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Structures of Di-μ-acetato-(*O,O'*)-μ-aqua-bis[acetato(*N,N,N',N'*-tetramethylethylenediamine)cobalt(II)] and μ-Aqua-di-μ-chloroacetato-(*O,O'*)-bis[chloroacetato(*N,N,N',N'*-tetramethylethylenediamine)cobalt(II)]

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(*Received* 26 *October* 1981; *accepted* 15 *December* 1981)

Abstract. $C_{20}H_{46}Co_2N_4O_9$, orthorhombic, *Pbca*, $a = 12·056$ (3), $b = 15·917$ (5), $c = 31·330$ (10) Å, $M_r = 604·6$, $Z = 8$, $D_c = 1·336$ Mg m^{-3}, final $R = 0·038$ for 3134 observed reflections; $C_{20}H_{42}Cl_4Co_2N_4O_9$, monoclinic, $P2/n$, $a = 10·834$ (3), $b = 8·328$ (3), $c = 18·749$ (3) Å, $\beta = 105·9$ (2)°, $M_r = 742·4$, $Z = 2$, $D_c = 1·515$ Mg m^{-3}, final $R = 0·033$ for 2248 observed reflections. The complexes are octahedral, with a dimeric structure in which Co atoms are joined by one water molecule and two carboxylate groups.

Introduction. This work is a continuation of earlier studies on the crystal structures of Cu^{II} and Ni^{II} carboxylate complexes with *N,N,N',N'*-tetramethylethylenediamine as the second ligand. The Cu^{II} complexes are monomeric and five- or six-coordinated depending upon the carboxylate ligands (Turpeinen, Ahlgrén & Hämäläinen, 1978; Ahlgrén, Hämäläinen & Turpeinen, 1978), and the Ni^{II} complexes are octahedral, with a dimeric structure in which bridging occurs *via* the water O atom and two carboxylate groups (Ahlgrén, Turpeinen & Hämäläinen, 1978). This type of bridging system is novel in the first-row transition elements and is of interest because of possible low-dimensional magnetic interaction.

The title compounds $C_{20}H_{46}Co_2N_4O_9$ (I) and $C_{20}H_{42}Cl_4Co_2N_4O_9$ (II) were obtained by mixing equal amounts of the appropriate Co^{II} carboxylate and *N,N,N',N'*-tetramethylethylenediamine. Compound (I) was crystallized from cyclohexane and compound (II) from aqueous ethanol solution. Unit-cell parameters and intensity data for both compounds were obtained with a Syntex $P2_1$ diffractometer and graphite-monochromatized Mo $K\alpha$ radiation. The ω-scan technique was used and the scan rate varied from 2·0 to 30·0° min^{-1} depending upon peak intensity. The total numbers of reflections collected for (I) and (II) were 5286 and 3235 respectively, of which 3134 and 2248 were considered observed, having $I > 3\sigma(I)$. Systematically absent reflections indicated the space groups to be *Pbca* for (I) and *Pn* or *P2/n* for (II); *P2/n* was verified by successful refinement. No absorption correction was applied.

The structures were solved with *MULTAN* 78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and Fourier methods of the XRAY 76 program system (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976) and refined by full-matrix least squares minimizing $\sum w(|F_o| - |F_c|)^2$ with unit weights for (I) and the weighting scheme $w = 1/(40 + |F_o| + 0·011|F_o|^2)$ for (II). After all non-hydrogen atoms were located difference Fourier maps showed the positions of the H atoms, which were then refined isotropically together with the anisotropic

0567-7408/82/051580-04$01.00

Table 1. *Atomic coordinates* ($\times 10^4$ *for nonhydrogen atoms and* $\times 10^3$ *for hydrogen atoms*) *of* (I)

For nonhydrogen atoms $B_{eq} = \frac{1}{3}(B_{11} + B_{22} + B_{33})$.

