C(60)	0.3360 (5)	0.5545 (5)	0.7555 (5)	0.072 (6)
C(61)	0.3815 (9)	0.5773 (7)	0.8511 (8)	0.105 (9)
C(62)	0.2708 (6)	0.6097 (6)	0.7352 (6)	0.079 (7)

Table 2. *Selected geometric parameters (Å, °)*

Fe(1)—C(1)	2.060 (7)	Fe(3)—C(41)	2.088 (6)
Fe(1)—C(2)	2.064 (7)	Fe(3)—C(43)	2.112 (6)
Fe(1)—C(4)	2.077 (7)	Fe(3)—C(45)	2.088 (6)
Fe(1)—C(6)	2.076 (7)	Fe(3)—C(47)	2.089 (6)
Fe(1)—C(8)	2.079 (7)	N(1)—C(50)	1.13 (1)
Fe(1)—C(10)	2.087 (6)	N(2)—C(51)	1.10 (1)
Fe(1)—C(12)	2.092 (6)	N(3)—C(53)	1.09 (1)
Fe(1)—C(14)	2.091 (6)	N(4)—C(54)	1.12 (1)
Fe(1)—C(16)	2.089 (5)	N(5)—C(56)	1.15 (1)
Fe(1)—C(18)	2.091 (5)	N(6)—C(57)	1.13 (1)
Fe(2)—C(20)	2.039 (5)	N(7)—C(59)	1.133 (9)
Fe(2)—C(21)	2.035 (6)	N(8)—C(61)	1.15 (1)
Fe(2)—C(22)	2.031 (6)	N(9)—C(62)	1.145 (9)
Fe(2)—C(23)	2.028 (6)	C(49)—C(50)	1.39 (2)
Fe(2)—C(24)	2.024 (6)	C(49)—C(51)	1.59 (2)
Fe(2)—C(25)	2.024 (6)	C(49)—C(52)	1.13 (2)
Fe(2)—C(26)	2.023 (6)	C(52)—C(53)	1.61 (2)
Fe(2)—C(27)	2.024 (6)	C(52)—C(54)	1.40 (1)
Fe(2)—C(28)	2.029 (6)	C(55)—C(56)	1.40 (1)
Fe(2)—C(29)	2.035 (5)	C(55)—C(57)	1.43 (1)
Fe(3)—C(31)	2.082 (5)	C(55)—C(58)	1.38 (1)
Fe(3)—C(32)	2.102 (5)	C(58)—C(59)	1.48 (1)
Fe(3)—C(34)	2.100 (5)	C(58)—C(60)	1.37 (1)
Fe(3)—C(36)	2.097 (5)	C(60)—C(61)	1.46 (1)
Fe(3)—C(38)	2.085 (5)	C(60)—C(62)	1.37 (1)
Fe(3)—C(40)	2.065 (6)		
C(50)—C(49)—C(51)	117.0 (13)	C(57)—C(55)—C(58)	120.6 (7)
C(50)—C(49)—C(52)	133.0 (20)	N(5)—C(56)—C(55)	175.9 (11)
C(51)—C(49)—C(52)	110.0 (14)	N(6)—C(57)—C(55)	177.3 (10)
N(1)—C(50)—C(49)	175.8 (15)	C(55)—C(58)—C(59)	115.9 (7)
N(2)—C(51)—C(49)	174.5 (16)	C(55)—C(58)—C(60)	129.6 (7)
C(49)—C(52)—C(53)	109.6 (14)	C(59)—C(58)—C(60)	114.4 (7)
C(49)—C(52)—C(54)	133.5 (18)	N(7)—C(59)—C(58)	177.3 (8)
C(53)—C(52)—C(54)	116.5 (11)	C(58)—C(60)—C(61)	120.7 (7)
N(3)—C(53)—C(52)	168.6 (10)	C(58)—C(60)—C(62)	125.5 (8)
N(4)—C(54)—C(52)	172.3 (12)	C(61)—C(60)—C(62)	113.8 (7)
C(56)—C(55)—C(57)	117.4 (8)	N(8)—C(61)—C(60)	176.8 (11)
C(56)—C(55)—C(58)	122.0 (8)	N(9)—C(62)—C(60)	177.6 (11)

