Table 2 (*cont.*)

W(1)—Cu—W(3)	57·85 (2)	Cl(3)—C(2)—C(1)	110·5 (6)
W(1)—Cu—I	139·97 (5)	N(1)—C(3)—C(4)	177 (2)
W(2)—CU—I	152·38 (5)	O—C—C(av.) (in OEt)	109 (4)†
W(3)—Cu—I	147·35 (6)		
S(1)—Cu—S(2)	104·43 (9)		
S(1)—Cu—S(3)	102·67 (8)		

* Anomalously short, probably because of disorder of C(51) and C(52).

† Standard e.s.d. $\sigma = [(\sum x^2 - n\overline{x}^2)/n]^{1/2}$.

except the C atoms of the OEt groups (355 variables). Final $R = 0{\cdot}33$, $wR = 0{\cdot}040$ and $S = 4{\cdot}56$, the function minimized was $\sum w(|F_o| - |F_c|)^2$, $w = 4F_o^2/\sigma(F_o^2)$, $\sigma^2(F_o^2) = [\sigma_o^2(F_o^2) + (0{\cdot}04F_o^2)^2]$ where $\sigma_o^2(F_o^2)$ is the standard deviation based on counting statistics. $(\Delta/\sigma)_{\max} = 0{\cdot}27$. In final difference synthesis function values were between $-1{\cdot}1$ and $1{\cdot}2$ Å^{-3}. For the 6230 unique reflections with $I \geq 3\sigma(I)$ the final parameters give $R = 0{\cdot}044$, $wR = 0{\cdot}048$, $S = 4{\cdot}2$. All calculations were performed on a VAX 785 computer using *SDP* (Frenz, 1978), the scattering factors were taken from Cromer & Waber (1974). *ORTEP*II (Johnson, 1976) was used to produce Fig. 1. The atomic coordinates and thermal parameters are listed in Table 1; important bond lengths and bond angles are given in Table 2.*

Related literature. $[CuW_3S_4\{S_2P(OC_2H_5)_2\}_3(I)(\mu_2\text{-}CH_3COO)(C_5H_5N)]$, a derivative of the title compound, has recently been reported (Zhan, Zheng, Wu & Lu, 1989).

This research was supported by grants from the Structural Chemistry Research Laboratory of the Fujian Institute of Research on the Structure of Matter, Chinese Academy of Sciences, and the National Science Foundation of China.

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* Lists of H-atom coordinates, full bond lengths and angles, structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52064 (44 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Acta Cryst. (1989). C**45**, 1992–1994

Structure of *trans*-Diacetato-*trans*-diaqua-*trans*-bis(isoquinoline)cobalt(II)

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(*Received* 28 *June* 1989; *accepted* 6 *July* 1989)

Abstract. $[Co(C_2H_3O_2)_2(C_9H_7N)_2(H_2O)_2]$, $M_r = 471{\cdot}4$, orthorhombic, $Pbca$, $a = 19{\cdot}484\ (2)$, $b = 13{\cdot}283\ (1)$, $c = 8{\cdot}352\ (1)$ Å, $V = 2161{\cdot}6$ Å^3, $Z = 4$, $D_x = 1{\cdot}448$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 8{\cdot}3$ cm^{-1}, $F(000) = 980$, $T = 291$ K, $R = 0{\cdot}0294$ for 1652 unique observed reflections. The complex, with octahedral coordination of Co, has exact inversion symmetry. The acetate ligands are monodentate, but the C—O bond lengths [1·255 (2) and 1·256 (2) Å] do not indicate localized single and double bonds within the ligand. The uncoordinated acetate O atom and the aqua ligand engage in a network of hydrogen bonds with O···O distances 2·657 (5) and 2·748 (5) Å to give sheets of connected molecules perpendicular to *a*.

