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# Structure of *cis*-Dichloro[bis(dimethylaminoethyl) ether]copper

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**Abstract.** $[CuCl_2(C_8H_{20}N_2O)]$, $M_r = 294{\cdot}72$, tetragonal, $P4_12_12$, $a = 12{\cdot}040\ (4)$, $c = 18{\cdot}198\ (9)$ Å, $V = 2638$ Å$^3$, $Z = 8$, $D_x = 1{\cdot}484$ Mg m$^{-3}$, $\lambda(\text{Cu}\ K\alpha) = 1{\cdot}54184$ Å, $\mu = 5{\cdot}946$ mm$^{-1}$, $F(000) = 1224$, $T = 298$ K, $R = 0{\cdot}0753$ for 1668 unique observed reflections. The copper occupies a distorted square-pyramidal environment, lying 0·372 Å above the base defined by Cl(1) and the N(1), O(4) and N(7) donors of the tridentate bis(2-dimethylaminoethyl) ether ligand. The apical site is occupied by a second chloride, Cl(2), at a distance of 2·436 (3) Å: this is considerably longer than the Cu—Cl(1) distance of 2·255 (4) Å. The geometric constraints of the ether ligand lead to large variations in the non-apical angles around Cu, the OCuN angles of 81·5 (3) and 80·4 (4)° being narrower than the Cl(1)CuN angles of 95·6 (3) and 95·3 (3)°. The complex has no crystallographically-imposed symmetry but has an approximate molecular mirror plane passing through Cl(1), Cl(2), Cu and O(4).

**Experimental.** Compound prepared by reaction of $CuCl_2.2H_2O$ with bis(2-dimethylaminoethyl) ether in boiling ethanol, crystals obtained by cooling of reaction mixture. Blue-green columnar crystal, 1·08 × 0·35 × 0·35 mm, AED2 four-circle diffractometer, graphite-monochromated Cu $K\alpha$ radiation, cell parameters from $\theta$ values of 128 reflections measured at $\pm\omega$ ($15 < \theta < 17{\cdot}5°$). For data collection, $\omega$–$2\theta$ scans with $\omega$ scan width $(0{\cdot}70 + 0{\cdot}347\tan\theta)°$, $2\theta_{\max} = 120°$, $h$ 0→13, $k$ 0→13, $l$ 0→20, no significant crystal movement or decay, initial absorption correction using $\psi$ scans, 1872 unique reflections, giving 1668 with $F > 6\sigma(F)$ for structure solution [from a Patterson synthesis (Cu) followed by iterative cycles of least-squares refinement and difference Fourier synthesis] and refinement [using full-matrix least squares on $F$ (Sheldrick, 1976)]. At isotropic convergence, final absorption correction applied empirically using *DIFABS* (Walker & Stuart, 1983). Anisotropic thermal

Table 1. *Atomic fractional coordinates with e.s.d.'s in parentheses*

| | $x$ | $y$ | $z$ | $U_{\text{iso}}$(Å$^2$) |
|---|---|---|---|---|
| Cu | 0·18845 (11) | 0·27756 (11) | 0·23467 (7) | 0·0545 (8) |
| Cl(1) | 0·3166 (3) | 0·1478 (3) | 0·26273 (19) | 0·0772 (20) |
| Cl(2) | 0·06676 (23) | 0·20028 (22) | 0·14073 (15) | 0·0579 (16) |
| N(1) | 0·2932 (7) | 0·3738 (8) | 0·1710 (4) | 0·055 (5) |
| C(1N1) | 0·3343 (12) | 0·3132 (14) | 0·1056 (7) | 0·090 (10) |
| C(1N2) | 0·3842 (9) | 0·4131 (11) | 0·2175 (7) | 0·064 (7) |
| C(2) | 0·2249 (13) | 0·4713 (12) | 0·1449 (7) | 0·078 (9) |
| C(3) | 0·1579 (11) | 0·5173 (9) | 0·2088 (8) | 0·072 (8) |
| O(4) | 0·0996 (7) | 0·4218 (6) | 0·2365 (5) | 0·067 (5) |
| C(5) | 0·0357 (12) | 0·4390 (11) | 0·3029 (8) | 0·075 (9) |
| C(6) | −0·0059 (10) | 0·3278 (12) | 0·3190 (7) | 0·067 (8) |
| N(7) | 0·0901 (9) | 0·2477 (8) | 0·3248 (5) | 0·063 (6) |
| C(7N1) | 0·0504 (13) | 0·1322 (12) | 0·3271 (8) | 0·086 (10) |
| C(7N2) | 0·1515 (11) | 0·2689 (13) | 0·3925 (6) | 0·079 (9) |