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1987). Cell refinement: *CAD*-4 *Software*. Data reduction: *CRYSTALS* (Watkin, Carruthers & Betteridge, 1975). Program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994). Program(s) used to refine structure: *CRYSTALS*. Molecular graphics: *CAMERON* (Pearce & Watkin, 1993). Software used to prepare material for publication: *CRYSTALS*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1195). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Monoclinic Form of $[Cu(C_{10}H_{24}N_4)]CuCl_4$

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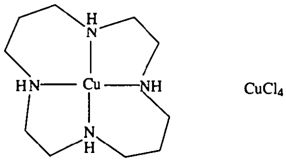
Abstract

The monoclinic form of 1,4,8,11-tetraazacyclotetradecanecopper(II) tetrachlorocuprate, [Cu(14-ane)]CuCl$_4$, has a chain structure consisting of Cu(14-ane)$^{2+}$ cations linked by CuCl$_4^{2-}$ anions. The Cu^{II} ion has square-planar coordination in the cation [average Cu—N distance = 2.020 (7) Å] while the anion has flattened tetrahedral coordination geometry [average Cu—Cl distance = 2.246 (13) Å with average *trans* Cl—Cu—Cl angles = 130 (1)°]. The cations and anions are linked by semi-

coordinate Cu···Cl bonds with a distance of 2.855 (2) Å to form chains which run parallel to the *c* axis and pack in a hexagonal array.

Comment

The title compound, (I), was prepared as part of our studies of the magnetic interactions in bimetallic chains (Vasilevesky, Rose, Stenkamp & Willett, 1991; Willett & Wang, 1993; Willett, Wang, Molnar, Brewer, Landee, Turnbull & Zhang, 1993). A previous report on the crystal structure of $[Cu(14\text{-ane})]CuCl_4$ described an orthorhombic form (Studer, Reisen & Kaden, 1989). The two forms are very closely related. The orthorhombic space group is *Pmcn* (non-standard setting for No. 62) with $a = 12.804$, $b = 8.370$, $c = 16.691$ Å and $V = 1799.5$ Å^3, with the axes relabeled to agree with the monoclinic form reported here [where we observe $(a^*)^{-1} = 12.934$, $b = 8.352$, $c = 16.746$ Å with $V = 1809.0$ Å^3]. The conformations of the chains in the two forms are identical, although local symmetries are different (line groups *p*121 for the monoclinic form *versus pmc*1 for the orthorhombic form). The distances between the chains are 8.370 (× 7) and 10.582 Å (×4) in the monoclinic form. In both forms, the chains pack in hexagonal arrays. For efficient packing, the chains separated by ~10.5 Å are out-of-registry. In the monoclinic form, the out-of-registry displacement of adjacent chains is always in the same direction, thus defining a monoclinic structure. For the orthorhombic form, displacements of adjacent chains are in alternate directions and thus define an orthorhombic structure.



(I)