Experimental. The compound was prepared electrolytically with a cobalt anode and platinum cathode in a solution of tetraethylammonium bromide and acetic acid in ethanol/water, and recrystallized from ethanol. Crystal size 0·23 × 0·45 × 0·58 mm, Stoe–Siemens diffractometer, unit-cell parameters from 2θ values of 32 reflections (20–25°) measured at $\pm\omega$. Data collection in ω/θ scan mode with on-line profile fitting (Clegg, 1981), $2\theta_{\max}$ 50°, index ranges h 0→23, k 0→15, l 0→9, no significant variation in intensities

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Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($Å^2 \times 10^4$)

$U_{eq} = \frac{1}{3}$(trace of the orthogonalized U_{ij} matrix).

	x	y	z	U_{eq}
Co	5000	5000	5000	241 (1)
C(1)	3642 (1)	6150 (1)	5334 (2)	318 (5)
N(2)	3980 (1)	5365 (1)	5882 (2)	300 (4)
C(3)	3642 (1)	4727 (1)	6887 (2)	354 (5)
C(4)	2973 (1)	4853 (1)	7297 (2)	402 (6)
C(5)	2596 (1)	5672 (1)	6691 (2)	340 (5)
C(6)	1887 (1)	5834 (1)	7000 (2)	481 (7)
C(7)	1558 (1)	6632 (2)	6335 (2)	530 (7)
C(8)	1907 (1)	7315 (2)	5351 (2)	493 (7)
C(9)	2590 (1)	7189 (2)	5030 (2)	415 (6)
C(10)	2945 (1)	6359 (1)	5692 (2)	309 (5)
O(11)	5315 (1)	4645 (1)	7319 (1)	333 (4)
O(12)	5063 (1)	3032 (1)	7669 (2)	488 (5)
C(13)	5311 (1)	3853 (1)	8139 (2)	307 (5)
C(14)	5623 (1)	3904 (2)	9785 (2)	527 (7)
O(15)	5326 (1)	6512 (1)	5292 (1)	320 (4)
H(15*a*)	5175 (9)	6969 (14)	5912 (25)	492 (54)
H(15*b*)	5185 (9)	6761 (14)	4359 (25)	460 (51)

Table 2. *Bond lengths* (Å) *and angles* (°)

Co—N(2)	2·175 (1)	Co—O(11)	2·086 (1)
Co—O(15)	2·121 (1)	C(1)—N(2)	1·315 (2)
C(1)—C(10)	1·418 (2)	N(2)—C(3)	1·363 (2)
C(3)—C(4)	1·359 (3)	C(4)—C(5)	1·407 (2)
C(5)—C(6)	1·421 (2)	C(5)—C(10)	1·411 (2)
C(6)—C(7)	1·358 (3)	C(7)—C(8)	1·400 (3)
C(8)—C(9)	1·368 (3)	C(9)—C(10)	1·414 (3)
O(11)—C(13)	1·255 (2)	O(12)—C(13)	1·256 (2)
C(13)—C(14)	1·504 (3)	O(15)—H(15*a*)	0·850 (19)
O(15)—H(15*b*)	0·891 (21)		
N(2)—Co—O(11)	90·3 (1)	N(2)—Co—O(15)	91·4 (1)
O(11)—Co—O(15)	91·1 (1)	N(2)—C(1)—C(10)	124·2 (1)
Co—N(2)—C(1)	121·1 (1)	Co—N(2)—C(3)	120·7 (1)
C(1)—N(2)—C(3)	117·8 (1)	N(2)—C(3)—C(4)	122·8 (2)
C(3)—C(4)—C(5)	120·4 (2)	C(4)—C(5)—C(6)	124·0 (2)
C(4)—C(5)—C(10)	117·5 (1)	C(6)—C(5)—C(10)	118·6 (2)
C(5)—C(6)—C(7)	120·1 (2)	C(6)—C(7)—C(8)	121·2 (2)
C(7)—C(8)—C(9)	120·5 (2)	C(8)—C(9)—C(10)	119·7 (2)
C(1)—C(10)—C(5)	117·4 (1)	C(1)—C(10)—C(9)	122·6 (2)
C(5)—C(10)—C(9)	120·0 (1)	Co—O(11)—C(13)	134·0 (1)
O(11)—C(13)—O(12)	124·0 (2)	O(11)—C(13)—C(14)	117·3 (2)
O(12)—C(13)—C(14)	118·7 (2)	Co—O(15)—H(15*a*)	129·9 (12)
Co—O(15)—H(15*b*)	99·1 (12)	H(15*a*)—O(15)—H(15*b*)	99·2 (18)

of three standard reflections, no absorption correction, 1889 unique reflections measured, 1652 with $F > 4\sigma(F)$.

