was used to produce the view of the molecule (Fig. 1). The atom coordinates and thermal parameters are listed in Table 1; the important bond lengths and bond angles are given in Table 2.*

Related literature. A related derivative of the title compound $[CuMo_3S_4\{S_2P(OC_2H_5)_3\}(I)(\mu_2\text{-}CH_3COO)\{HCON(CH_3)_2\}]$ has been reported (Wu *et al.*, 1987).

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* Fuller lists of bond lengths and angles, and lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52065 (40 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

CROMER, D. T. & WABER, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Tables 2.2A, 2.3.1. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

FRENZ, B. A. (1978). *The Enraf–Nonius CAD-4 SDP – A Real-Time System for Concurrent X-ray Data Collection and Crystal Structure Solution.* In *Computing in Crystallography*, edited by H. SCHENK, R. OLTHOF-HAZEKAMP, H. VAN KONINGSVELD & G. C. BASSI, pp. 64–71. Delft Univ. Press.

JOHNSON, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1982). *MULTAN*11/82. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

WU, X. T., LU, L. F., ZHU, N. Y., WU, Q. J. & LU, J. X. (1987). *Inorg. Chim. Acta*, **113**, 43–47.

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Structure of 2-(Acetonitrile)-2,3,4-tris(diethyl dithiophosphato-*S*,*S*′)-1-iodo-tetrakis(μ_3-sulfido)-3,4-μ_2-trichloroacetato-copper(I)tritungsten(IV)

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Abstract. $[CuW_3S_4(I)(C_2Cl_3O_2)(C_4H_{10}O_2PS_2)_3(C_2H_3N)]$, $M_r = 1629$, triclinic, $P\bar{1}$, $a = 11{\cdot}684\ (6)$, $b = 14{\cdot}243\ (5)$, $c = 15{\cdot}455\ (4)$ Å, $\alpha = 103{\cdot}88\ (3)$, $\beta = 109{\cdot}01\ (3)$, $\gamma = 65{\cdot}64\ (4)°$, $V = 2198{\cdot}4$ Å^3, $Z = 2$, $D_x = 2{\cdot}46$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 99{\cdot}5$ cm^{-1}, $F(000) = 1453$, $T = 296$ K, $R = 0{\cdot}033$ for 3659 unique observed reflections with $I \geq 10\sigma(I)$. Each W atom is octahedrally coordinated by three μ_3-S atoms (W—μ_3-S av. 2·334 Å) and an $S_2P(OEt)_2$(dtp) chelating ligand [W—S_t(dtp) 2·517 Å]. The octahedra surrounding the W(2) and W(3) atoms are completed by a CCl_3COO bridging ligand (W—O_b 2·200 Å), and that surrounding W(1) by a CH_3CN molecule (W—N 2·211 Å). The Cu atom is tetrahedrally coordinated by three μ_3-S atoms (Cu—μ_3-S av. 2·300 Å) and one I atom. There are some distortions in the cubane-like $(W_3CuS_4)^{5+}$ core, with three W—W bonds and three weak W—Cu bonds: W—W (av.) 2·728, W—Cu(av.) 2·874 Å. The molecule can alternatively be described in terms of a W_3Cu tetrahedral cluster with a μ-S atom bridging each triangular face.

Experimental. Crystals of the title compound were prepared by the method described by Zhan (1989). Crystal dimensions 0·25 × 0·20 × 0·25 mm. Data were collected using a CAD-4 κ-geometry diffractometer, $\omega/2\theta$ scans, scan speed varied from 1 to 7° min^{-1} (in ω), the scan width was $(0{\cdot}40 + 0{\cdot}35\tan\theta)°$. Cell constants were obtained by least-squares analy-

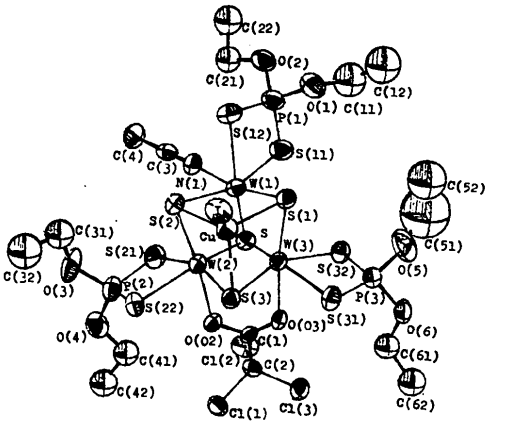

Fig. 1. Drawing of the title compound with thermal ellipsoids.

