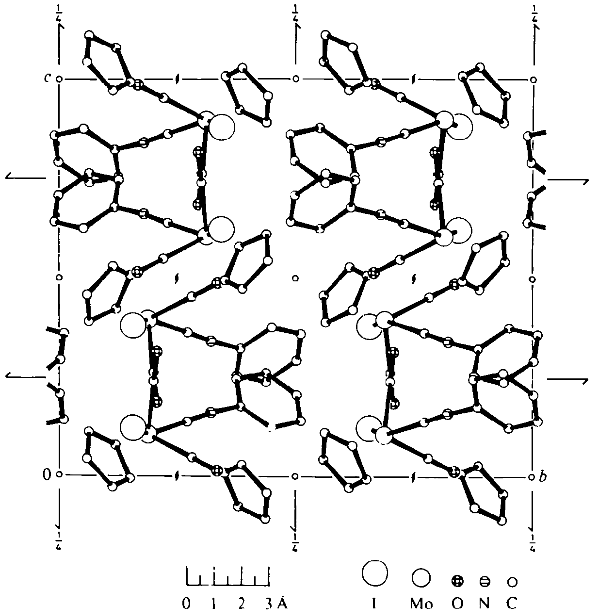

Fig. 2. The crystal structure viewed along **a**.

The Mo—CO lengths, 1·973 (10) and 1·983 (10) Å, do not differ significantly and the mean, 1·978 (7) Å, may be compared with the 1·96 Å in (η^5-C_5H_5)-$Mo(CO)_2(PPh_3)(COMe)$ (Churchill & Fennessey, 1968), 1·98 Å in (η^5-C_5H_5)$Mo(CO)_2(PPh_3)I$, 2·00 Å in (η^5-$C_5H_4.CH_3$)$Mo(CO)_2[P(OMe)_3]I$ and 2·02 Å in (η^5-C_5H_5)$Mo(CO)_2[P(OMe)_3]I$. The Mo—CNPh length of 2·025 (10) Å is only slightly greater than the mean Mo—CO distance, indicating that the bond has significant double-bond character (Sim, Sime, Woodhouse & Knox, 1974).

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The Structure of Bis(*N*-methylethylenediamine)copper(II) Adipate Dihydrate

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Abstract. $C_6H_{20}CuN_4^{2+}.C_6H_8O_4^{2-}.2H_2O$, $[Cu(C_3H_{10}N_2)_2](C_6H_8O_4).2H_2O$, $M_r = 391.95$, triclinic, $P\bar{1}$, $a = 7.241$ (2), $b = 8.271$ (3), $c = 8.304$ (3) Å, $\alpha = 89.92$ (3), $\beta = 114.87$ (2), $\gamma = 90.45$ (2)°, $V = 451.2$ Å^3, $Z = 1$, D_m (flotation) = 1·43, $D_x = 1.443$ Mg m^{-3}, μ(Mo $K\alpha$) = 1·29 mm^{-1}. The structure was solved by direct methods and refined by the block-diagonal least-squares technique, with anisotropic temperature parameters for nonhydrogen atoms and isotropic parameters for hydrogens, to an R value of 0·035. The analysis was based on 1931 reflections with $I > 2\sigma(I)$. The four N atoms from the diamine molecules form the coordination plane around the Cu^{II} atom; the two water O atoms lying in axial positions complete the elongated octahedral coordination sphere. The adipate group is hydrogen bonded to the N and water O atoms of the adjacent complex cations.

Introduction. The structures of some bis(*N*-methylethylenediamine)copper(II) carboxylates have been investigated previously in this laboratory (Hämäläinen, 1973; Hämäläinen & Pajunen, 1973, 1974). The carboxylate groups in these bis compounds are joined to the cations by hydrogen bonds, giving rise to net-like structures. We have now synthesized a bis chelate using a longer-chain dicarboxylic acid, adipic acid, as the anion and studied the structure by X-ray analysis.