	x	y	z	B_{eq}/B (Å^2)
Co(1)	3575 (1)	1943 (1)	3457 (1)	3·3 (1)
Co(2)	5670 (1)	3070 (1)	4040 (1)	3·8 (1)
O(1)	3979 (3)	1278 (2)	4000 (1)	5·7 (2)
O(2)	5259 (3)	1999 (2)	4365 (1)	5·6 (2)
O(3)	2862 (3)	2895 (2)	3799 (1)	5·2 (2)
O(4)	4142 (3)	3625 (2)	4145 (1)	5·7 (2)
O(5)	4229 (3)	953 (2)	3103 (1)	5·0 (2)
O(6)	6019 (2)	1059 (2)	3245 (1)	6·2 (2)
O(7)	6196 (3)	4108 (2)	3693 (1)	5·3 (2)
O(8)	5710 (3)	3793 (2)	3033 (1)	6·1 (2)
O(9)	5205 (2)	2486 (2)	3460 (1)	3·4 (2)
N(1)	2996 (3)	2526 (2)	2854 (1)	3·9 (2)
N(2)	1919 (3)	1335 (3)	3426 (1)	4·6 (2)
N(3)	7481 (3)	2613 (3)	4046 (1)	5·4 (3)
N(4)	6219 (4)	3655 (3)	4647 (1)	6·3 (3)
C(1)	2011 (5)	2036 (4)	2731 (2)	6·0 (4)
C(2)	1332 (5)	1808 (4)	3099 (2)	6·8 (4)
C(3)	3813 (5)	2451 (4)	2506 (2)	6·0 (4)
C(4)	2735 (5)	3431 (3)	2898 (2)	6·5 (4)
C(5)	1987 (5)	444 (4)	3311 (3)	8·2 (4)
C(6)	1330 (5)	1386 (5)	3835 (2)	8·0 (4)
C(7)	7998 (5)	3142 (5)	4366 (2)	8·7 (4)
C(8)	7309 (6)	3287 (5)	4745 (2)	9·7 (5)
C(9)	8012 (5)	2707 (5)	3634 (2)	8·5 (4)
C(10)	7614 (6)	1726 (4)	4167 (2)	8·0 (4)
C(11)	6269 (7)	4562 (4)	4608 (2)	10·0 (5)
C(12)	5447 (6)	3440 (5)	4997 (2)	8·9 (4)
C(13)	4676 (4)	1362 (3)	4285 (2)	4·9 (3)
C(14)	4892 (6)	623 (4)	4569 (2)	9·7 (5)
C(15)	3179 (4)	3430 (3)	4063 (1)	4·4 (3)
C(16)	2279 (5)	3891 (4)	4304 (2)	7·2 (3)
C(17)	5192 (4)	667 (3)	3125 (2)	4·7 (3)
C(18)	5339 (6)	−240 (4)	2983 (3)	8·6 (4)
C(19)	6078 (4)	4284 (3)	3308 (2)	4·4 (3)
C(20)	6426 (5)	5153 (3)	3158 (2)	6·8 (3)
H(1)	156 (3)	235 (2)	250 (2)	7 (1)
H(2)	225 (5)	142 (3)	258 (2)	7 (1)
H(3)	68 (5)	155 (3)	303 (2)	10 (1)
H(4)	103 (5)	234 (4)	327 (2)	7 (1)
H(5)	407 (4)	176 (3)	249 (2)	6 (1)
H(6)	346 (4)	267 (3)	218 (2)	12 (1)
H(7)	453 (4)	279 (3)	258 (2)	8 (1)
H(8)	241 (5)	359 (3)	261 (1)	6 (1)
H(9)	215 (5)	345 (3)	314 (2)	10 (1)
H(10)	346 (4)	373 (3)	296 (2)	12 (2)
H(11)	128 (5)	15 (4)	329 (2)	3 (1)
H(12)	238 (5)	39 (3)	305 (2)	12 (2)
H(13)	240 (5)	14 (4)	353 (2)	10 (1)
H(14)	125 (4)	202 (3)	388 (1)	10 (1)
H(15)	57 (4)	112 (3)	385 (2)	8 (1)
H(16)	183 (5)	117 (4)	401 (2)	9 (1)
H(17)	883 (5)	301 (4)	454 (2)	14 (2)
H(18)	807 (5)	377 (4)	420 (2)	8 (1)
H(19)	774 (4)	360 (3)	495 (2)	11 (1)
H(20)	716 (5)	264 (4)	490 (2)	7 (1)
H(21)	795 (4)	342 (3)	357 (1)	9 (1)
H(22)	891 (4)	255 (3)	360 (2)	10 (1)
H(23)	772 (6)	242 (4)	334 (2)	9 (1)
H(24)	847 (4)	144 (3)	416 (2)	11 (1)
H(25)	722 (4)	152 (3)	452 (2)	11 (1)
H(26)	726 (5)	132 (4)	394 (2)	4 (1)
H(27)	638 (4)	483 (3)	488 (2)	6 (1)

