métallique: 2,13 Å dans Na_2S_2 (Föppl, Busmann & Frorath, 1962) et celle de la molécule S_2 à l'état vapeur dans l'état fondamental $^3\Sigma_g^-$ (1,887 Å; Meyer, 1976). Comme dans le compsé (I), $[Nb(CH_3)(C_5H_5)_2(S_2)]$ (Mercier *et al.*, 1980), les deux liaisons Nb—S ne sont pas équivalentes, la liaison centrale étant plus courte que la liaison latérale [2,457 (4) et 2,529 (4) Å; 2,432 (2) et 2,515 (2) Å dans la molécule (I)].

Cette distribution dissymétrique des électrons entre Nb—S(1) et Nb—S(2) ne résulte donc pas de la nature du troisième ligand. La troisième liaison Nb—S(3) est nettement plus longue que les deux liaisons Nb—S du cycle $Nb—S_2$ puisqu'elle vaut 2,602 (4) Å (Tableau 2). Cet atome S(3), à la différence du groupe S_2 chélaté sur Nb joue le rôle de pont entre Nb et P. On peut donc penser que la liaison Nb—S(3) est typique d'une liaison σ-Nb—S et que les liaisons $Nb{=}S_2$ sont sensiblement renforcées, en particulier la liaison centrale.

Ce groupement est lié à Nb par l'atome S(3); la particularité de groupe unidenté est l'existence d'une liaison P=S terminale plus courte que la liaison P—S du pont [1,935 (7) et 2,036 (6) Å]. Les paramètres géométriques de ce groupe sont rassemblés dans le Tableau 3. L'atome de phosphore possède un environnement grossièrement tétraédrique avec des angles allant de 100,4 (0,8) à 119,4 (0,3)°, en passant de O—P—O, O—P—S, O—P=S à S—P=S. Les caractéristiques des groupes éthoxy OC_2H_5 sont comparables avec des longueurs voisines de 1,45 Å pour la liaison O—C et 1,49 Å pour la liaison C—C. Les caractéristiques de ce groupement sont voisines de celui présent dans la molécule $[Sn(C_6H_5)_3\{S—P(=S)(OC_2H_5)_2\}]$ (Molloy, Hossain, van der Helm, Zuckerman & Haiduc, 1979). La cohésion de la structure est assurée par des liaisons de van der Waals entre atomes de carbone des cycles (C···C ~3,4 Å) et par des interactions S···S (3,6 Å) entre deux ligands S_2.

Tableau 3. *Distances interatomiques* (Å) *et angles* (°) *dans le groupe diéthyldithiophosphate*

Les écarts types sont donnés entre parenthèses.

P—S(3)	2,036 (6)	O(1)—C(1)	1,46 (2)
P—S(4)	1,935 (7)	O(2)—C(3)	1,45 (3)
P—O(1)	1,58 (1)	C(1)—C(2)	1,50 (3)
P—O(2)	1,58 (1)	C(3)—C(4)	1,48 (4)
S(3)—P—S(4)	119,4 (3)	O(1)—P—O(2)	100,4 (8)
S(3)—P—O(1)	104,1 (7)	P—O(1)—C(1)	120 (1)
S(3)—P—O(2)	103,2 (7)	P—O(2)—C(3)	116 (1)
S(4)—P—O(1)	113,3 (7)	O(1)—C(1)—C(2)	109 (1)
S(4)—P—O(2)	114,1 (7)	O(2)—C(3)—C(4)	110 (1)

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Bis(2-amino-2-methyl-1-propanolato)copper(II) Monohydrate

