)—C(6)—C(5^{ii})	118.7 (8)
O(2)—C(2)—Mn	176.5 (8)		

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

Data collection: *P3/P4-PC Diffractometer Program* (Siemens, 1991). Cell refinement: *P3/P4-PC Diffractometer Program*. Data reduction: *SHELXTL*5.2 (Siemens, 1994). Program(s) used to solve structure: *SHELXTL*5.2. Program(s) used to refine structure: *SHELXTL*5.2. Molecular graphics: *SHELXTL*5.2. Software used to prepare material for publication: *SHELXTL*5.2.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BK1272). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Aqua[2,6-diacetylpyridinedi(benzoic acid hydrazone)]nitratocobalt(II) Nitrate at 173 K

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Abstract

The structure of aqua(nitrato-*O*){pyridine-2,6-diylbis[*N*-(1-ethylideneamino)benzamide]-O^1,N^2,N^1,N^5,O^2}cobalt(II) nitrate, $[Co(NO_3)(C_{23}H_{21}N_5O_2)(H_2O)]NO_3$, was redetermined at 173 K. There are only small differences between the bond distances in the cation at 173 K relative to the room-temperature determination reported previously. The average difference is 0.006 Å for all 45 bond distances and only 0.004 Å for 27 bonds if the two phenyl rings and two nitrate ions, which have large thermal motions, are excluded. The Co^{II} atom is in the center of a pentagonal bipyramid consisting of the pentadentate ligand, with a water molecule and monodentate nitrate ligand in the axial positions.

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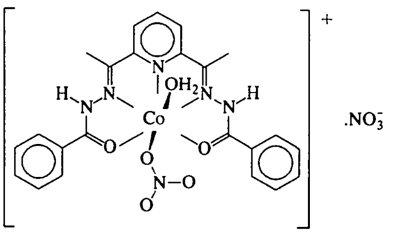
Comment

There is a lack of structural data for non-organometallic seven-coordinate cobalt complexes, as pointed out by Belletti, Carcelli, Pelizzi & Pelizzi (1992), henceforth BCPP. In the compilation by Orpen, Brammer, Allen, Kennard, Watson & Taylor (1989), there were no entries for Co—OH_2 with CN = 7 (CN = coordination number). In addition, comparisons between room- and low-temperature studies of coordination compounds are rare. This report describes the seven-coordinate cobalt complex aqua[2,6-diacetylpyridinedi(benzoic acid hydrazone)]nitratocobalt(II) nitrate, (I), at 173 K and compares it with the room-temperature study reported by Giordano, Palenik, Palenik & Sullivan (1979). The complex contains the planar pentadentate ligand H_2dapbah, which was prepared as reported previously.



(I)

Excluding the terminal phenyl rings and the nitrate ions, there is an average difference of 0.004 Å in the bond distances between the room- and low-temperature studies. The differences in Co–donor distances range from 0.003 to 0.008 Å, which are within two e.s.d.'s (0.004 Å) of the room-temperature study. For the terminal phenyl rings, the C—C bond lengths at 173 K are all longer than at room temperature, which is expected because of reduced motion at 173 K. A similar situation exists for the two nitrate anions.

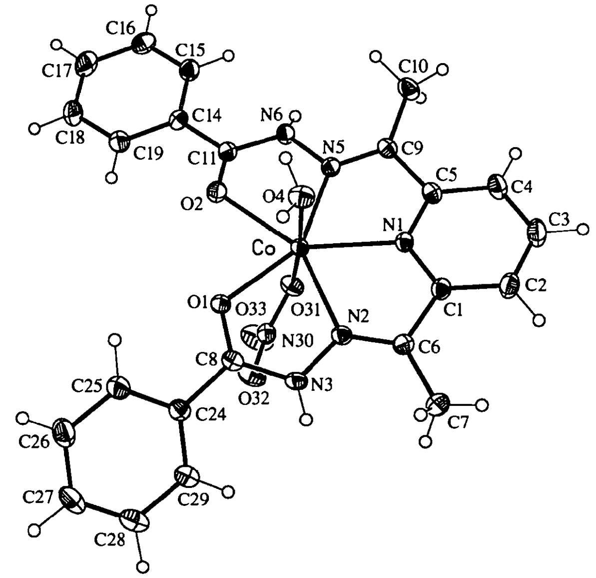


Fig. 1. The molecular structure of CoH_2dapbah showing the atom-numbering scheme and 50% probability ellipsoids.

