

Table 2. Bond angles (°) with their standard deviations in parentheses

N(1)—Co—N(2)	81.9 (2)	N(1)—C(3)—C(4)	122.0 (6)
N(1)—Co—N(3)	90.7 (2)	C(3)—C(4)—C(5)	129.1 (7)
N(1)—Co—N(4)	94.3 (2)	C(4)—C(5)—C(6)	116.8 (7)
N(1)—Co—C(1)	176.7 (2)	C(5)—C(6)—C(7)	120.2 (7)
N(1)—Co—C(2)	91.9 (2)	N(1)—C(7)—C(6)	122.0 (6)
N(2)—Co—N(3)	93.8 (2)	N(1)—C(7)—C(8)	115.0 (5)
N(2)—Co—N(4)	174.2 (2)	C(6)—C(7)—C(8)	123.1 (6)
N(2)—Co—C(1)	94.9 (3)	N(2)—C(8)—C(7)	115.2 (5)
N(2)—Co—C(2)	89.9 (3)	N(2)—C(8)—C(9)	120.9 (6)
N(3)—Co—N(4)	81.8 (2)	C(7)—C(8)—C(9)	123.9 (6)
N(3)—Co—C(1)	88.6 (2)	C(8)—C(9)—C(10)	118.7 (7)
N(3)—Co—C(2)	175.7 (3)	C(9)—C(10)—C(11)	119.4 (8)
N(4)—Co—C(1)	88.8 (3)	C(10)—C(11)—C(12)	121.3 (8)
N(4)—Co—C(2)	94.6 (3)	N(2)—C(12)—C(11)	118.7 (7)
C(1)—Co—C(2)	89.0 (3)	N(3)—C(13)—C(14)	121.6 (6)
C(23)—Al—C(25)	110.4 (4)	C(13)—C(14)—C(15)	119.7 (7)
C(23)—Al—C(27)	114.6 (6)	C(14)—C(15)—C(16)	121.2 (8)
C(23)—Al—C(29)	106.7 (6)	C(15)—C(16)—C(17)	116.2 (7)
C(25)—Al—C(27)	117.4 (7)	N(3)—C(17)—C(16)	122.9 (6)
C(25)—Al—C(29)	108.8 (7)	N(3)—C(17)—C(18)	114.7 (5)
C(27)—Al—C(29)	97.4 (8)	C(16)—C(17)—C(18)	122.4 (6)
Co—N(1)—C(3)	128.0 (4)	N(4)—C(18)—C(17)	113.6 (5)
Co—N(1)—C(7)	113.1 (4)	N(4)—C(18)—C(19)	123.7 (6)
C(3)—N(1)—C(7)	118.8 (5)	C(17)—C(18)—C(19)	122.7 (6)
Co—N(2)—C(8)	114.4 (4)	C(18)—C(19)—C(20)	117.5 (7)
Co—N(2)—C(12)	124.7 (5)	C(19)—C(20)—C(21)	120.7 (7)
C(8)—N(2)—C(12)	120.9 (6)	C(20)—C(21)—C(22)	117.7 (7)
Co—N(3)—C(13)	128.7 (4)	N(4)—C(22)—C(21)	122.4 (6)
Co—N(3)—C(17)	112.9 (4)	Al—C(23)—C(24)	115.5 (6)
C(13)—N(3)—C(17)	118.4 (5)	Al—C(25)—C(26)	115.9 (8)
Co—N(4)—C(18)	116.9 (4)	Al—C(27)—C(28)	113.6 (14)
Co—N(4)—C(22)	125.2 (4)	Al—C(29)—C(30)	108.3 (17)
C(18)—N(4)—C(22)	117.9 (5)		

those of [Co(2,2'-bipyridine)<sub>2</sub>NO<sub>3</sub>](NO<sub>3</sub>)(OH)·4H<sub>2</sub>O (Reimann, Zocchi, Mighell & Santoro, 1971).