Table 1 (*cont.*)

	x	y	z	B (Å^2)
H(28)	680 (4)	468 (3)	439 (2)	17 (2)
H(29)	553 (5)	485 (4)	461 (2)	11 (1)
H(30)	540 (5)	274 (3)	503 (2)	8 (1)
H(31)	578 (5)	363 (4)	533 (2)	10 (1)
H(32)	466 (4)	368 (3)	490 (1)	10 (1)
H(33)	426 (5)	32 (4)	472 (2)	13 (2)
H(34)	521 (5)	15 (4)	436 (2)	17 (2)
H(35)	545 (5)	67 (3)	475 (2)	15 (2)
H(36)	172 (5)	399 (4)	408 (2)	12 (2)
H(37)	186 (5)	352 (4)	447 (2)	11 (2)
H(38)	252 (5)	417 (3)	451 (2)	13 (2)
H(39)	502 (5)	−45 (3)	323 (2)	11 (2)
H(40)	507 (5)	−33 (4)	273 (2)	16 (2)
H(41)	603 (5)	−42 (4)	304 (2)	13 (2)
H(42)	605 (5)	540 (3)	328 (2)	8 (1)
H(43)	710 (5)	515 (4)	328 (2)	5 (1)
H(44)	631 (5)	526 (4)	293 (2)	16 (2)
H(45)	559 (4)	206 (3)	335 (2)	11 (1)
H(46)	541 (4)	291 (3)	330 (1)	7 (1)

refinement of the other atoms. Final electron density maps showed no distinguishing features. The final R values were 0·038 and 0·033 for (I) and (II) respectively.* Scattering factors for neutral nonhydrogen atoms were from Cromer & Mann (1968) and the H scattering factors were those of Stewart, Davidson & Simpson (1965). An anomalous-dispersion correction was included for Co and Cl (Cromer & Liberman, 1970). Atomic coordinates are given in Tables 1 and 2.

Discussion. Interatomic distances and angles are listed in Tables 3, 4 and 5. Both structures are formed from discrete dimeric molecules, with intermolecular dis-

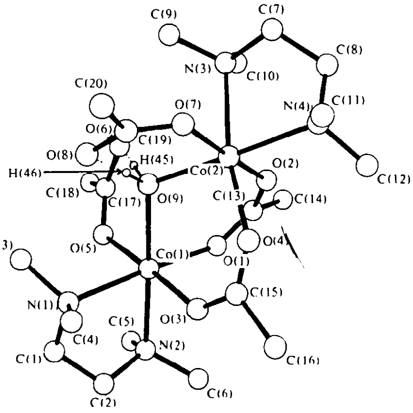

Fig. 1. A view of the molecular structure of compound (I).

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* Lists of structure factors and anisotropic thermal parameters for (I) and (II) have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36607 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Atomic coordinates* ($\times 10^4$ *for nonhydrogen atoms and* $\times 10^3$ *for hydrogen atoms*) *of* (II)

For nonhydrogen atoms $B_{eq} = \frac{1}{3}(B_{11} + B_{22} + B_{33} + 2B_{13}\cos\beta)$.