The Co—O axial bond lengths of 2.129 (1) and 2.140 (1) Å are among the shortest found in the compilation given by BCPP, whereas, the Co—O bonds in the equatorial plane, 2.153 (1) and 2.224 (1) Å, are both longer and appear to be significantly different.

The biggest differences occur in the H-atom parameters and distances. Although the H atoms were refined in both studies, the low-temperature data appear to be more reasonable.

Experimental

Crystals of (I) were obtained by slow evaporation from an ethanol–water solution.

Crystal data

$[Co(NO_3)(C_{23}H_{21}N_5O_2)(H_2O)]NO_3$
$M_r = 600.41$
Monoclinic
$P2_1/n$
$a = 7.3170\ (1)$ Å
$b = 17.3766\ (1)$ Å
$c = 19.7908\ (3)$ Å
$\beta = 98.025\ (1)°$
$V = 2491.65\ (5)$ Å^3
$Z = 4$
$D_x = 1.601$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 5075 reflections
$\theta = 1.5–27.5°$
$\mu = 0.757$ mm^{-1}
$T = 173\ (2)$ K
Plate
0.26 × 0.20 × 0.19 mm
Orange–red

Data collection

SMART PLATFORM diffractometer
ω scans
Absorption correction: empirical *via* ψ scan (*SHELXTL*; Sheldrick, 1995)
$T_{min} = 0.82$, $T_{max} = 0.91$
15 896 measured reflections
5732 independent reflections

5075 observed reflections $[I > 2\sigma(I)]$
$R_{int} = 0.057$
$\theta_{max} = 27.5°$
$h = -10 \rightarrow 10$
$k = -23 \rightarrow 16$
$l = -23 \rightarrow 26$
Standard reflections: see below
intensity decay: <1%

Refinement

Refinement on F^2
$R(F) = 0.034$
$wR(F^2) = 0.095$
$S = 1.04$
5697 reflections
454 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0431P)^2 + 1.637P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = -0.008$