The title compound was synthesized by allowing 0·045 mol of the finely powdered Cu^{II} salt of adipic acid [prepared by the method of Rajan (1962)] to react

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Table 1. *Fractional atomic coordinates* ($\times 10^4$; *for* H $\times 10^3$)

	x	*y*	*z*		*x*	*y*	*z*
Cu	0000	0000	0000	H(3)	−310 (5)	55 (4)	−381 (5)
O(1)	3703 (3)	−903 (3)	1076 (3)	H(4)	−260 (6)	−104 (5)	−390 (5)
O(2)	5215 (3)	2324 (3)	1604 (3)	H(5)	202 (4)	179 (3)	−211 (4)
O(3)	8478 (3)	2979 (3)	2572 (3)	H(6)	28 (5)	222 (4)	−370 (4)
N(1)	−131 (3)	259 (2)	−2499 (3)	H(7)	54 (4)	402 (4)	−153 (4)
N(2)	717 (3)	2364 (2)	287 (3)	H(8)	−123 (5)	316 (4)	−194 (4)
C(1)	−2149 (4)	−39 (4)	−3959 (4)	H(9)	205 (5)	250 (4)	88 (4)
C(2)	706 (4)	1884 (3)	−2579 (4)	H(10)	18 (5)	277 (4)	71 (4)
C(3)	72 (4)	3053 (3)	−1509 (4)	H(11)	432 (7)	−34 (5)	137 (6)
C(4)	6731 (4)	3197 (3)	2477 (3)	H(12)	399 (7)	−124 (6)	48 (6)
C(5)	6442 (4)	4633 (3)	3513 (4)	H(13)	586 (5)	548 (4)	265 (4)
C(6)	5009 (4)	4311 (3)	4391 (3)	H(14)	769 (6)	496 (5)	434 (5)
H(1)	61 (5)	−31 (4)	−251 (4)	H(15)	367 (5)	415 (4)	349 (4)
H(2)	−210 (5)	17 (4)	−506 (4)	H(16)	526 (5)	335 (4)	501 (4)

with 0·090 mol of *N*-methylethylenediamine in 100 ml methanol. The mixture was stirred mechanically. A crude product separated after concentration and the addition of ether. Recrystallization was performed from ethanol.

The Cu was analyzed electroanalytically, and C, N and O by microanalytical methods. Analysis, calculated: Cu, 16·21; C, 36·77; N, 14·29; O, 24·49%; found: Cu, 16·36; C, 36·75; N, 14·15 and O, 24·42%.

A crystal of approximate dimensions 0·45 × 0·15 × 0·15 mm was used for the X-ray measurements. The cell dimensions were calculated by least-squares refinement of the angular settings of 24 reflections measured on a four-circle Syntex $P2_1$ diffractometer with graphite-monochromatized Mo *Kα* radiation (λ = 0·71069 Å). The intensities of 2082 independent reflections with 2θ values between 3·0 and 55·0° were collected by the ω-scan technique (scan rate 2·0–10·0° min^{-1}) measuring one check reflection every 60 reflections. Lorentz and polarization corrections were applied to the data.

The positions of the nonhydrogen atoms were obtained using *MULTAN* 78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and the remaining calculations were carried out with XRAY 76 (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976). The structure was refined with isotropic temperature parameters to an *R* value of 0·084 assuming unit weight. Block-diagonal refinement with anisotropic parameters and the weighting scheme $w = 1/(60{\cdot}0 + |F_o| + 0{\cdot}009|F_o|^2)$ further reduced *R* to 0·053. When the positions of the H atoms obtained from a difference Fourier map were included in the structure determination with isotropic temperature parameters, the conventional *R* value fell to 0·035. The final difference synthesis was featureless. The scattering factors were those of Cromer & Mann (1968) and Stewart, Davidson & Simpson (1965) for neutral atoms.

Table 2. *Interatomic distances* (Å) *and angles* (°)

Symmetry code: (i) $-x + 1, -y + 1, -z + 1$.

Cu–N(1)	2·048 (2)	C(2)–C(3)	1·513 (5)
Cu–N(2)	2·008 (2)	O(2)–C(4)	1·253 (3)
Cu–O(1)	2·561 (2)	O(3)–C(4)	1·249 (4)
N(1)–C(1)	1·474 (3)	C(4)–C(5)	1·534 (4)
N(1)–C(2)	1·483 (4)	C(5)–C(6)	1·520 (5)
N(2)–C(3)	1·477 (4)	C(6)–C(6^{i})	1·529 (4)
N(1)–Cu–N(2)	85·4 (1)	Cu–N(2)–C(3)	107·5 (1)
O(1)–Cu–N(1)	88·9 (1)	N(2)–C(3)–C(2)	107·4 (2)
O(1)–Cu–N(2)	94·0 (1)	O(2)–C(4)–O(3)	124·6 (3)
Cu–N(1)–C(1)	115·1 (2)	O(2)–C(4)–C(5)	118·3 (3)
Cu–N(1)–C(2)	107·1 (2)	O(3)–C(4)–C(5)	117·1 (2)
C(1)–N(1)–C(2)	113·0 (2)	C(4)–C(5)–C(6)	114·8 (2)
N(1)–C(2)–C(3)	108·8 (3)	C(5)–C(6)–C(6^{i})	112·8 (2)

Positional parameters are given in Table 1,* with interatomic distances and angles in Table 2.