Table 2. *Selected bond lengths* (Å) *and angles* (°)

| | | | |
|---|---|---|---|
| Cu—Cl(1) | 2·255 (4) | C(2)—C(3) | 1·521 (19) |
| Cu—Cl(2) | 2·436 (3) | C(3)—O(4) | 1·438 (15) |
| Cu—N(1) | 2·068 (9) | O(4)—C(5) | 1·448 (16) |
| Cu—O(4) | 2·040 (8) | C(5)—C(6) | 1·459 (19) |
| Cu—N(7) | 2·054 (10) | C(6)—N(7) | 1·510 (16) |
| N(1)—C(1N1) | 1·482 (17) | N(7)—C(7N1) | 1·472 (18) |
| N(1)—C(1N2) | 1·464 (15) | N(7)—C(7N2) | 1·460 (17) |
| N(1)—C(2) | 1·510 (17) | | |

| | | | |
|---|---|---|---|
| Cl(1)—Cu—Cl(2) | 107·81 (12) | C(1N2)—N(1)—C(2) | 109·8 (9) |
| Cl(1)—Cu—N(1) | 95·6 (3) | N(1)—C(2)—C(3) | 109·3 (11) |
| Cl(1)—Cu—O(4) | 160·9 (3) | C(2)—C(3)—O(4) | 103·6 (10) |
| Cl(1)—Cu—N(7) | 95·3 (3) | Cu—O(4)—C(3) | 114·8 (7) |
| Cl(2)—Cu—N(1) | 100·8 (3) | Cu—O(4)—C(5) | 114·5 (7) |
| Cl(2)—Cu—O(4) | 91·22 (24) | C(3)—O(4)—C(5) | 115·9 (9) |
| Cl(2)—Cu—N(7) | 98·5 (3) | O(4)—C(5)—C(6) | 102·6 (10) |
| N(1)—Cu—O(4) | 81·5 (3) | C(5)—C(6)—N(7) | 109·7 (10) |
| N(1)—Cu—N(7) | 153·7 (4) | Cu—N(7)—C(6) | 105·9 (7) |
| O(4)—Cu—N(7) | 80·4 (4) | Cu—N(7)—C(7N1) | 112·1 (8) |
| Cu—N(1)—C(1N1) | 112·2 (8) | Cu—N(7)—C(7N2) | 110·6 (8) |
| Cu—N(1)—C(1N2) | 108·3 (7) | C(6)—N(7)—C(7N1) | 110·9 (10) |
| Cu—N(1)—C(2) | 106·3 (7) | C(6)—N(7)—C(7N2) | 109·6 (10) |
| C(1N1)—N(1)—C(1N2) | 112·0 (9) | C(7N1)—N(7)—C(7N2) | 107·8 (10) |
| C(1N1)—N(1)—C(2) | 108·2 (10) | | |

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Table 3. *Torsion angles* (°)

| | | | |
|---|---|---|---|
| Cl(1)—Cu—N(1)—C(1N1) | 62·5 (8) | Cl(1)—Cu—N(7)—C(7N2) | 61·2 (8) |
| Cl(1)—Cu—N(1)—C(1N2) | −61·5 (7) | Cl(2)—Cu—N(7)—C(6) | −71·2 (7) |
| Cl(1)—Cu—N(1)—C(2) | −179·4 (7) | Cl(2)—Cu—N(7)—C(7N1) | 49·8 (8) |
| Cl(2)—Cu—N(1)—C(1N1) | −46·9 (8) | Cl(2)—Cu—N(7)—C(7N2) | 170·1 (8) |
| Cl(2)—Cu—N(1)—C(1N2) | −170·9 (7) | N(1)—Cu—N(7)—C(6) | 65·6 (12) |
| Cl(2)—Cu—N(1)—C(2) | 71·2 (7) | N(1)—Cu—N(7)—C(7N1) | −173·4 (9) |
| O(4)—Cu—N(1)—C(1N1) | −136·5 (8) | N(1)—Cu—N(7)—C(7N2) | −53·1 (13) |
| O(4)—Cu—N(1)—C(1N2) | 99·5 (7) | O(4)—Cu—N(7)—C(6) | 18·6 (7) |
| O(4)—Cu—N(1)—C(2) | −18·4 (7) | O(4)—Cu—N(7)—C(7N1) | 139·6 (9) |
| N(7)—Cu—N(1)—C(1N1) | 176·7 (9) | O(4)—Cu—N(7)—C(7N2) | −100·1 (8) |
| N(7)—Cu—N(1)—C(1N2) | 52·6 (12) | Cu—N(1)—C(2)—C(3) | 44·9 (11) |
| N(7)—Cu—N(1)—C(2) | −65·2 (11) | C(1N1)—N(1)—C(2)—C(3) | 165·6 (10) |
| Cl(1)—Cu—O(4)—C(3) | 71·0 (11) | C(1N2)—N(1)—C(2)—C(3) | −72·0 (12) |
| Cl(1)—Cu—O(4)—C(5) | −66·7 (12) | N(1)—C(2)—C(3)—O(4) | −53·6 (12) |
| Cl(2)—Cu—O(4)—C(3) | −112·3 (7) | C(2)—C(3)—O(4)—Cu | 37·6 (11) |
| Cl(2)—Cu—O(4)—C(5) | 110·1 (8) | C(2)—C(3)—O(4)—C(5) | 174·6 (10) |
| N(1)—Cu—O(4)—C(3) | −11·6 (8) | Cu—O(4)—C(5)—C(6) | −39·2 (11) |
| N(1)—Cu—O(4)—C(5) | −149·2 (8) | C(3)—O(4)—C(5)—C(6) | −176·4 (10) |
| N(7)—Cu—O(4)—C(3) | 149·3 (8) | O(4)—C(5)—C(6)—N(7) | 56·0 (12) |
| N(7)—Cu—O(4)—C(5) | 11·7 (8) | C(5)—C(6)—N(7)—Cu | −47·6 (11) |
| Cl(1)—Cu—N(7)—C(6) | 179·8 (7) | C(5)—C(6)—N(7)—C(7N1) | −169·4 (11) |
| Cl(1)—Cu—N(7)—C(7N1) | −59·1 (8) | C(5)—C(6)—N(7)—C(7N2) | 71·7 (13) |

