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**Key indicators**

Single-crystal X-ray study  
 T = 293 K  
 Mean  $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$   
 R factor = 0.043  
 wR factor = 0.107  
 Data-to-parameter ratio = 27.7

For details of how these key indicators were  
 automatically derived from the article, see  
<http://journals.iucr.org/e>.

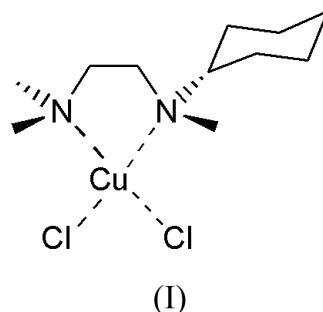
**Dichloro(*N*-cyclohexyl-*N,N',N'*-trimethyl-  
 ethylenediamine- $\kappa^2$ *N,N'*)copper(II)**

The title compound,  $[\text{CuCl}_2(\text{C}_{11}\text{H}_{24}\text{N}_2)]$ , crystallizes as four-coordinate monomers with distorted square-planar geometry. The complex is chiral and forms racemic crystals. Molecules which have an *S*-configuration at nitrogen have a  $\lambda$ -conformation of the five-membered chelate ring, and those with *R*-configuration have a  $\delta$ -conformation. The title compound forms infinite one-dimensional chains *via* weak intermolecular  $\text{Cl} \cdots \text{H}-\text{C}$  hydrogen contacts.

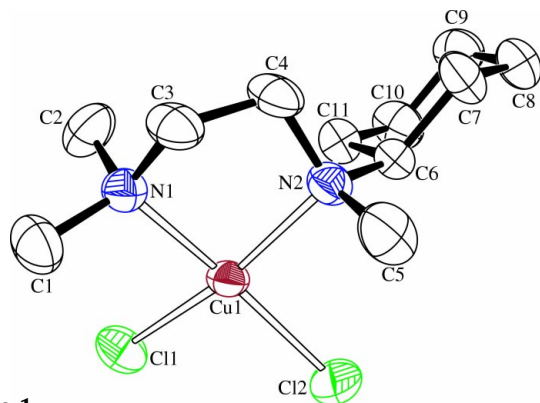
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**Comment**

The *N*-cyclohexyl-*N,N',N'*-trimethylethylenediamine (cytrimedia) ligand is chiral due to a stereogenic N atom but, in solution, it is optically inactive. Reaction of cytrimedia with copper(II) chloride yields the title compound, (I), which is also chiral. Since  $[\text{CuCl}_2(\text{cytrimedia})]$  is stereochemically labile and racemizes in solution, it could be subject to total spontaneous resolution if the complex crystallizes as a conglomerate (Eliel *et al.*, 1994). Crystal growth from secondary nucleation (or single-colony growth) could produce a highly enantio-enriched product in high yield (since the enantiomer that does not crystallize is converted in solution), which would constitute a rare example of absolute asymmetric synthesis (Vestergren *et al.*, 2003). However, we have so far only been able to isolate a racemic phase of  $[\text{CuCl}_2(\text{cytrimedia})]$ , which crystallizes in centrosymmetric  $P2_1/n$ . Apart from the stereogenic N atom, the complex also displays another element of chirality: the five-membered cytrimedia chelate ring is conformationally chiral. Molecules which have an *S*-configuration at nitrogen have a  $\lambda$ -conformation of the five-membered chelate ring, and those with *R*-configuration have a  $\delta$ -conformation.



Reacting the *N,N,N',N'*-tetramethylethylenediamine (tmeda) or *N,N,N',N'*-tetraethylethylenediamine (teeda) ligands with equimolar amounts of copper(II) chloride or copper(II) bromide gives complexes of formula  $[\text{CuX}_2L]_n$  ( $X = \text{Cl}$  or  $\text{Br}$ ,  $L = \text{tmeda}$  or  $\text{teeda}$ ; Bertini & Mani, 1967). Both di- $\mu$ -chloro-bis[chloro[tmeda]copper(II)],  $[\text{CuCl}_2(\text{tmeda})]_2$