Structure solution by Patterson and difference syntheses, blocked-cascade least-squares refinement on F, weighting $w^{-1} = \sigma^2(F) + 0{\cdot}00024F^2$, anisotropic thermal parameters for all non-H atoms, H_2O H atoms freely refined, other H atoms constrained [C—H 0·96 Å, H—C—H 109·5°, aromatic H on ring angle external bisectors, twofold rotational disorder of acetate methyl group, $U(\mathrm{H}) = 1{\cdot}2U_{eq}(\mathrm{C})$], extinction parameter $x = 5{\cdot}1\ (5) \times 10^{-6}\ [F_c' = F_c/(1 + xF_c^2/\sin 2\theta)^{1/4}]$. $R = 0{\cdot}0294$, $wR = 0{\cdot}0395$ for 154 parameters, slope of normal probability plot = 1·52, mean $\Delta/\sigma = 0{\cdot}005$, max. $\Delta/\sigma = 0{\cdot}024$, $(\Delta\rho)_{max} = 0{\cdot}32$, $(\Delta\rho)_{min} = -0{\cdot}38$ e Å^{-3}. Scattering factors from *International Tables for X-ray Crystallography* (1974); *SHELXTL* (Sheldrick, 1985) and local computer programs.

Refined atomic coordinates and equivalent isotropic thermal parameters are given in Table 1, bond lengths and angles in Table 2.* Fig. 1 shows the molecular structure, and Fig. 2 the packing arrangement and hydrogen bonding.

Related literature. The structures of several compounds of formula $Co(O_2CR)_2L_2$ have been reported, in which L is an imidazole-based ligand; these display either an essentially tetrahedral coordination of Co with monodentate carboxylate ligands, or distorted octahedral coordination with bidentate carboxylates (Horrocks, Ishley, Holmquist & Thompson, 1980; Horrocks, Ishley & Whittle, 1982*a*,*b*). A number of anhydrous mononuclear, binuclear and trinuclear Co complexes with carboxylate and heterocyclic nitrogen base ligands (including pyridine and quinoline) have been studied by Catterick & Thornton (1976). In the diaqua complex $Co(H_2O)_2(CO_3)(imidazole)_2$ (Baraniak, Freeman, James & Nockolds, 1970), the single

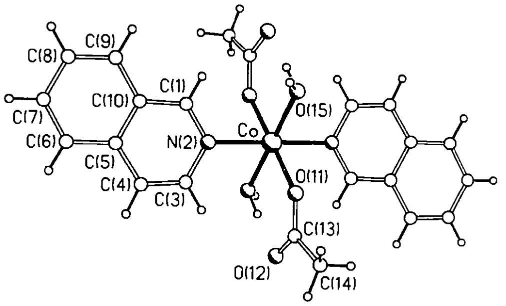

Fig. 1. The molecular structure with the numbering scheme.

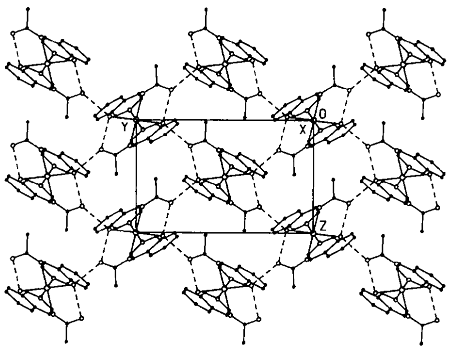

Fig. 2. The sheet structure formed by hydrogen bonding, as seen in projection down the *a* axis.

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* Lists of structure factors, anisotropic thermal parameters and constrained H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52097 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

carbonato ligand is bidentate, in contrast to the pair of monodentate acetate ligands reported here.

We thank SERC for a research grant and R. McLaughlan for technical assistance.