The [Al(C<sub>2</sub>H<sub>5</sub>)<sub>4</sub>]<sup>-</sup> ion is nearly tetrahedral. However, one of the four Al—C bond distances, 2.269 (18) Å [accompanied by an unusually short C—C bond, 1.12 (3) Å], is considerably longer than the others,

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### trans-Dichlorobis(2,4-dimethylthiazole)copper(II)

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**Abstract.** C<sub>10</sub>H<sub>14</sub>Cl<sub>2</sub>CuN<sub>2</sub>S<sub>2</sub>, Cu(C<sub>4</sub>H<sub>7</sub>NS)<sub>2</sub>Cl<sub>2</sub>, monoclinic, C2/c, *a* = 12.320 (13), *b* = 8.760 (7), *c* = 14.592 (11) Å, β = 105.81 (6)°, *U* = 1515.2 Å<sup>3</sup>, *Z* = 4, *D<sub>c</sub>* = 1.580, *D<sub>m</sub>* (floatation in benzene/bromoforn) = 1.60 (2) Mg m<sup>-3</sup>. The complex is square planar and monomeric, with Cu—N and Cu—Cl bond lengths of 1.985 (4) and 2.261 (2) Å, respectively. There is

2.002 (18)–2.031 (11) Å, which are nearly equal to those found in Li[Al(C<sub>2</sub>H<sub>5</sub>)<sub>4</sub>] (Gerteis, Dickerson & Brown, 1964). The other C—C bond lengths range from 1.54 (2) to 1.41 (3) Å. Such unusual bond lengths are probably due to the degradation of the anion in the crystal used; such anions are usually highly reactive (Mole & Jeffery, 1972).

Figs. 1 and 2 were drawn by *TSD: XTAL* which is an interactive modelling program system for computer graphics (Takenaka & Sasada, 1978).

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approximate 75/25 disorder of the dimethylthiazole ring, involving ring atoms S and C(5).

**Introduction.** The complex was prepared by an analogous method to that used previously for the synthesis of the thiazole analog (Estes, Gavel, Hatfield & Hodgson, 1978). To a warm solution of 6 mmol 2,4-dimethylthiazole (Pfaltz and Bauer, Inc., Stamford, Connecticut) in 1 ml of absolute ethanol was added 3

mmol of  $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$  in 8 ml of absolute ethanol. The precipitate was recrystallized from dimethylformamide/dichloromethane, yielding purple crystals of the title complex. The crystal used for data collection was a hexagonal plate; the separation between opposite faces of the hexagon was approximately 0.4 mm while the thickness of the plate was approximately 0.2 mm. Precession and Weissenberg photography indicated that the crystals were monoclinic, the observed systematic absences of  $hkl$  for  $(h+k)$  odd and  $h0l$  for  $l$  odd indicating the space groups  $C2/c$  or  $Cc$ ; the failure of our attempts (see below) to refine the structure in the latter space group, coupled with the successful refinement in the former, suggest that the centrosymmetric choice is correct. Intensity data were collected using Mo  $K\alpha$  radiation on a four-circle Picker automatic diffractometer equipped with a graphite monochromator. The cell constants, based on an assumed wavelength  $\lambda(\text{Mo } K\alpha_1) = 0.70926 \text{ \AA}$ , were obtained by least-squares methods from the diffractometer settings of 12 reflections. The intensities were recorded by the  $\theta/2\theta$  scan method at a scan rate of  $1.0^\circ (2\theta) \text{ min}^{-1}$  up to  $2\theta(\text{Mo}) = 60^\circ$ . Stationary-crystal, stationary-counter background counts of 20 s were measured on each side of the peak. To allow for the presence of both  $K\alpha_1$  and  $K\alpha_2$  radiations, the peaks were scanned from  $0.9^\circ$  in  $2\theta$  below the calculated  $K\alpha_1$  peak position to  $0.9^\circ$  in  $2\theta$  above the calculated  $K\alpha_2$  peak position. The data were corrected for the usual geometric factors and for absorption; the transmission coefficients ranged from 0.49 to 0.70 with an average value of 0.63. Of the 2300 reflections recorded, 1742 were independent data with  $I > 3\sigma(I)$ ; only these latter reflections were used in the subsequent analysis. The computer programs used in this analysis have been described elsewhere (Lewis & Hodgson, 1974).