	x	y	z	B_{eq}/B (Å^2)
Co	6894 (1)	3156 (1)	3309 (1)	5·4 (1)
Cl(1)	3528 (1)	−259 (2)	1912 (1)	5·9 (2)
Cl(2)	8237 (2)	6584 (2)	5468 (1)	7·6 (1)
O(1)	6512 (2)	1652 (3)	1487 (1)	3·9 (2)
O(2)	5842 (3)	1670 (3)	2507 (1)	4·1 (2)
O(3)	7912 (2)	4704 (3)	4127 (1)	3·6 (2)
O(4)	8835 (3)	6442 (3)	3539 (2)	5·0 (2)
O(5)	7500	4582 (4)	2500	3·0 (2)
N(1)	6239 (3)	1907 (4)	4176 (2)	3·7 (2)
N(2)	5099 (3)	4581 (4)	3200 (2)	3·7 (2)
C(1)	5802 (3)	1223 (4)	1872 (2)	2·9 (2)
C(2)	4820 (3)	−28 (4)	1501 (2)	3·6 (2)
C(3)	8545 (3)	5931 (4)	4091 (2)	3·3 (2)
C(4)	9028 (4)	6954 (5)	4781 (2)	4·3 (2)
C(5)	5215 (5)	2915 (8)	4304 (3)	7·6 (4)
C(6)	4400 (5)	3623 (8)	3622 (3)	7·0 (4)
C(7)	7234 (5)	1799 (6)	4887 (2)	5·6 (3)
C(8)	5820 (6)	271 (6)	3952 (3)	6·7 (3)
C(9)	5333 (5)	6244 (7)	3463 (3)	7·1 (4)
C(10)	4273 (5)	4674 (9)	2432 (3)	5·6 (3)
H(1)	447 (4)	29 (5)	99 (2)	5 (1)
H(2)	520 (4)	−94 (6)	149 (3)	7 (1)
H(3)	987 (4)	675 (5)	499 (2)	5 (1)
H(4)	911 (5)	810 (6)	464 (3)	7 (1)
H(5)	469 (4)	224 (6)	455 (2)	6 (1)
H(6)	533 (10)	439 (12)	436 (5)	16 (3)
H(7)	373 (4)	425 (6)	369 (3)	6 (1)
H(8)	396 (9)	227 (11)	329 (5)	16 (3)
H(9)	752 (4)	271 (6)	506 (2)	6 (1)
H(10)	684 (5)	124 (6)	527 (3)	7 (1)
H(11)	808 (7)	121 (9)	480 (4)	12 (2)
H(12)	556 (6)	−39 (7)	430 (3)	10 (2)
H(13)	498 (6)	18 (7)	348 (4)	10 (2)
H(14)	661 (6)	−22 (7)	396 (3)	9 (1)
H(15)	440 (6)	679 (7)	341 (4)	10 (2)
H(16)	588 (6)	626 (8)	401 (4)	10 (1)
H(17)	571 (7)	692 (8)	317 (4)	12 (2)
H(18)	408 (6)	363 (8)	232 (3)	8 (2)
H(19)	349 (5)	547 (6)	241 (3)	8 (1)
H(20)	473 (6)	518 (7)	212 (4)	9 (1)
H(21)	805 (4)	518 (4)	276 (2)	5 (1)

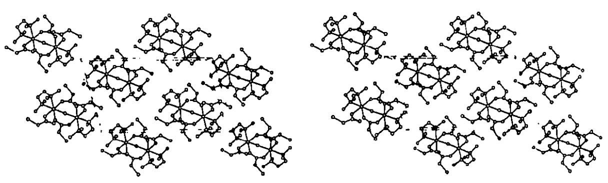

Fig. 2. A stereoview of the unit-cell packing of compound (II).

tances between all nonhydrogen atoms greater than 3·29 Å. The molecular symmetry of (I) is pseudo C_2 as in the analogous Ni$^{\text{II}}$ complexes (Ahlgrén, Turpeinen & Hämäläinen, 1978); compound (II) possesses C_2 symmetry (Figs. 1–3).

Table 3. *Interatomic distances* (Å) *in* (I)

Co(1)–N(1)	2·219 (4)	N(3)–C(7)	1·449 (9)
Co(1)–N(2)	2·221 (4)	N(3)–C(9)	1·448 (7)
Co(1)–O(1)	2·062 (4)	N(3)–C(10)	1·471 (8)
Co(1)–O(3)	2·046 (3)	C(7)–C(8)	1·467 (10)
Co(1)–O(5)	2·081 (3)	N(4)–C(8)	1·471 (9)
Co(1)–O(9)	2·147 (3)	N(4)–C(11)	1·450 (8)
Co(2)–N(3)	2·302 (4)	N(4)–C(12)	1·478 (8)
Co(2)–N(4)	2·218 (4)	C(13)–O(1)	1·233 (6)
Co(2)–O(2)	2·047 (4)	C(13)–O(2)	1·258 (6)
Co(2)–O(4)	2·070 (4)	C(13)–C(14)	1·498 (9)
Co(2)–O(7)	2·077 (3)	C(15)–O(3)	1·247 (6)
Co(2)–O(9)	2·117 (3)	C(15)–O(4)	1·230 (6)
N(1)–C(1)	1·472 (7)	C(15)–C(16)	1·512 (8)
N(1)–C(3)	1·473 (7)	C(17)–O(5)	1·249 (6)
N(1)–C(4)	1·481 (7)	C(17)–O(6)	1·234 (6)
C(1)–C(2)	1·458 (8)	C(17)–C(18)	1·521 (8)
N(2)–C(2)	1·455 (7)	C(19)–O(7)	1·246 (6)
N(2)–C(5)	1·465 (7)	C(19)–O(8)	1·246 (6)
N(2)–C(6)	1·469 (7)	C(19)–C(20)	1·519 (7)
Co(1)···Co(2)	3·597 (1)		