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Abstract. $[Cu(C_4H_{10}NO)_2].H_2O$, $C_8H_{20}CuN_2O_2.H_2O$, monoclinic, $C2/c$, $a = 19{\cdot}531\ (7)$, $b = 5{\cdot}966\ (1)$, $c = 10{\cdot}328\ (4)$ Å, $\beta = 92{\cdot}55\ (3)°$, $V = 1202{\cdot}2\ (6)$ Å^3, $Z = 4$, $D_m = 1{\cdot}42$ (flotation CH_2Cl_2/CH_3Cl), $D_c = 1{\cdot}42$ Mg m^{-3}, μ(Mo $K\alpha$) = $1{\cdot}87$ mm^{-1}. Final $R = 0{\cdot}039$ for 720 unique reflections. Cu is at a centre of symmetry and mononuclear, dichelated Cu complexes are joined into chains along c by hydrogen bonds from NH_2 groups to alkoxide O atoms. Additional hydrogen bonding, which links the complexes to form layers, occurs between water O and ligand O and N atoms. All hydrogen-bond distances are normal.

Introduction. Cooperative hydrogen bonding, in which separate complexes are joined into larger units by two or more hydrogen bonds, has recently been discussed by Bertrand, Fujita & Vanderveer (1980) for

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transition-metal–aminoalcohol complexes. Aminoalcohol ligands coordinated to different metal ions may be hydrogen bonded to one another through N and O atoms of the ligands, the separate complexes thus being connected by hydrogen-bond bridges.

Violet crystals of $[Cu(C_4H_{10}NO)_2].H_2O$, mainly thin plates, were obtained together with $C_4H_{12}NO^+.Br^-$ (Muhonen, 1980) in the reaction of $CuBr_2$ with 2-amino-2-methyl-1-propanol. The space group was determined from the systematic absences in Weissenberg photographs, the centrosymmetric choice being confirmed by the successful structure determination. All other X-ray measurements were made with a Syntex $P2_1$ diffractometer using graphite-monochromated Mo $K\alpha$ radiation (λ = 0·7107 Å) and a crystal 0·28 × 0·14 × 0·04 mm. With the θ–2θ scan technique and a variable scan rate, from 2·2 to 29·3° min^{-1}, 1382 unique reflections with $4 < 2\theta < 53°$ were collected. Of these, 1255 were used in the early stage of structure determination and 720 with $I > 3{\cdot}0\sigma(I)$ [$\sigma(I)$ from counting statistics] in the final refinement. Two test reflections checked after every 98 intensity measurements showed no significant changes during the data collection. Lorentz, polarization and empirical absorption corrections were applied.

The structure was solved by direct methods and refined by full-matrix least-squares techniques using programs of the XRAY 76 system (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976). When R was 0·063, H atom positions, located from the difference map, were included in the refinement. Final refinement with anisotropic thermal parameters for non-hydrogen atoms and isotropic thermal parameters for H atoms led to R = 0·039 and R_w = 0·041. The weighting scheme $w = 1/[\sigma^2(F_o) + 0{\cdot}0005F_o^2]$ was employed. The atomic scattering factors were those contained in the XRAY 76 system and the anomalous-dispersion correction for the scattering factor for Cu was taken from *International Tables for X-ray Crystallography* (1974). Final atomic parameters are given in Table 1,* and bond lengths and angles in Table 2.

Table 1. *Fractional coordinates* (×10^4, *for* H ×10^3) *and* U_{eq} *or* U_{iso} *values* (×10^2)

	x	y	z	U_{eq}* or U_{iso} (Å^2)
Cu	0	5000	5000	2·3 (5)
O(1)	637 (2)	3471 (6)	3951 (3)	2·9 (6)
O(W)	0	249 (12)	2500	4·5 (6)
N	615 (2)	4022 (7)	6486 (3)	2·3 (6)
C(1)	1155 (3)	2395 (11)	4690 (5)	3·4 (6)
C(2)	1306 (3)	3615 (10)	5977 (5)	2·6 (6)
C(3)	1636 (3)	5871 (12)	5764 (6)	4·7 (7)
C(4)	1759 (3)	2200 (11)	6889 (5)	4·5 (6)
H(1)	154 (3)	228 (10)	421 (5)	4 (2)
H(2)	108 (3)	76 (9)	495 (5)	5 (2)
H(3)	142 (2)	679 (8)	508 (5)	3 (1)
H(4)	213 (2)	567 (8)	546 (4)	4 (2)
H(5)	162 (2)	671 (9)	649 (4)	4 (1)
H(6)	218 (3)	192 (11)	651 (5)	7 (2)
H(7)	185 (2)	287 (9)	756 (5)	3 (1)
H(8)	154 (3)	71 (10)	704 (5)	7 (2)
H(9)	63 (2)	494 (11)	717 (4)	4 (1)
H(10)	50 (2)	289 (8)	670 (4)	2 (1)
H(11)	22 (3)	105 (10)	299 (5)	8 (2)