Discussion. The atom-labelling scheme is shown in Fig. 1. The four N atoms of the two diamine molecules form the coordination plane around the Cu^{II} atom [Cu–N(1) = 2·048 and Cu–N(2) = 2·008 Å]. The slightly longer Cu–N(1) bond joins the Cu to the N atom carrying the methyl group. The O atoms of the water molecules lying in axial positions complete the six-coordination. The interatomic distances and angles in the coordination sphere and in the diamine group are normal (Table 2). The diamine ligand has the usual *gauche* conformation, the deviations of the ring C atoms [C(2) and C(3)] from the coordination plane being 0·242 and −0·460 Å, respectively.

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* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34585 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

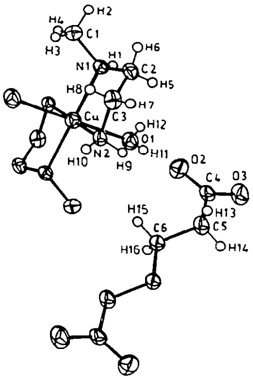

Fig. 1. The labelling scheme of bis(*N*-methylethylenediamine)-copper(II) adipate dihydrate.

Table 3. *Hydrogen bonds*

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

X–H···Y	X···Y	X–H	H···Y	∠X–H···Y
O(1)–H(11)···O(2)	2·840 (3) Å	0·62 Å	2·28 Å	153°
N(2)–H(9)···O(2)	2·969 (3)	0·89	2·12	161
N(2)–H(10)···O(3^{i})	3·015 (4)	0·71	2·36	155
O(1)–H(12)···O(2^{ii})	2·905 (4)	0·67	2·23	177
N(1)–H(1)···O(3^{ii})	2·954 (3)	0·72	2·32	149

The adipate ion is situated between the complex cations, attached to them by hydrogen bonds through the carboxylate O atoms (Table 3). Similar structures have also been found in bis(*N*-methylethylenediamine)copper(II) complexes formed with oxalate, malonate or tartrate ions (Hämäläinen, 1973; Hämäläinen & Pajunen, 1973; Hämäläinen & Pajunen, 1974). The C–O bonds in the carboxylate group are equal within error limits and typical for the free, ionic form. The other bond lengths and angles in the adipate ion are comparable with those reported for adipato-(*N*,*N*-diethylethylenediamine)copper(II) (Pajunen & Näsäkkälä, 1977). A stereoview of the unit-cell packing is given in Fig. 2.

The equations of the planes calculated through the carboxylate group and through the two N atoms and the Cu ion are given in Table 4. The carboxylate group is planar, the largest deviation being 0·001 Å.

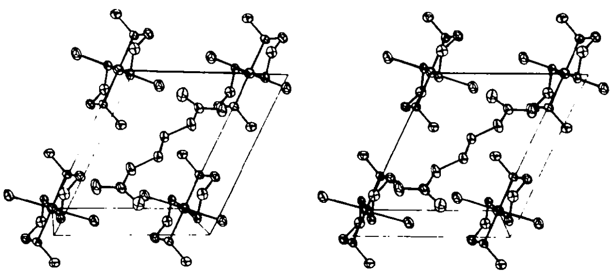

Fig. 2. Stereoview of the unit-cell packing. The *a* axis is horizontal and the *c* axis vertical.

Table 4. *Deviations* (Å) *of selected atoms from the planes calculated through the coordination plane and the carboxylate group*

x,y,z refer to direct-space coordinates.

(1) Plane defined by Cu, N(1), N(2)

$$6\cdot5856x - 1\cdot9299y - 0\cdot5449z = 0$$

C(2)	0·242 (10)	C(3)	−0·460 (10)

(2) Plane defined by O(2), O(3), C(4), C(5)

$$-0\cdot9462x - 4\cdot8538y + 6\cdot4801z = -0\cdot5820$$

O(2)	0·000 (9)	C(4)	−0·001 (9)
O(3)	0·000 (9)	C(5)	0·000 (9)

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