Table 4. *Interatomic angles* (°) *in* (I)

O(1)–Co(1)–O(3)	92·8 (1)	C(2)–N(2)–C(6)	110·5 (4)
O(1)–Co(1)–O(5)	87·8 (1)	C(5)–N(2)–C(6)	107·2 (5)
O(1)–Co(1)–O(9)	89·3 (1)	N(2)–C(2)–C(1)	114·3 (4)
O(1)–Co(1)–N(2)	91·4 (1)	C(7)–N(3)–C(9)	111·5 (5)
O(3)–Co(1)–O(9)	94·8 (1)	C(7)–N(3)–C(10)	109·5 (5)
O(3)–Co(1)–N(1)	90·2 (1)	C(9)–N(3)–C(10)	106·3 (5)
O(3)–Co(1)–N(2)	88·1 (1)	N(3)–C(7)–C(8)	114·0 (6)
O(5)–Co(1)–O(9)	87·8 (1)	C(8)–N(4)–C(11)	112·2 (6)
O(5)–Co(1)–N(1)	89·0 (1)	C(8)–N(4)–C(12)	108·4 (5)
O(5)–Co(1)–N(2)	89·3 (1)	C(11)–N(4)–C(12)	108·7 (5)
O(9)–Co(1)–N(1)	97·1 (1)	N(4)–C(8)–C(7)	113·6 (5)
N(1)–Co(1)–N(2)	82·0 (1)	Co(1)–O(1)–C(13)	134·5 (3)
O(2)–Co(2)–O(4)	93·5 (1)	Co(2)–O(2)–C(13)	135·0 (3)
O(2)–Co(2)–O(9)	89·8 (1)	O(1)–C(13)–O(2)	127·8 (5)
O(2)–Co(2)–N(3)	87·8 (2)	O(1)–C(13)–C(14)	117·5 (5)
O(2)–Co(2)–N(4)	89·8 (1)	O(2)–C(13)–C(14)	114·6 (5)
O(4)–Co(2)–O(7)	90·9 (1)	Co(1)–O(3)–C(15)	136·4 (3)
O(4)–Co(2)–O(9)	95·1 (1)	Co(2)–O(4)–C(15)	134·4 (3)
O(4)–Co(2)–N(4)	87·1 (2)	O(3)–C(15)–O(4)	127·0 (4)
O(7)–Co(2)–O(9)	88·9 (1)	O(3)–C(15)–C(16)	116·3 (4)
O(7)–Co(2)–N(3)	88·1 (1)	O(4)–C(15)–C(16)	116·7 (4)
O(7)–Co(2)–N(4)	91·4 (2)	Co(1)–O(5)–C(17)	126·7 (3)
O(9)–Co(2)–N(3)	96·9 (1)	O(5)–C(17)–O(6)	125·7 (4)
N(3)–Co(2)–N(4)	81·0 (2)	O(5)–C(17)–C(18)	116·0 (4)
Co(1)–O(9)–Co(2)	115·1 (1)	O(6)–C(17)–C(18)	118·3 (5)
C(1)–N(1)–C(3)	107·7 (4)	Co(2)–O(7)–C(19)	130·6 (3)
C(1)–N(1)–C(4)	111·6 (4)	O(7)–C(19)–O(8)	124·7 (4)
C(3)–N(1)–C(4)	106·9 (4)	O(7)–C(19)–C(20)	118·2 (4)
N(1)–C(1)–C(2)	112·3 (4)	O(8)–C(19)–C(20)	117·1 (5)
C(2)–N(2)–C(5)	110·9 (5)		

The complexes are octahedral, with a dimeric structure in which bridging occurs *via* the water O atom and the two carboxylate groups. The rest of the coordination sphere of each Co atom is completed by the N atoms of the diamine ligand and the O atom of the monodentate carboxylate group. The interatomic angles and the Co–Co, Co–O and Co–N distances are essentially similar in both compounds. Only the Co(2)–N(3) distance of 2·302 (4) Å is exceptional, being much longer than the distances for the other amino groups, which range from 2·205 (3) to 2·239 (3) Å.