* $U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

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

Cu–N	1·994 (4)	C(3)–H(3)	0·98 (5)
Cu–O(1)	1·916 (3)	C(3)–H(4)	1·04 (5)
C(1)–O(1)	1·397 (6)	C(3)–H(5)	0·90 (5)
C(1)–C(2)	1·532 (7)	C(4)–H(6)	0·94 (6)
C(2)–N	1·489 (6)	C(4)–H(7)	0·82 (5)
C(2)–C(3)	1·513 (9)	C(4)–H(8)	1·00 (6)
C(2)–C(4)	1·520 (8)	N–H(9)	0·89 (5)
C(1)–H(1)	0·92 (5)	N–H(10)	0·75 (5)
C(1)–H(2)	1·03 (5)	O(W)–H(11)	0·81 (6)
N–Cu–O(1)	84·9 (1)	N–C(2)–C(4)	112·6 (4)
Cu–N–C(2)	107·5 (3)	C(1)–C(2)–C(3)	111·3 (4)
Cu–O(1)–C(1)	112·5 (3)	C(1)–C(2)–C(4)	111·0 (5)
O(1)–C(1)–C(2)	111·2 (5)	C(3)–C(2)–C(4)	110·1 (4)
N–C(2)–C(1)	104·0 (4)	H(11)–O(W)–H(11^i)	108 (6)
N–C(2)–C(3)	107·7 (5)		

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

Discussion. The Cu atom lies at a centre of symmetry and is planar coordinated by aminoalcohol O and N atoms (Fig. 1). All bond lengths and angles and the folding of the ligand are the same as the corresponding values found for the mononuclear complex bis-(2-amino-2-methyl-1-propanol)copper(II) dibenzoate (Muhonen & Hämäläinen, 1978). The dihedral angle between the planes O–C(1)–C(2) and N–C(2)–C(1) is 47·1 (6)°. C(1) and C(2) lie on opposite sides of the plane through Cu, N and O(1) and at distances of

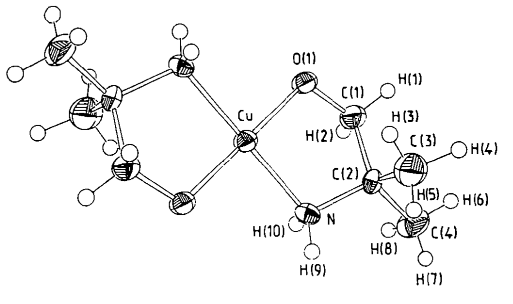

Fig. 1. View of the $[Cu(C_4H_{10}NO)_2]$ unit.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35870 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

0·075 (6) and 0·566 (5) Å, respectively, from the plane.

The only direct hydrogen bonding between complexes occurs in the *c* direction, from the NH_2 group of one ligand to the alkoxide O of a ligand from a neighbouring molecule. The N···O distance is 2·951 (5) Å, the magnitude normally found for N···O hydrogen bonds. The Cu···Cu distance between complexes is 5·164 Å (*c*/2).