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Table 1. *Atomic coordinates and thermal parameters* ($Å^2$)

Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameter defined as: $(4/3)[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)]$.

	x	*y*	*z*	B_{eq}
W(1)	0·68674 (7)	0·31987 (6)	0·67410(5)	3·95 (2)
W(2)	0·76066 (7)	0·43738 (5)	0·83496 (5)	3·63 (2)
W(3)	0·91030 (6)	0·23596 (5)	0·80673 (4)	3·46 (2)
I	1·0156 (1)	0·4009 (1)	0·59783 (8)	5·76 (4)
Cu	0·9050 (2)	0·3700 (2)	0·6933 (1)	4·66 (6)
Cl(1)	0·9505 (5)	0·4166 (3)	1·1835 (3)	5·3 (1)
Cl(2)	0·7667 (5)	0·3149 (4)	1·1098 (3)	6.7 (1)
Cl(3)	1·0400 (5)	0·1960 (4)	1·1426 (3)	6·1 (2)
S	0·6983 (4)	0·2976 (3)	0·8222 (3)	3·9 (1)
S(1)	0·8875 (4)	0·2106 (3)	0·6492 (3)	4·1 (1)
S(2)	0·6934 (4)	0·4853 (3)	0·6873 (3)	4·2 (1)
S(3)	0·9854 (4)	0·3736 (3)	0·8503 (3)	3·8 (1)
S(11)	0·5925 (5)	0·1804 (4)	0·6236 (4)	5·9 (1)
S(12)	0·6068 (5)	0·3232 (4)	0·4988 (3)	5·7 (2)
S(21)	0·5542 (5)	0·5384 (4)	0·8802 (4)	5·7 (2)
S(22)	0·7859 (5)	0·6106 (4)	0·9033 (3)	5·2 (1)
S(31)	1·1507 (4)	0·1281 (4)	0·8317 (3)	5·0 (1)
S(32)	0·9158 (5)	0·0616 (3)	0·8142 (3)	4·8 (1)
P(1)	0·5671 (5)	0·1997 (4)	0·4928 (4)	5·8 (2)
P(2)	0·6102 (5)	0·6612 (4)	0·9241 (4)	5·6 (2)
P(3)	1·1050 (5)	0·0052 (4)	0·8193 (3)	4·8 (1)
N(1)	0·480 (1)	0·416 (1)	0·665 (1)	5·1 (4)
O(02)	0·8318 (9)	0·4091 (8)	0·9816 (7)	3·9 (3)
O(03)	0·9537 (9)	0·2406 (7)	0·9570 (7)	3·7 (3)
O(1)	0·658 (1)	0·103 (1)	0·4412 (9)	7·9 (5)
O(2)	0·429 (1)	0·206 (1)	0·4300 (9)	6·9 (4)
O(3)	0·510 (1)	0·759 (1)	0·878 (1)	8·9 (5)
O(4)	0·603 (1)	0·708 (1)	1·0256 (9)	7·1 (5)
O(5)	1·139 (1)	−0·073 (1)	0·731 (1)	9·4 (6)
O(6)	1·191 (1)	−0·0670 (9)	0·8943 (8)	5·8 (4)
C(1)	0·903 (1)	0·320 (1)	1·007 (1)	3·9 (4)
C(2)	0·916 (1)	0·314 (1)	1·107 (1)	3·8 (4)
C(3)	0·375 (2)	0·477 (1)	0·657 (1)	4·6 (5)
C(4)	0·242 (2)	0·556 (2)	0·643 (1)	6·7 (7)
C(11)	0·651 (3)	−0·002 (2)	0·442 (2)	12 (1)*
C(12)	0·733 (3)	−0·080 (3)	0·389 (2)	14 (1)*
C(21)	0·312 (2)	0·291 (2)	0·449 (2)	7·6 (6)*
C(22)	0·197 (3)	0·276 (2)	0·378 (2)	9·9 (8)*
C(31)	0·500 (2)	0·781 (2)	0·789 (2)	9·3 (7)*
C(32)	0·529 (3)	0·872 (2)	0·806 (2)	13 (1)*
C(41)	0·664 (2)	0·640 (2)	1·101 (2)	8·0 (6)*
C(42)	0·646 (2)	0·707 (2)	1·189 (2)	9·1 (7)*
C(61)	1·190 (2)	−0·025 (2)	0·990 (1)	7·0 (6)*
C(62)	1·317 (2)	−0·073 (2)	1·049 (2)	8·8 (7)*
C(51)	1·081 (6)	−0·140 (5)	0·677 (4)	28 (3)*
C(52)	1·061 (4)	−0·144 (3)	0·596 (3)	16 (1)*