The locations of Cu, Cl and S were deduced from a three-dimensional Patterson function. A difference Fourier map phased by these three atoms showed all of the remaining non-hydrogen atoms. The H atoms were located in a subsequent difference Fourier map, with the exception of the H atom on ring atom C(5) (see below). In view of the apparent disorder of the rings (see below) it is, perhaps, surprising that these methyl H atoms appeared so prominently in this difference Fourier synthesis as to be readily and unambiguously identified. The metrical parameters involving these H atoms in the final refinement (C—H distances of 0.95 to 1.09 Å, H—C—H angles of  $120.4$  to  $131.9^\circ$ ), which are well within the normally observed ranges (Churchill, 1973), strongly support our assignment. The H atoms were assigned fixed isotropic thermal parameters of  $7.0 \text{ \AA}^2$ , and their locations were not varied; the non-hydrogen atoms were refined anisotropically. Least-squares refinements were carried out on  $F$ , the function minimized being  $\sum w(|F_o| - |F_c|)^2$  where the weight  $w$  was assigned as  $4F_o^2/\sigma^2(F_o^2)$ . No correction

for secondary extinction was applied. The scattering factors, including the anomalous-dispersion terms for Cu, Cl, and S, were taken from *International Tables for X-ray Crystallography* (1974). Anisotropic refinement of this model converged to values of the usual agreement factors  $R_1 = \sum (|F_o| - |F_c|)/\sum |F_o|$  and  $R_2 = [\sum w(|F_o| - |F_c|)^2/\sum w(F_o^2)]^{1/2}$  of 0.080 and 0.137. Attempts to refine the structure in space group  $Cc$  were no more successful, converging to values of 0.079 and 0.133 for  $R_1$  and  $R_2$ , respectively. It was apparent that the thermal parameters for the S atom were excessive while those for ring atom C(5) were extremely small; this suggested to us that there may be some disorder of the rings, and so models with one of these positions occupied by various fractions  $\alpha$  of S and  $(1 - \alpha)$  of C and the other occupied by  $\alpha$  of C and  $(1 - \alpha)$  of S were attempted. The best agreement was obtained with a model in which the position originally ascribed to S contained 75% S and 25% C, while the position originally assigned as C(5) contained 25% S and 75% C. Refinement of this model yielded  $R_1 = 0.052$  and  $R_2 = 0.074$ , based on 1742 observations and 78 variables. The fact that this model did not lead to an apparent 50/50 disorder lends strong support to our conclusion that the system is disordered. If the space group were in fact  $Cc$ , with no disorder, the centrosymmetric disordered model would be expected to yield approximate 50/50 disorder. The positional parameters, along with their estimated standard deviations as estimated from the inverse matrix, are listed in Table 1.\*

\* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34612 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Positional parameters in  $\text{Cu}(\text{C}_5\text{H}_7\text{NS})_2\text{Cl}_2$

	Hydrogen-atom parameters were not varied.		
	<i>x</i>	<i>y</i>	<i>z</i>
Cu	0.0	0.19986 (6)	0.25
Cl	0.06330 (7)	0.20355 (12)	0.11841 (6)
S*	-0.34898 (9)	0.11658 (18)	0.07065 (9)
N	-0.1576 (2)	0.2018 (3)	0.1677 (2)
C(2)	-0.2154 (3)	0.0770 (5)	0.1328 (2)
C(4)	-0.2176 (3)	0.3318 (5)	0.1451 (2)
C(5)†	-0.3395 (2)	0.3087 (3)	0.0860 (2)
C(6)	-0.1674 (4)	-0.0778 (6)	0.1448 (4)
C(7)	-0.1650 (3)	0.4825 (5)	0.1747 (3)
H(1C6)	-0.1500	-0.1100	0.2200
H(2C6)	-0.0931	-0.0833	0.1270
H(3C6)	-0.2158	-0.1545	0.1044
H(1C7)	-0.1300	0.5000	0.2400
H(2C7)	-0.2211	0.5592	0.1528
H(3C7)	-0.1051	0.4942	0.1423

\* Position occupied by 75% S, 25% C.

† Position occupied by 25% S, 75% C.

**Discussion.** The solid-state coordination geometries of  $\text{Cu}^{\text{II}}$  complexes of the type  $\text{ML}_2\text{X}_2$  have been of considerable interest for many years. For the case where  $L$  is pyridine (py) or its derivatives, it has been found that increased substitution on the ligand at sites near the ligating atom leads to decreased coordination number at the metal. Thus, the parent complexes  $\text{Cu}(\text{py})_2\text{X}_2$  are six-coordinate chains (Dunitz, 1957; Kupčik & Āurovič, 1960; Morosin, 1975), the monosubstituted 2-methylpyridine complexes are five-coordinate dimers (Duckworth & Stephenson, 1969; Singh, Jeter, Hatfield & Hodgson, 1972), and the disubstituted 2,3-dimethylpyridine analogs are four-coordinate monomers (Stählin & Oswald, 1971). Our recent crystallographic and magnetic study of the thiazole complexes  $\text{Cu}(\text{tz})_2\text{X}_2$  (Estes *et al.*, 1978), along with the spectroscopic investigations of Eilbeck, Holmes & Underhill (1967), has demonstrated conclusively that these unsubstituted complexes have six-coordinate structures which are similar to those of the pyridine complexes. Consequently, we believed that it was of value to study the structures of complexes of substituted thiazoles to see if the trend described above would hold for this ligand system also.