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Acta Cryst. (1989). **C45**, 1994–1996

Structure of $Mn(CO)_3[C_5H_4CHMeC_5H_4]Mn(CO)_3$ and its Benzene Solvate

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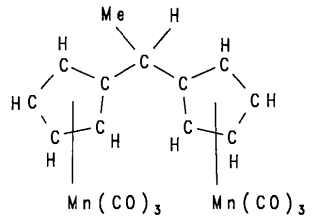
(*Received* 12 *June* 1989; *accepted* 4 *July* 1989)

Abstract. (I) 1,1-Bis(η^5-cyclopentadienyl)ethanebis(tricarbonylmanganese), $C_{18}H_{12}Mn_2O_6$, $M_r = 434{\cdot}2$, monoclinic, $P2_1/c$, $a = 12{\cdot}045\,(2)$, $b = 14{\cdot}169\,(2)$, $c = 11{\cdot}468\,(4)$ Å, $\beta = 115{\cdot}91\,(2)°$, $V = 1760\,(2)$ Å^3, $Z = 4$, $D_x = 1{\cdot}64$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 14{\cdot}1$ cm^{-1}, $F(000) = 872$, $T = 293$ K, $R = 0{\cdot}043$ for 1251 reflections with $F_o^2 > 3\sigma(F_o^2)$. (II), the 0·5 benzene solvate of (I), $C_{18}H_{12}Mn_2O_6.\frac{1}{2}C_6H_6$, $M_r = 473{\cdot}2$, triclinic, $P\bar{1}$, $a = 7{\cdot}850\,(3)$, $b = 9{\cdot}205\,(4)$, $c = 14{\cdot}291\,(4)$ Å, $\alpha = 85{\cdot}42\,(3)$, $\beta = 85{\cdot}79\,(3)$, $\gamma = 75{\cdot}50\,(3)°$, $V = 995\,(2)$ Å^3, $Z = 2$, $D_x = 1{\cdot}58$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 12{\cdot}5$ cm^{-1}, $F(000) = 478$, $T = 293$ K, $R = 0{\cdot}028$ for 1800 reflections with $F_o^2 > 3\sigma(F_o^2)$. These two crystals show large differences of conformation of the dicyclopentadiene ligand: in (I) the cp rings are each within 9·1° of being perpendicular to the central C(cp)—C—C(cp) plane and the $Mn(CO)_3$ units are both on the outside of the ligand unit, while in (II) one cp ring is perpendicular to the C(cp)—C—C(cp) plane while the other is twisted 32·6° away from the perpendicular orientation; the $Mn(CO)_3$ unit bonded to the latter cp ring is on the inside of the fold of the ligand.

Experimental. The title complex (I) was obtained by the procedure described by Kolobova, Valueva, Anisimov & Suleimanov (1978). Yellow crystals of (I) obtained from pentane/benzene (20:1) solution and of (II) from benzene solution by slow evaporation of the solvents. Data crystal was 0·08 × 0·26 × 0·32 mm for (I) and approximately 0·5 × 0·5 × 0·5 mm for (II); both were mounted with epoxy on a glass fiber. Intensities measured with an Enraf–Nonius CAD-4 diffractometer using ω–2θ scans of 4–16° min^{-1} in θ. Unit cell for each determined from least-squares analysis of angle data for 25 reflections with $14 < 2\theta < 20°$. Analytical absorption correction for (I) varied from 0·74 to 1·00; an absorption correction based on ψ scans for (II) varied from 0·95 to 1·00. Data collected to $(\sin\theta)/\lambda$ of 0·53 Å^{-1} for both crystals; $0 < h < 12$, $0 < k < 14$, $-12 < l < 12$ for (I) and $-8 < h < 8$, $0 < k < 9$, $-15 < l < 15$ for (II). Three standard reflections [$40\bar{4}$, $24\bar{4}$, 160 for (I); 007, 040, 300 for (II)] indicated crystal decomposition of less than 2·5% over 20·1 h (I) and 12·7 h (II) of data collection. For (I): 2390 reflections measured, 2147 unique ($R_{\rm int} = 0{\cdot}033$), 896 reflections with $I < 3\sigma(I)$ where $\sigma^2(I) = \sigma_{\rm cs}^2(I) + (0{\cdot}05I)^2$; $\sigma_{\rm cs}(I)$ is standard deviation of I based on counting statistics. For (II): 2612 reflections measured, 2428 unique ($R_{\rm int} = 0{\cdot}014$), 628 with $I < 3\sigma(I)$. Both structures solved by direct methods using *MULTAN*11/82 (Main, Fiske,



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