* Starred atoms were refined isotropically.

sis based on 25 reflections with $26 < 2\theta < 27°$. The intensities were corrected empirically for absorption (maximum and minimum transmission factors 1·61 and 0·76 respectively) and for Lp effects to give a total of 8125 intensities, up to a maximum 2θ of 50° ($0 < h < 13$, $-15 < k < 16$, $-18 < l < 17$). 3659 reflections with $I \geq 10\sigma(I)$ were used in the analysis. Three standard reflections were measured periodically, but no significant variation in intensity was observed.

The structure was solved by direct methods using *MULTAN*11/82 (Main *et al.*, 1982), seven heavy atoms (W, Cu, I) were located in the *E* map. The remaining non-H atoms were located in succeeding difference syntheses. H atoms were placed in geometrically calculated positions (C—H 0·95 Å), which were not subsequently refined. The structure was refined by full-matrix least-squares techniques, with anisotropic thermal parameters for all non-H atoms

Table 2. *Selected bond distances* (Å) *and bond angles* (°)

W(1)—W(2)	2·7433 (6)	Cl(3)—C(2)	1·770 (8)
W(1)—W(3)	2·7503 (6)	S(11)—P(1)	2·014 (4)
W(1)—Cu	2·830 (1)	S(12)—P(1)	1·971 (4)
W(1)—S	2·339 (2)	S(21)—P(2)	2·008 (5)
W(1)—S(1)	2·315 (2)	S(22)—P(2)	1·984 (4)
W(1)—S(2)	2·348 (2)	S(31)—P(3)	1·981 (4)
W(1)—S(11)	2·504 (3)	S(32)—P(3)	1·997 (4)
W(1)—S(12)	2·567 (2)	P(1)—O(1)	1·572 (7)
W(1)—N(1)	2·211 (8)	P(1)—O(2)	1·572 (7)
W(2)—W(3)	2·6899 (6)	P(2)—O(3)	1·558 (7)
W(2)—Cu	2·935 (1)	P(2)—O(4)	1·564 (6)
W(2)—S	2·334 (2)	P(3)—O(5)	1·582 (7)
W(2)—S(2)	2·330 (2)	P(3)—O(6)	1·541 (6)
W(2)—S(3)	2·346 (2)	N(1)—C(3)	1·16 (2)
W(2)—S(21)	2·476 (3)	O(02)—C(1)	1·273 (9)
W(2)—S(22)	2·537 (2)	O(03)—C(1)	1·248 (9)
W(2)—O(02)	2·219 (6)	O(1)—C(11)	1·53 (2)
W(3)—Cu	2·856 (1)	O(2)—C(21)	1·46 (1)
W(3)—S	2·336 (2)	O(3)—C(31)	1·44 (1)
W(3)—S(1)	2·316 (2)	O(4)—C(41)	1·49 (2)
W(3)—S(3)	2·339 (2)	O(5)—C(51)	1·37 (4)
W(3)—S(31)	2·533 (2)	O(6)—C(61)	1·46 (1)
W(3)—S(32)	2·487 (2)	C(1)—C(2)	1·52 (1)
W(3)—O(03)	2·200 (5)	C(3)—C(4)	1·48 (1)
I—Cu	2·463 (1)	C(11)—C(12)	1·42 (3)
Cu—S(1)	2·284 (3)	C(21)—C(22)	1·49 (2)
Cu—S(2)	2·322 (3)	C(31)—C(32)	1·42 (2)
Cu—S(3)	2·294 (2)	C(41)—C(42)	1·49 (2)
Cl(1)—C(2)	1·746 (8)	C(51)—C(52)	1·19 (4)*
Cl(2)—C(2)	1·756 (8)	C(61)—C(62)	1·44 (3)