parameters for all non-H atoms, H atoms in fixed, calculated positions, secondary-extinction parameter refined to $0{\cdot}0017\,(7) \times 10^{-4}$. At final convergence, $R = 0{\cdot}0753$, $wR = 0{\cdot}1021$, $S = 1{\cdot}161$ for 128 parameters, max. shift/e.s.d. in final cycle 0·02, max. and min. residues in final difference Fourier synthesis 0·84, −0·90 e Å$^{-3}$ respectively. The weighting scheme $w^{-1} = \sigma^2(F) + 0{\cdot}000214F^2$ gave satisfactory agreement analyses. Scattering factors were inlaid (Sheldrick, 1976) except for Cu (Cromer & Mann, 1968). Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1, while selected bond lengths, angles and torsion angles appear in Tables 2 and 3.* The atom-numbering scheme for the molecule is shown in Fig. 1, which was generated using *ORTEP* (Mallinson & Muir, 1985). Molecular geometry calculations were performed using *CALC* (Gould & Taylor, 1985). The high *R* values reflect the poor quality of the crystal, which was the best that could be obtained.

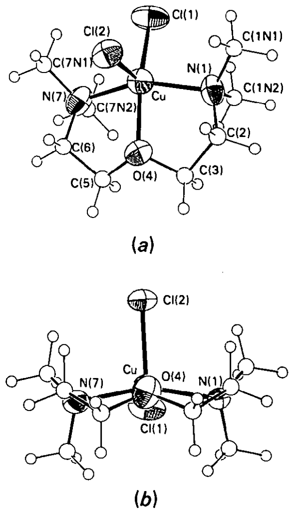

Fig. 1. (*a*) A general view of the molecule showing atom-numbering scheme. (*b*) A view parallel to the mean basal plane defined by Cl(1), N(1), N(7) and O(4). In both views thermal ellipsoids are drawn at the 30% probability level, excepting those of C and H which have artificial radii of 0·10 and 0·15 Å respectively for clarity.

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* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and details of least-squares mean planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51105 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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# Structure of Di-μ-thio-bis[(diethyldithiophosphinato)thiotungsten(V)]

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**Abstract.** $[\mathrm{W_2S_4\{(C_2H_5)_2PS_2\}_2}]$, $M_r = 802{\cdot}4$, monoclinic, $P2_1/c$, $a = 12{\cdot}059\,(3)$, $b = 12{\cdot}691\,(2)$, $c = 14{\cdot}697\,(4)$ Å, $\beta = 101{\cdot}25\,(2)°$, $V = 2206\,(1)$ Å$^3$, $Z = 4$, $D_m = 2{\cdot}41$, $D_x = 2{\cdot}42$ Mg m$^{-3}$, $\lambda(\mathrm{Mo}\ K\alpha) = 0{\cdot}71073$ Å, $\mu = 12{\cdot}02$ mm$^{-1}$, $F(000) = 1496$, room temperature, $R = 0{\cdot}037$ for 4877 observed reflections

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