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Aqua(cyclobutane-1,1-dicarboxylato)(*N*,*N*,*N*′,*N*′-tetramethylethylenediamine)copper(II) Monohydrate

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Abstract. $[Cu(C_6H_{16}N_2)(C_6H_6O_4)(H_2O)].H_2O$, $C_{12}H_{24}CuN_2O_5.H_2O$, monoclinic, $P2_1$, $a = 9.354$ (4), $b = 8.525$ (3), $c = 10.363$ (8) Å, $\beta = 102.02$ (5)°, $Z = 2$. The structure was refined to $R = 0.032$ for 1330 unique reflections. The coordination polyhedron is square-pyramidal, with the base formed by two N atoms of the diamine and two O atoms of the dicarboxylic acid. The apex of the pyramid is occupied by a water O atom.

Introduction. The title compound was prepared from copper(II) carbonate, cyclobutane-1,1-dicarboxylic acid and *N*,*N*,*N*′,*N*′-tetramethylethylenediamine (molar ratio 1:1:1) in a water–methanol mixture. The cell parameters and intensities were measured on a Syntex $P2_1$ diffractometer with monochromated Mo $K\alpha$ radiation ($\lambda = 0.7107$ Å). Systematic absences $0k0$ with k odd indicated space group $P2_1$ or $P2_1/m$. The noncentrosymmetric $P2_1$ was suggested by normalized structure factor statistics. Intensities for 1609 unique reflections in the range $3 < 2\theta < 55°$ were collected by the ω-scan technique.

The structure was solved by *MULTAN* (Main, Lessinger, Woolfson, Germain & Declercq, 1978) and refined by full-matrix least squares with experimental weights $w = 4F_o^2/\sigma_I^2$. Only the 1330 most significant reflections [$I > 3\sigma(I)$] were used in the refinement. The positions of the H atoms were deduced partly from a difference synthesis and partly from chemical considerations. The H atoms were included in the refinement with fixed isotropic temperature parameters ($U = 0.06$ Å^2) and fixed positional coordinates. The final R was 0.032.* Scattering factors for the non-hydrogen atoms and the H atoms were taken from Cromer &

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34577 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional atomic coordinates* ($\times 10^4$) *of the non-hydrogen atoms*

	x	y	z
Cu	3436 (1)	5000	3286 (1)
O(1)	4827 (13)	3448 (12)	3125 (10)
O(2)	6884 (12)	2640 (14)	2552 (12)
O(3)	4845 (12)	6731 (12)	3153 (10)
O(4)	6842 (14)	7509 (12)	2558 (11)
O(5)	4132 (4)	5004 (16)	5574 (3)
O(6)	1322 (5)	5084 (22)	7745 (8)
N(1)	1855 (13)	3400 (13)	3073 (12)
N(2)	1729 (13)	6672 (15)	3092 (12)
C(1)	5828 (5)	4939 (20)	1659 (4)
C(2)	4476 (5)	5081 (25)	446 (4)
C(3)	5528 (6)	4987 (29)	−480 (5)
C(4)	6843 (6)	4971 (30)	651 (5)
C(5)	5820 (13)	3563 (11)	2525 (11)
C(6)	5932 (14)	6495 (17)	2486 (12)
C(7)	395 (9)	4203 (13)	2733 (9)
C(8)	498 (8)	5728 (12)	3438 (9)
C(9)	1575 (23)	2613 (28)	1803 (18)
C(10)	2036 (22)	2196 (22)	4074 (19)
C(11)	1683 (23)	7600 (27)	1885 (17)
C(12)	2027 (21)	7767 (19)	4258 (17)

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Table 2. *Fractional atomic coordinates* ($\times 10^3$) *of the hydrogen atoms*

	x	y	z		x	y	z
H(C2)	388	410	32	H(C10)	123	139	401
H'(C2)	387	592	36	H'(C10)	296	163	409
H(C3)	540	608	−98	H"(C10)	210	274	496
H'(C3)	558	425	−110	H(C11)	64	798	174
H(C4)	751	398	64	H'(C11)	237	846	200
H'(C4)	759	588	71	H"(C11)	179	691	112
H(C7)	17	439	178	H(C12)	126	867	401
H'(C7)	−34	359	295	H'(C12)	190	721	507
H(C8)	−37	637	301	H"(C12)	304	819	438
H'(C8)	63	560	444	H(O5)	430	597	603
H(C9)	80	169	176	H'(O5)	428	398	602
H'(C9)	121	339	110	H(O6)	189	596	771
H"(C9)	252	216	166	H'(O6)	188	402	786

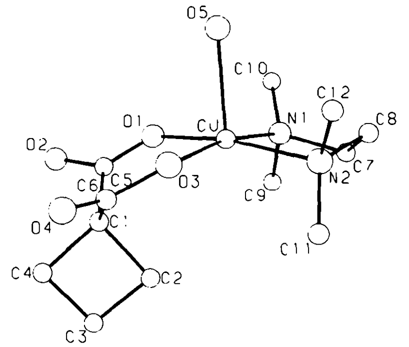

Fig. 1. Perspective view of the complex with the numbering scheme.