W(2)—W(1)—W(3)	58·63 (1)	S(2)—Cu—S(3)	99·21 (9)
W(2)—W(1)—Cu	63·55 (2)	W(1)—S—W(2)	71·92 (6)
W(3)—W(1)—Cu	61·55 (2)	W(1)—S—W(3)	72·10 (6)
S—W(1)—S(1)	104·69 (8)	W(2)—S—W(3)	70·36 (6)
S—W(1)—S(2)	105·79 (8)	W(1)—S(1)—W(3)	72·85 (6)
S—W(1)—S(11)	83·98 (8)	W(1)—S(1)—Cu	75·95 (7)
S—W(1)—S(12)	159·76 (8)	W(3)—S(1)—Cu	76·76 (7)
S—W(1)—N(1)	85·9 (3)	W(1)—S(2)—W(2)	71·81 (7)
S(1)—W(1)—S(2)	102·62 (9)	W(1)—S(2)—Cu	74·60 (7)
S(1)—W(1)—S(11)	94·14 (9)	W(2)—S(2)—Cu	78·26 (7)
S(1)—W(1)—S(12)	86·43 (9)	W(2)—S(3)—W(3)	70·09 (6)
S(1)—W(1)—N(1)	167·6 (2)	W(2)—S(3)—Cu	78·47 (7)
S(2)—W(1)—S(11)	157·48 (9)	W(3)—S(3)—Cu	76·11 (8)
S(2)—W(1)—S(12)	87·73 (8)	W(1)—S(11)—P(1)	87·4 (1)
S(2)—W(1)—N(1)	80·2 (3)	W(1)—S(12)—P(1)	86·5 (1)
S(11)—W(1)—S(12)	78·31 (9)	W(2)—S(21)—P(2)	89·1 (1)
S(11)—W(1)—N(1)	80·3 (2)	W(2)—S(22)—P(2)	87·9 (1)
S(12)—W(1)—N(1)	81·6 (3)	W(3)—S(31)—P(3)	87·3 (1)
W(1)—W(2)—W(3)	60·81 (1)	W(3)—S(32)—P(3)	88·2 (1)
W(1)—W(2)—Cu	59·67 (2)	S(11)—P(1)—S(12)	107·0 (2)
W(3)—W(2)—Cu	60·83 (2)	S(11)—P(1)—O(1)	112·5 (3)
S—W(2)—S(2)	106·51 (8)	S(11)—P(1)—O(2)	112·7 (3)
S—W(2)—S(3)	108·54 (8)	S(12)—P(1)—O(1)	109·8 (4)
S—W(2)—S(21)	82·88 (9)	S(12)—P(1)—O(2)	113·5 (3)
S—W(2)—S(22)	156·45 (8)	O(1)—P(1)—O(2)	101·5 (4)
S—W(2)—O(02)	80·7 (2)	S(21)—P(2)—S(22)	104·9 (2)
S(2)—W(2)—S(3)	97·49 (8)	S(21)—P(2)—O(3)	112·7 (4)
S(2)—W(2)—S(21)	98·01 (8)	S(21)—P(2)—O(4)	112·6 (3)
S(2)—W(2)—S(22)	90·06 (9)	S(22)—P(2)—O(3)	115·2 (4)
S(2)—W(2)—O(02)	172·9 (2)	S(22)—P(2)—O(4)	114·7 (3)
S(3)—W(2)—S(21)	157·15 (8)	O(3)—P(2)—O(4)	97·2 (4)
S(3)—W(2)—S(22)	85·09 (9)	S(31)—P(3)—S(32)	105·6 (1)
S(3)—W(2)—O(02)	80·6 (1)	S(31)—P(3)—O(5)	111·1 (4)
S(21)—W(2)—S(22)	78·22 (9)	S(31)—P(3)—O(6)	114·2 (3)
S(21)—W(2)—O(02)	81·9 (1)	S(32)—P(3)—O(5)	113·0 (4)
S(22)—W(2)—O(02)	82·9 (2)	S(32)—P(3)—O(6)	114·0 (4)
W(1)—W(3)—W(2)	60·56 (1)	O(5)—P(3)—O(6)	99·2 (4)
W(1)—W(3)—Cu	60·59 (3)	W(1)—N(1)—C(3)	170·7 (8)
S—W(3)—S(1)	104·73 (8)	W(2)—O(02)—C(1)	122·9 (5)
S—W(3)—S(3)	108·75 (8)	W(3)—O(03)—C(1)	122·5 (5)
S—W(3)—(S31)	159·14 (9)	P(1)—O(1)—C(11)	116·0 (9)
S—W(3)—S(32)	85·52 (8)	P(1)—O(2)—C(21)	120·8 (6)
S—W(3)—O(03)	80·9 (2)	P(2)—O(3)—C(31)	125·3 (8)
S(1)—W(3)—S(3)	100·33 (8)	P(2)—O(4)—O(41)	120·9 (6)
S(1)—W(3)—S(31)	89·85 (9)	P(3)—O(5)—C(51)	130 (2)
S(1)—W(3)—S(32)	95·13 (8)	P(3)—O(6)—C(61)	118·2 (7)
S(1)—W(3)—O(03)	172·5 (2)	O(02)—C(1)—O(03)	125·7 (8)
S(3)—W(3)—S(31)	82·57 (8)	O(02)—C(1)—C(2)	113·8 (8)
S(3)—W(3)—S(32)	155·27 (8)	O(03)—C(1)—C(2)	120·4 (7)
S(3)—W(3)—O(03)	82·3 (1)	Cl(1)—C(2)—Cl(2)	109·6 (5)
S(31)—W(3)—S(32)	78·26 (8)	Cl(1)—C(2)—Cl(3)	108·4 (4)
S(31)—W(3)—O(03)	83·5 (2)	Cl(1)—C(2)—C(1)	113·8 (5)
S(32)—W(3)—O(03)	80·2 (2)	Cl(2)—C(2)—Cl(3)	108·8 (4)
W(1)—Cu—W(2)	56·79 (2)	Cl(2)—C(2)—C(1)	105·7 (5)