A view of the complex is shown in Fig. 1. The complex consists of monomeric four-coordinate  $\text{Cu}(\text{Me}_2\text{tz})_2\text{Cl}_2$  units which are well separated from each other. The closest intermolecular  $\text{Cu}\cdots\text{Cl}$  separation in the crystal is 6.302 Å, while the shortest  $\text{Cu}\cdots\text{Cu}$  separation is  $>6.5$  Å. The  $\text{Cu}^{\text{II}}$  center lies on a crystallographic twofold axis. The geometry around Cu is square planar, with no atom deviating from the five-atom  $\text{CuN}_2\text{Cl}_2$  least-squares plane by more than 0.02 Å. The two independent *cis* Cl—Cu—N angles are 89.6 (1) and 90.4 (1)° while the N—Cu—N' and Cl—Cu—Cl' *trans* angles are 179.0 (1) and 178.36 (8)°, respectively. The Cu—Cl bond length of 2.261 (2) Å is, as expected, shorter than the bridging bond [2.322 (2) Å] in the thiazole complex, but is similar to other terminal Cu—Cl bonds (Bream, Estes & Hodgson, 1975; Estes & Hodgson, 1976; Phelps,

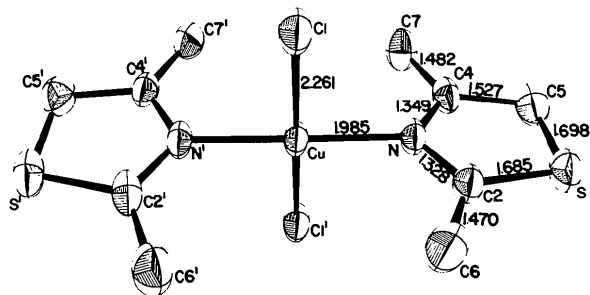


Fig. 1. View of the complex  $[\text{Cu}(\text{Me}_2\text{tz})_2\text{Cl}_2]$ , showing bond lengths (Å). Thermal ellipsoids are drawn at the 40% probability level, and H atoms are omitted for clarity. The model shown is the major contributor of the two disordered structures (see text). E.s.d.'s on bond lengths are 0.002 Å for Cu—Cl, 0.004 Å for Cu—N, 0.006 Å for others.

Table 2. Bond angles (°) in  $\text{Cu}(\text{Me}_2\text{tz})_2\text{Cl}_2$

Cl—Cu—Cl'	178.36 (8)	S—C(2)—N	112.3 (4)
Cl—Cu—N	89.6 (1)	S—C(2)—C(6)	123.8 (4)
Cl—Cu—N'	90.4 (1)	N—C(2)—C(6)	123.9 (4)
N—Cu—N'	179.0 (1)	N—C(4)—C(5)	114.5 (4)
Cu—N—C(2)	124.0 (3)	N—C(4)—C(7)	120.9 (4)
Cu—N—C(4)	122.4 (3)	C(5)—C(4)—C(7)	124.6 (4)
C(2)—N—C(4)	113.6 (4)	S—C(5)—C(4)	103.1 (4)
C(2)—S—C(5)	96.4 (2)		

Goodman & Hodgson, 1976; Wilson, Wasson, Hatfield & Hodgson, 1978) in related complexes. The Cu—N length of 1.985 (4) Å is similar to those of 1.990 (2) Å in  $\text{Cu}(\text{tz})_2\text{Cl}_2$  and 2.004 (5) in  $\text{Cu}(\text{py})_2\text{Cl}_2$ , and to those in a variety of related  $\text{Cu}^{\text{II}}$  complexes (Hodgson, 1975, 1977, and references therein).