Table 3. *Bond lengths* (Å) *and angles* (°)

Cu—N(1)	1·991 (12)	C(5)—O(1)	1·224 (18)
Cu—N(2)	2·119 (12)	C(5)—O(2)	1·264 (16)
Cu—O(1)	1·887 (11)	C(6)—O(3)	1·358 (19)
Cu—O(3)	2·002 (11)	C(6)—O(4)	1·205 (18)
Cu—O(5)	2·325 (3)	N(1)—C(7)	1·502 (15)
C(1)—C(2)	1·592 (6)	N(1)—C(9)	1·452 (23)
C(2)—C(3)	1·513 (8)	N(1)—C(10)	1·443 (22)
C(3)—C(4)	1·512 (7)	N(2)—C(8)	1·508 (16)
C(1)—C(4)	1·552 (7)	N(2)—C(11)	1·473 (23)
C(1)—C(5)	1·477 (17)	N(2)—C(12)	1·507 (21)
C(1)—C(6)	1·571 (20)	C(7)—C(8)	1·485 (14)
N(1)—Cu—N(2)	85·5 (5)	C(9)—N(1)—C(10)	107·1 (14)
N(1)—Cu—O(1)	91·2 (5)	C(8)—N(2)—C(11)	126·9 (11)
N(1)—Cu—O(3)	169·1 (4)	C(8)—N(2)—C(12)	99·4 (12)
N(2)—Cu—O(1)	169·5 (4)	C(11)—N(2)—C(12)	108·4 (13)
N(2)—Cu—O(3)	89·5 (4)	N(1)—C(7)—C(8)	108·7 (7)
O(1)—Cu—O(3)	92·0 (4)	N(2)—C(8)—C(7)	108·9 (8)
N(1)—Cu—O(5)	99·1 (4)	O(1)—C(5)—O(2)	128·0 (11)
N(2)—Cu—O(5)	98·1 (4)	O(1)—C(5)—C(1)	118·2 (9)
O(1)—Cu—O(5)	92·3 (4)	O(2)—C(5)—C(1)	113·9 (11)
O(3)—Cu—O(5)	91·2 (4)	O(3)—C(6)—O(4)	117·1 (13)
Cu—N(1)—C(7)	109·4 (7)	O(3)—C(6)—C(1)	115·8 (10)
Cu—N(1)—C(9)	114·0 (12)	O(4)—C(6)—C(1)	127·1 (13)
Cu—N(1)—C(10)	115·7 (10)	C(2)—C(1)—C(4)	87·9 (3)
Cu—N(2)—C(8)	102·5 (7)	C(2)—C(1)—C(6)	108·4 (12)
Cu—N(2)—C(11)	109·3 (11)	C(4)—C(1)—C(6)	112·1 (13)
Cu—N(2)—C(12)	109·3 (9)	C(5)—C(1)—C(2)	115·9 (11)
Cu—O(1)—C(5)	126·8 (8)	C(5)—C(1)—C(4)	120·3 (13)
Cu—O(3)—C(6)	119·9 (8)	C(5)—C(1)—C(6)	110·3 (7)
C(7)—N(1)—C(10)	117·2 (13)	C(1)—C(2)—C(3)	89·0 (4)
C(7)—N(1)—C(9)	90·8 (11)	C(2)—C(3)—C(4)	92·3 (4)
		C(1)—C(4)—C(3)	90·5 (4)

Mann (1968) and Stewart, Davidson & Simpson (1965) respectively. The computations were made with XRAY 76 (Stewart, 1976). Table 1 lists the final coordinates for the non-hydrogen atoms; the positional parameters of the H atoms are given in Table 2. Interatomic distances and angles are listed in Table 3. A *PLUTO* (Motherwell, 1976) view of the complex with the numbering scheme appears in Fig. 1.

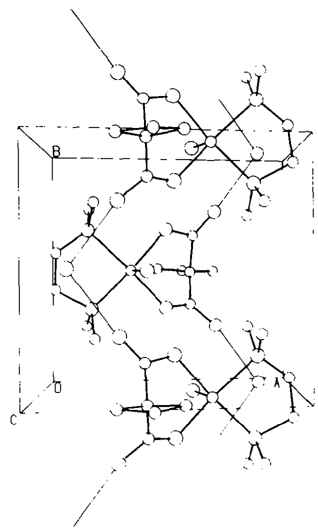

Fig. 2. The unit-cell contents.