The compound contains one molecule of water of crystallization per formula unit, with O(*W*) on a twofold axis. This causes additional hydrogen bonding which connects complexes that have Cu atoms on the same (200) plane. The O(*W*)···N and O(*W*)···O(1) distances of 3·024 (7) and 2·706 (6) Å are normal values for O···N and O···O hydrogen-bond contacts. Fig. 2 depicts the crystal structure and Table 3 gives the hydrogen-bond parameters.

The hydrogen-bonding network differs from that found for the dichelate complex cation $[Cu(C_4H_{10}NO)(C_4H_{11}NO)(H_2O)]_2^{2+}$ in which the same amino-alcohol is the ligand (Bertrand, Fujita & Vanderveer, 1980). This cation is a hydrogen-bonded dimer with a short O···O distance of 2·516 (3) Å between pairs of ligands coordinated to different Cu atoms. All other hydrogen-bond donor–acceptor distances in the compound are normal. Since all hydrogen-bond contacts in $[Cu(C_4H_{10}NO)_2].H_2O$ are normal, the complex is to be regarded as monomeric.

In $[Cu(C_4H_{11}NO)_2](C_7H_5O_2)_2$ (Muhonen & Hämäläinen, 1978) the complex ions are connected by hydrogen bonds involving the benzoate ions. The Cu···Cu distance is 5·654 (2) Å and there is a short O···O distance of 2·431 (7) Å between the ligand and the benzoate ion.

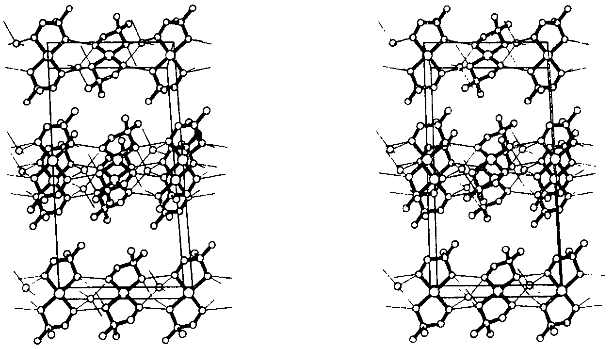

Fig. 2. Stereoview of overall structure. Fine lines indicate hydrogen bonds. The *a* axis is up the page, *b* towards the viewer, and *c* across from left to right.

Table 3. *Hydrogen-bond distances* (Å) *and angles* (°)

D–H···*A*	∠*D*–H···*A*	*D*···*A*	H···*A*	*D*–H
N–H(9)···O(1^{ii})	169 (5)	2·951 (5)	2·07 (5)	0·89 (5)
N–H(10)···O(W^{iii})	170 (4)	3·024 (7)	2·29 (5)	0·75 (5)
O(*W*)–H(11)···O(1)	167 (6)	2·706 (6)	1·91 (6)	0·81 (6)

Symmetry codes: (ii) *x*, 1 − *y*, ½ + *z*; (iii) −*x*, −*y*, 1 − *z*.

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Tetrakis(dimethyl sulphoxide)palladium(II) Bis(tetrafluoroborate) Dimethyl Sulphoxide Solvate

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Abstract. $C_{10}H_{30}B_2F_8O_5PdS_5$, $[Pd\{(CH_3)_2SO\}_4][BF_4]_2.(CH_3)_2SO$, monoclinic, $P2_1/c$, a = 10·650 (3), b = 14·163 (5), c = 17·778 (7) Å, β = 95·85 (3)°, U = 2667·6 Å^3, Z = 4, D_c = 1·67 Mg m^{-3}, μ(Cu $K\alpha$) = 9·95 mm^{-1}; final R = 0·086 for 2475 unique diffractometer data. The Pd^{II} atom displays the expected four-coordinate square-planar geometry. Two of the coordinated dimethyl sulphoxide (Me_2SO) ligands bond to the metal *via* S and the other two *via* O atoms. The dication exhibits a *cis* geometry. The anions and the Me_2SO solvate molecule are not coordinated.

Introduction. Dimethyl sulphoxide is an interesting ligand because of the coordination isomerism which it

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