$\Delta\rho_{max} = 0.34$ e Å^{-3}
$\Delta\rho_{min} = -0.40$ e Å^{-3}
Extinction correction: *SHELXTL*
Extinction coefficient: 0.0035 (5)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	*y*	*z*	U_{eq}
Co	0.06270 (3)	0.70990 (1)	0.21777 (1)	0.01459 (8)
O1	0.1196 (2)	0.63839 (7)	0.13433 (6)	0.0172 (2)
O2	0.0433 (2)	0.59265 (7)	0.26132 (6)	0.0214 (3)
O4	0.3525 (2)	0.71039 (8)	0.25060 (7)	0.0234 (3)
O31	−0.2305 (2)	0.69943 (7)	0.19339 (6)	0.0219 (3)
O32	−0.2591 (2)	0.67768 (9)	0.08381 (6)	0.0277 (3)
O33	−0.4908 (2)	0.66064 (10)	0.13891 (7)	0.0377 (4)
O41	0.5135 (3)	0.61839 (9)	0.35835 (8)	0.0464 (4)
O42	0.3803 (2)	0.66325 (11)	0.44164 (9)	0.0442 (4)
O43	0.6743 (2)	0.67614 (8)	0.44304 (7)	0.0271 (3)
N1	0.0629 (2)	0.83134 (8)	0.24956 (7)	0.0166 (3)
N2	0.1011 (2)	0.78547 (8)	0.13168 (7)	0.0171 (3)
N3	0.1183 (2)	0.74738 (8)	0.07250 (7)	0.0186 (3)
N5	−0.0056 (2)	0.71906 (8)	0.32189 (7)	0.0171 (3)
N6	−0.0484 (2)	0.65175 (8)	0.35236 (7)	0.0188 (3)
N30	−0.3253 (2)	0.67869 (8)	0.13768 (7)	0.0192 (3)
N40	0.5192 (2)	0.65225 (9)	0.41420 (8)	0.0263 (3)
C1	0.0917 (2)	0.88720 (10)	0.20562 (8)	0.0184 (3)
C2	0.0993 (3)	0.96466 (10)	0.22493 (10)	0.0250 (4)
C3	0.0757 (3)	0.98311 (11)	0.29109 (10)	0.0293 (4)
C4	0.0454 (3)	0.92527 (10)	0.33635 (10)	0.0245 (4)
C5	0.0405 (2)	0.84939 (10)	0.31363 (8)	0.0180 (3)
C6	0.1135 (2)	0.85909 (10)	0.13628 (8)	0.0179 (3)
C7	0.1460 (3)	0.91358 (11)	0.08089 (10)	0.0265 (4)
C8	0.1201 (2)	0.66956 (10)	0.07828 (8)	0.0162 (3)
C9	0.0075 (2)	0.78218 (10)	0.35629 (8)	0.0179 (3)
C10	−0.0031 (3)	0.79068 (11)	0.43062 (9)	0.0242 (4)
C11	−0.0245 (2)	0.58782 (9)	0.31523 (8)	0.0175 (3)
C14	−0.0737 (2)	0.51282 (9)	0.34347 (8)	0.0184 (3)
C15	−0.1204 (3)	0.50505 (10)	0.40934 (9)	0.0219 (3)
C16	−0.1596 (3)	0.43302 (11)	0.43378 (10)	0.0260 (4)
C17	−0.1529 (3)	0.36828 (11)	0.39295 (11)	0.0289 (4)
C18	−0.1078 (3)	0.37541 (11)	0.32725 (10)	0.0287 (4)
C19	−0.0682 (3)	0.44743 (11)	0.30279 (9)	0.0235 (4)
C24	0.1237 (2)	0.62412 (10)	0.01502 (8)	0.0188 (3)
C25	0.2236 (3)	0.55565 (11)	0.01946 (9)	0.0234 (4)
C26	0.2239 (3)	0.50948 (11)	−0.03771 (10)	0.0288 (4)
C27	0.1237 (3)	0.53175 (12)	−0.09957 (10)	0.0298 (4)
C28	0.0261 (3)	0.60009 (12)	−0.10441 (9)	0.0289 (4)
C29	0.0249 (2)	0.64686 (11)	−0.04725 (9)	0.0222 (3)

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

Co—O4	2.129 (1)	Co—N2	2.200 (1)
Co—O31	2.140 (1)	Co—N1	2.202 (1)
Co—O1	2.153 (1)	Co—O2	2.224 (1)
Co—N5	2.193 (1)		
O4—Co—O31	173.44 (5)	O4—Co—N1	87.02 (5)
O4—Co—O1	86.88 (5)	O31—Co—N1	96.12 (5)
O31—Co—O1	94.19 (5)	N5—Co—N1	69.70 (5)
O4—Co—N5	93.49 (5)	N2—Co—N1	69.77 (5)
O31—Co—N5	82.25 (5)	O4—Co—O2	90.01 (5)
O4—Co—N2	90.27 (5)	O31—Co—O2	83.87 (5)
O31—Co—N2	96.22 (5)	O1—Co—O2	78.36 (4)
O1—Co—N2	72.02 (5)	N5—Co—O2	70.55 (5)

All H atoms were located in a difference Fourier synthesis and were refined isotropically. A hemisphere of frames (0.3° in ω) was collected. The first 50 frames were remeasured at the end of data collection to monitor instrument and crystal stability.

Data collection: *SMART* (Siemens, 1995). Cell refinement: *SMART* and *SAINT* (Siemens, 1995). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL* (Sheldrick, 1995). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL*.

KAA wishes to acknowledge the National Science Foundation for funding of the purchase of the X-ray equipment.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BK1270). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Triaqua(oxydiacetato-*O*,*O*′,*O*″)strontium(II) Monohydrate

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Abstract

The title complex, $[Sr(C_4H_4O_5)(H_2O)_3]_n.nH_2O$, consists of polymeric zigzag chains formed by SrO_9 units bridged by SrO_2Sr groups along the *b* axis, with Sr···Sr separations of 4.518 (1) Å. Each Sr atom is associated with three O atoms from a tridentate oxydiacetate ligand, three O atoms from adjacent carboxylate groups, one monodentate and one chelating, and three O atoms from metal-bound water molecules. The chains in the crystal are linked through an extensive hydrogen-bonding system into a complex three-dimensional network.