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. $\sigma = [(\sum x^2 - n\bar{x}^2)/n]^{1/2}$.

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 $\sum w(|F_o| - |F_o|)^2$, $w = 4F_o^2/\sigma(F_o^2)$, $\sigma^2(F_o^2) = [\sigma_o^2(F_o^2) + (0{\cdot}04F_o^2)^2]$ 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.*

* 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.

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).

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References

CROMER, D.T. & WABER, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV., Tables 2.2A, 2.3.1.Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

FRENZ, B. A. (1978). *The Enraf–Nonius CAD-4 SDP – A Real-Time System for Concurrent X-ray Data Collection and Crystal Structure Determination.* In *Computing in Crystallography*, edited by H. SCHENK, R. OLTHOF-HAZEKAMP, H. VAN KONINGSVELD & G. C. BASSI, pp. 64–71. Delft Univ. Press.

JOHNSON, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

MAIN, P., FISKE, S.J., HULL, S.E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1982). *MULTAN*11/82. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

ZHAN, H. Q. (1989). MS Thesis, Fujian Institute of Research on the Structure of Matter, People's Republic of China.

ZHAN, H. Q., ZHENG, Y. F., WU, X. T. & LU, J. X. (1989). *Inorg. Chim. Acta*, **156**, 277–280.

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

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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}, λ(Mo $K\alpha$) = 0·71069 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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