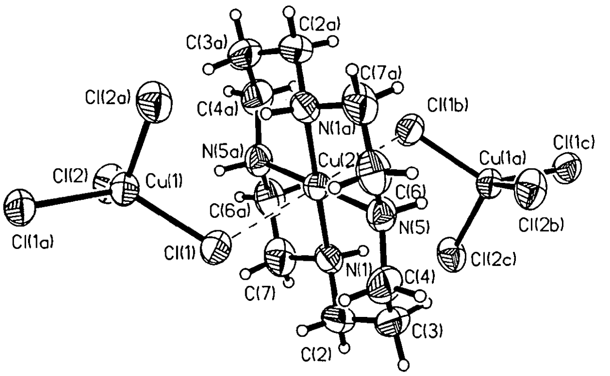
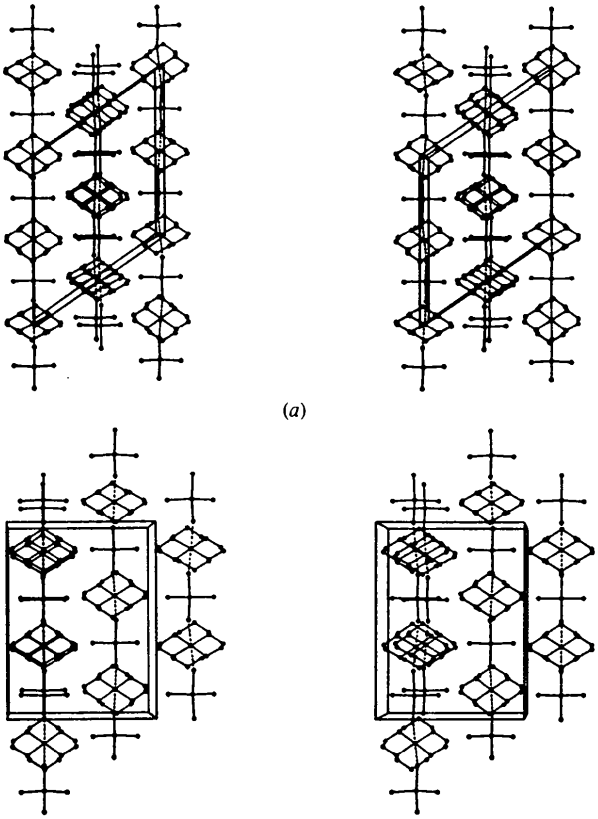


Fig. 1. Illustration of the chain structure. Displacement ellipsoids are shown at the 50% probability level.

In the monoclinic form, the $CuCl_4^{2-}$ and $Cu(14\text{-ane})^{2+}$ ions are situated on twofold axes, perpendicular to the chains and centers of inversion, respectively. In contrast, in the orthorhombic form they are located on mirror planes.



(*a*)

(*b*)

Fig. 2. Stereoscopic packing diagrams for (*a*) the monoclinic form and (*b*) the orthorhombic form.

Experimental

Crystals were grown by slow diffusion of an aqueous solution of $CuCl_2.2H_2O$ and the 14-ane ligand into a solution of $CuCl_2.2H_2O$ in acetone.

Crystal data

$[Cu(C_{10}H_{24}N_4)]CuCl_4$
$M_r = 469.2$
Monoclinic
$C2/c$
$a = 15.658\ (5)$ Å
$b = 8.352\ (3)$ Å
$c = 16.746\ (5)$ Å
$\beta = 124.31\ (2)^\circ$
$V = 1809.0\ (9)$ Å^3
$Z = 4$
$D_x = 1.723$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 14\text{–}15^\circ$
$\mu = 2.937$ mm^{-1}
$T = 22$ K
Needle
0.4 × 0.2 × 0.2 mm
Red

Data collection

Syntex $P2_1$ upgraded to Nicolet $R3$ diffractometer
ω scans
Absorption correction: ψ scan (*SHELXTL-Plus*; Sheldrick, 1991)
$T_{min} = 0.431$, $T_{max} = 0.675$
1335 measured reflections
1178 independent reflections
1045 observed reflections [$F > 3\sigma(F)$]
$R_{int} = 0.031$
$\theta_{max} = 25°$
$h = 0 \rightarrow 16$
$k = 0 \rightarrow 8$
$l = -17 \rightarrow 14$
2 standard reflections monitored every 96 reflections intensity decay: none