Discussion. As shown in Fig. 1, the coordination geometry around the Cu atom is square-pyramidal, the base of the pyramid consisting of two N atoms and two O atoms of the bidentate diamine and dicarboxylic acid ligands. The base of the pyramid is planar, the maximum deviation of atoms from the least-squares plane being 0·002 (2) Å. The Cu atom is displaced 0·177 (2) Å out of the basal plane towards the water O(5). The planes Cu, N(1), N(2) and Cu, O(1), O(3) are inclined 14·3 (1)° to each other. The five-membered Cu–diamine ring is in an unsymmetrical *gauche* configuration: C(7) and C(8) deviate −0·17 (1) and 0·53 (1) Å from the Cu, N(1), N(2) plane. The six-membered Cu–dicarboxylic acid ring has a boat conformation.

The complex molecules of the crystal are joined by hydrogen bonds to form chains parallel to **b**. Every molecule forms two O···H—O hydrogen bonds between the uncoordinated O(2) and O(4) of the dicarboxylic acid and water O(6). O(2)···O(6)($1-x$, $-\frac{1}{2}+y$, $1-z$) is 2·807 (19) Å and O(4)···O(6)($1-x$, $\frac{1}{2}+y$, $1-z$) is 2·845 (19) Å. These hydrogen bonds are indicated by thin lines in Fig. 2, which shows the unit-cell contents.

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Structure of *cis*-Bromodicarbonyl(η-cyclopentadienyl)(triphenylphosphine)molybdenum Dichloromethane Solvate

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Abstract. $[MoBr(C_5H_5)(C_{18}H_{15}P)(CO)_2].\frac{1}{4}CH_2Cl_2$, $C_{25}H_{20}BrMoO_2P.\frac{1}{4}CH_2Cl_2$, triclinic, $P\bar{1}$, a = 18·927 (4), b = 12·335 (2), c = 11·003 (2) Å, α = 103·61 (1), β = 105·58 (1), γ = 99·70 (1)°, U = 2330 Å^3, D_m = 1·66, Z = 4, D_c = 1·654 Mg m^{-3}, $F(000)$ = 1154, μ(Mo $K\alpha$) = 2·52 mm^{-1}. The structure was refined to R = 0·057 for 5212 diffractometer data. The Br and *trans* CO sites are partially mutually disordered. The mean Mo—P and Mo—Br lengths are 2·532 (2) and 2·671 (3) Å.

Introduction. $[MoBr(\eta\text{-}C_5H_5)(CO)_2(PPh_3)]$ was prepared by heating $[MoBr(\eta\text{-}C_5H_5)(CO)_3]$ with triphenylphosphine under reflux. Red polyhedral crystals were obtained from solution in a petrol–dichloromethane mixture and one 0·20 × 0·20 × 0·25 mm was selected for the X-ray investigation. The intensities of 6944 reflections with $\theta \leq 25°$ were measured by the ω–2θ step-scan procedure with Zr-filtered Mo $K\alpha$ radiation on a Hilger & Watts four-circle diffractometer. The 5212 observations that satisfied the criterion $I > 3\sigma(I)$ were used in the subsequent analysis.

The coordinates of the two unique Mo atoms were deduced from a Patterson synthesis and the remaining atoms, apart from those of the CO group *trans* to the Br atom in each molecule, were located from successive electron-density distributions. After preliminary least-squares refinement, a difference synthesis indicated that there is disorder involving some interchange between the Br atom and the *trans* CO group in each molecule, the effect being similar to the halogen–carbonyl disorder that has been reported in several Ir complexes, *e.g.* $[Ir(CO)ClO_2(PPh_3)]$ (La Placa & Ibers, 1965), $[Ir(CO)_2Cl(PPh_3)]$ (Payne & Ibers, 1969), $[Ir(CN)(CO)Cl(NCS)(PPh_3)_2]$ (Ibers, Hamilton & Baddley, 1973) and $[IrBr\{C_2(CN)_4\}(CO)(PPh_3)_2]$ (Manojlović-Muir, Muir & Ibers, 1969).

The population parameters of the Br atoms were initially estimated from peak heights in an electron-density distribution and were subsequently adjusted by least squares. The population parameters of the C and O atoms of the disordered CO group were kept equal to the appropriate Br parameters. The C and O positions in the disordered group were held fixed at Mo—C and C—O = 1·96 and 1·14 Å.

A peak of *ca* 13 e Å^{-3}, about 1·5 Å from a centre of symmetry, and the centrosymmetrically related peak were identified as Cl atoms of a CH_2Cl_2 molecule. The C atom of this solvent molecule is necessarily disordered and appeared as two peaks, of height 1·4 e Å^{-3}, related by the centre of symmetry.

When least-squares adjustment of the various parameters reached R = 0·064, most of the H atoms were apparent in a difference map. The H atoms were included at ideal positions and further refinement converged at R = 0·057, R_w = 0·050, with weights w^{-1} = 17 + 0·01(|F_o| − 70)^2 − 30 sin θ. The peaks and

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