The disorder at S and C(5) makes the bond lengths and angles involving these atoms less reliable than others, but within this limitation the geometry of the 2,4-dimethylthiazole ( $\text{Me}_2\text{tz}$ ) ligands is normal. The thiazole ring is planar, with no atom deviating from the least-squares plane by more than 0.006 Å; the exocyclic methyl C atoms are 0.04 and 0.06 Å out of this plane. The ring planes are inclined at an angle of 85.1° to the base plane of the metal, and at an angle of 9.8° to each other. It is apparent (see Fig. 1) that this orientation brings all four methyl groups into positions which hinder axial approach to Cu and, therefore, prevent the formation of dimeric or polymeric species. Unlike the situation in the thiazole complex, where the chains are linked by weak intermolecular S $\cdots$ S contacts of 3.62 Å, there are no intermolecular S $\cdots$ S separations less than twice the reported (Pauling, 1960) van der Waals radius (1.85 Å) of S. In the absence of any potential donor, there is no hydrogen bonding in the crystals. The bond lengths are shown in Fig. 1, while bond angles in the complex are listed in Table 2.

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**$\mu$ -Oxo-bis{[2,3-dimethyl-2,3-butanediolato(1-)] [2,3-dimethyl-2,3-butanediolato(2-)]oxomolybdenum(VI)}**

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**Abstract.**  $\text{Mo}_2(\text{C}_6\text{H}_{12}\text{O}_2)_2(\text{C}_6\text{H}_{13}\text{O}_2)_2\text{O}_3$ ,  $\text{C}_{24}\text{H}_{50}\text{Mo}_2\text{O}_{11}$ , monoclinic,  $C2/c$ ,  $a = 16.907$  (4),  $b = 13.450$  (2),  $c = 13.556$  (2) Å,  $\beta = 92.6$  (1)°,  $V = 3079.6$  Å<sup>3</sup>,  $M_r = 690.51$ ,  $D_o = 1.52$  (7),  $D_c = 1.52$  Mg m<sup>-3</sup>,  $Z = 4$ ,  $\mu(\text{Cu } K\alpha) = 7.478$  mm<sup>-1</sup>. The yellow complex is based on a O=Mo—O—Mo=O core in which each Mo atom is coordinated by two bidentate pinacol ligands, one of which is singly deprotonated and the other doubly deprotonated. Two intramolecular hydrogen bonds link ligands attached to different Mo atoms.

**Introduction.** Samples of the title compound were prepared (Knobler, Penfold, Robinson, Wilkins & Yong, 1979) as translucent yellow crystals. Crystals are stable in a dark, dry atmosphere but otherwise slowly turn dark blue. Laue symmetry,  $2/m$ , and the systematic absences observed in photographs, established the space group as either  $Cc$  or  $C2/c$ . The crystal used for intensity measurements was a hexagonal prism of length 0.2 mm and mean cross-section 0.1 mm. It was sealed in a 0.3 mm tapered capillary tube, and intensities were recorded with a Hilger & Watts four-circle diffractometer, Ni-filtered Cu  $K\alpha$  radiation ( $\lambda = 1.5418$  Å) and the  $\theta$ - $2\theta$  technique. 1007 independent reflections were collected in the range  $0 < 2\theta < 110^\circ$ . Of these, 885 had  $I > 3\sigma(I)$  and were used in the structure determination and refinement. The procedures for data collection and assignment of errors were as described by Countryman & Penfold (1972). Corrections for absorption and isotropic extinction were applied.

The structure was solved by the heavy-atom method with the unique Mo atom, located from the Patterson function, used for phasing. Consideration of vector peaks arising from symmetry-related atoms indicated the space group  $C2/c$  rather than  $Cc$  and this was confirmed by the satisfactory refinement. Difference syntheses showed all O, C and several H atoms. Least-squares refinement was carried out, varying three positional parameters for all Mo, C and O atoms and the non-methyl H atom, the methyl H positions being constrained to ideal tetrahedral geometries. Mo atoms only were assigned anisotropic thermal parameters. The final  $R$  was 0.073 for all 885 reflections. In the full-matrix least-squares procedure, the function minimized was  $w\|F_o - |F_c|\|^2$  and the weights,  $w$ , were  $4|F_o|^2/(\sigma|F_o|^2)^2$ . Scattering factors for Mo, C and O were from Cromer & Mann (1968), those of H from Stewart, Davidson & Simpson (1965); anomalous-dispersion corrections were from Cromer & Liberman (1970). Positional and thermal parameters are listed in Table 1,\* selected bond distances and angles in Tables 2 and 3.

**Discussion.** The title compound results from the coordination to a *cis*-dioxomolybdenum(VI) core of two molecules of a vicinal diol in which the C atoms

\* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34616 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.