Table 5. *Interatomic distances* (Å) *and angles* (°) *in* (II)

Symmetry code: (i) $-x + \frac{3}{2}, y, -z + \frac{1}{2}$.

Co–O(2)	2·039 (2)	N(2)–C(6)	1·472 (7)
Co–O(3)	2·074 (2)	N(2)–C(9)	1·468 (6)
Co–O(5)	2·165 (2)	N(2)–C(10)	1·475 (5)
Co–N(1)	2·205 (3)	C(1)–O(1)	1·244 (5)
Co–N(2)	2·239 (3)	C(1)–O(2)	1·235 (4)
Co–O(1^i)	2·083 (3)	C(1)–C(2)	1·515 (5)
N(1)–C(5)	1·462 (7)	C(2)–Cl(1)	1·782 (4)
N(1)–C(7)	1·469 (5)	C(3)–O(3)	1·242 (4)
N(1)–C(8)	1·461 (6)	C(3)–O(4)	1·238 (5)
C(5)–C(6)	1·464 (8)	C(3)–C(4)	1·517 (5)
Co···$Co^{(i)}$	3·621 (1)	C(4)–Cl(2)	1·759 (5)
O(2)–Co–O(5)	91·8 (1)	O(2)–C(1)–C(2)	119·0 (3)
O(2)–Co–N(1)	91·7 (1)	C(1)–C(2)–Cl(1)	114·3 (3)
O(2)–Co–N(2)	87·6 (1)	Co–O(3)–C(3)	131·4 (2)
O(2)–Co–O(1^i)	92·2 (1)	O(3)–C(3)–O(4)	126·7 (3)
O(3)–Co–O(5)	88·6 (1)	O(3)–C(3)–C(4)	118·9 (3)
O(3)–Co–N(1)	87·8 (1)	O(4)–C(3)–C(4)	114·4 (3)
O(3)–Co–N(2)	90·6 (1)	C(3) C(4) Cl(2)	113·7 (3)
O(3)–Co–O(1^i)	89·5 (1)	C(5)–N(1)–C(7)	106·7 (4)
O(5)–Co–N(2)	93·9 (1)	C(5)–N(1)–C(8)	113·1 (4)
O(5)–Co–O(1^i)	93·6 (1)	C(7)–N(1)–C(8)	107·3 (3)
N(1)–Co–N(2)	82·5 (1)	N(1)–C(5)–C(6)	113·1 (5)
N(1)–Co–O(1^i)	89·9 (1)	C(5)–C(6)–N(2)	114·3 (5)
Co–O(5)–$Co^{(i)}$	113·5 (2)	C(6)–N(2)–C(9)	113·5 (4)
Co–O(2)–C(1)	139·3 (2)	C(6)–N(2)–C(10)	106·8 (3)
O(1)–C(1)–O(2)	127·5 (3)	C(9)–N(2)–C(10)	106·3 (4)
O(1)–C(1)–C(2)	113·5 (3)		

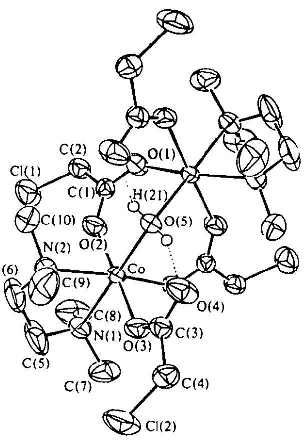

Fig. 3. A view of the molecular structure of compound (II).

The dimeric structure is additionally stabilized by the strong intramolecular hydrogen bonds formed between the noncoordinated O atoms of the monodentate carboxylate groups and the bridging H_2O molecule: O(6)···O(9) = 2·565 (4), O(8)···O(9) = 2·548 (4) and O(4)···O(5) = 2·597 (3) Å.

The bond lengths and angles associated with the diamine molecules are normal. The C—O bond lengths in bridging and nonbridging carboxylates are practically the same, while the O—C—O angles are slightly larger in bridging carboxylates. The O—C—C angles are not equivalent in chloroacetates because of electrostatic repulsion between the Cl and O atoms. Each O—C—C angle involving the atom nearer the Cl atom is greater than the other O—C—C angle in the same chloroacetate. Further, in the monodentate chloroacetate the Cl atom is located closer to the O atom that is bonded to the Co atom. This is also a common feature in dimeric Ni^{II} carboxylates (Ahlgrén, Turpeinen & Hämäläinen, 1978; Turpeinen, 1976, 1977).

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