Refinement

Refinement on F
$R = 0.0376$
$wR = 0.0565$
$S = 0.75$
1045 reflections
93 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F) + 0.0047F^2]$
$(\Delta/\sigma)_{max} = 0.003$
$\Delta\rho_{max} = 0.64$ e Å^{-3}
$\Delta\rho_{min} = -0.54$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
Cu(1)	1/2	0.1219 (1)	3/4	0.044 (1)
Cu(2)	1/2	0	1/2	0.040 (1)
Cl(1)	0.5013 (1)	0.2272 (1)	0.6280 (1)	0.060 (1)
Cl(2)	0.6562 (1)	0.0008 (2)	0.8322 (1)	0.059 (1)
N(1)	0.6183 (3)	0.1139 (4)	0.5083 (3)	0.049 (2)
C(2)	0.6052 (4)	0.2861 (6)	0.4865 (4)	0.062 (3)
C(3)	0.5107 (5)	0.3236 (6)	0.3883 (4)	0.065 (3)
C(4)	0.4099 (4)	0.2968 (6)	0.3793 (4)	0.064 (3)
N(5)	0.3895 (3)	0.1250 (4)	0.3831 (3)	0.049 (2)
C(6)	0.2912 (4)	0.0965 (7)	0.3736 (4)	0.063 (3)
C(7)	0.7115 (4)	0.0750 (8)	0.6046 (4)	0.066 (3)

Table 2. *Selected geometric parameters* (Å, °)

Cu(1)—Cl(1)	2.234 (2)	Cu(1)—Cl(2)	2.259 (1)
Cu(2)—N(1)	2.016 (5)	Cu(2)—N(5)	2.025 (3)
N(1)—C(2)	1.469 (6)	N(1)—C(7)	1.477 (5)
C(2)—C(3)	1.500 (7)	C(3)—C(4)	1.515 (11)
C(4)—N(5)	1.479 (6)	N(5)—C(6)	1.475 (8)
C(6)—C(7^i)	1.484 (9)	Cu(2)···Cl(1)	2.855 (2)
Cl(1)—Cu(1)—Cl(2)	99.7 (1)	Cl(1)—Cu(1)—Cl(1^{ii})	133.6 (1)
Cl(2)—Cu(1)—Cl(2^{ii})	126.8 (1)	Cl(2)—Cu(1)—Cl(1^{ii})	100.6 (1)
N(1)—Cu(2)—N(5)	94.3 (2)	N(5)—Cu(2)—N(1^i)	85.7 (2)
Cu(2)—N(1)—C(2)	116.8 (4)	Cu(2)—N(1)—C(7)	106.0 (4)
C(2)—N(1)—C(7)	113.6 (4)	N(1)—C(2)—C(3)	113.0 (4)
C(2)—C(3)—C(4)	114.0 (6)	C(3)—C(4)—N(5)	112.2 (5)
Cu(2)—N(5)—C(4)	117.0 (2)	Cu(2)—N(5)—C(6)	107.0 (3)
C(4)—N(5)—C(6)	112.9 (5)	N(5)—C(6)—C(7^i)	107.6 (5)
N(1)—C(7)—C(6^i)	109.5 (4)		

Symmetry codes: (i) $1 - x, -y, 1 - z$; (ii) $1 - x, y, \frac{3}{2} - z$.

Crystallographic computations were performed using the *SHELXTL-Plus* program package (Sheldrick, 1991).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CR1200). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Bis(μ-diphenylphosphinato-*O*:*O'*)bis(dimethylaluminium)

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Abstract

The title compound, $[Al_2(C_{12}H_{10}O_2P)_2(CH_3)_4]$, contains discrete centrosymmetric molecules each having a slightly puckered eight-membered (Al—O—P—O—)$_2$ ring. Displacements from the plane of the four O atoms are 0.28 (1) and 0.15 (1) Å for the P and Al atoms, respectively. The Al—O bond distances are 1.781 (3) and 1.794 (3) Å, while the P—O distances are 1.512 (3) and 1.505 (2) Å.

Comment

We have recently become interested in compounds containing Al—O—P linkages as soluble model compounds for aluminophosphate molecular sieves. Only a small amount of work has been carried out in this area and few compounds have been characterized fully. Coates & Mukherjee (1964) studied the reactions of trimethylaluminium with phosphinic acids to give compounds of the type $[Me_2Al(O_2PR_2)]_2$ (R = Me, Ph) and later, reactions of Al_2Me_6 with $HO(O)PX_2$ (X = F, Cl or H)

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