**Figure 1**

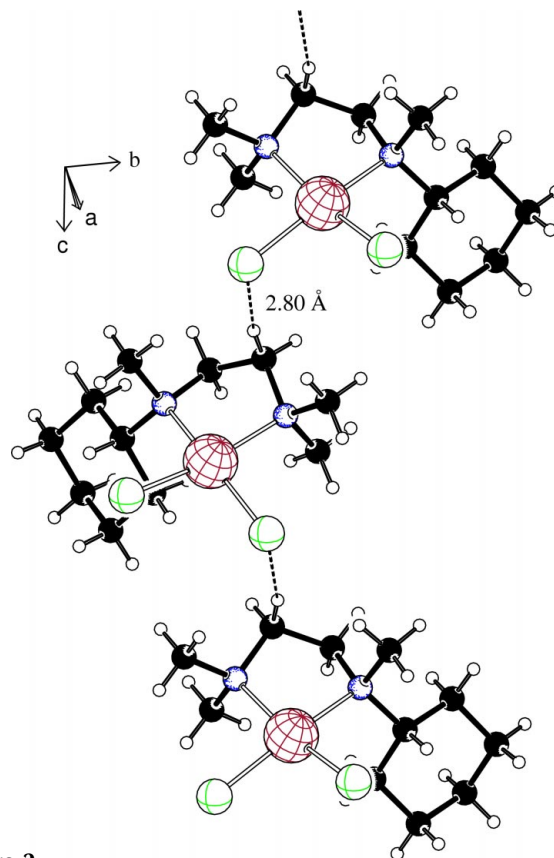
Plot of  $[\text{CuCl}_2(\text{cytrimedia})]$ , with displacement ellipsoids drawn at the 50% probability level. All H atoms have been omitted.

(Estes *et al.*, 1975) and di- $\mu$ -bromo-bis[bromo[tmeda]-copper(II)],  $[\text{CuBr}_2(\text{tmeda})]_2$  (Luukkonen & Pajunen, 1973) were found to be dimers bridged by two halide ligands. The copper(II) ion is coordinated by three chloride ligands and two N atoms in a square-pyramidal disposition. However,  $[\text{CuCl}_2(\text{teeda})]$  (Estes & Hodgson, 1975) is a four-coordinate monomer with distorted square-planar geometry. The more bulky ethyl groups probably prevent aggregation, resulting in a monomeric structure. Molecules of  $[\text{CuCl}_2(\text{cytrimedia})]$  have the even bulkier cyclohexyl group (in the chair conformation) and also form monomers, as can be seen in Fig. 1, displaying a roughly square-planar copper(II) ion, which is distorted towards tetrahedral geometry. The distortion is a result of the fact that N1 and Cl2 lie 0.2294 (9) and 0.1944 (8) Å, respectively, below the least-squares plane through atoms Cu, Cl1, Cl2, N1 and N2, and that N2 and Cl1 lie 0.2290 (9) and 0.1991 (8) Å, respectively, above the same plane. The distances between Cu1 and the two *trans* N1 and Cl2 atoms of 2.0471 (19) and 2.2239 (8) Å, respectively, are somewhat shorter than the Cu–N2 distance of 2.0722 (18) Å and the Cu–Cl1 distance of 2.2472 (7) Å. In the  $[\text{CuCl}_2(\text{teeda})]_2$  complex, the two Cu–N and the two Cu–Cl distances [2.074 (2) and 2.246 (1) Å, respectively] are the same as those in (I). The longer Cu–N2 distance in  $[\text{CuCl}_2(\text{cytrimedia})]$  may be explained by the presence of a more bulky cyclohexyl substituent on atom N2.

The methyl, and particularly the cyclohexyl groups, of the cytrimedia ligand probably prevent the formation of a six-coordinate chain structure, as found in pyridine complexes such as  $[\text{Cu}(\text{py})_2\text{Cl}_2]$ , where each Cu atom is surrounded by four bridging chloride ligands and two N atoms in a distorted octahedral arrangement (Dunitz, 1957). Instead, the title compound forms infinite one-dimensional chains *via* weak intermolecular hydrogen contacts ( $\text{Cl}\cdots\text{H}-\text{C}$ ), where the acceptor-hydrogen distance is 2.80 Å, as can be seen in Fig. 2.

## Experimental

For the synthesis of cytrimedia, toluene (100 ml), *N,N*-dimethylethylenediamine (20 ml, 0.18 mol) and cyclohexanone (19 ml, 0.18 mol) were refluxed for 12 h using a Dean–Stark trap. Evapora-



**Figure 2**

The infinite chain formed from weak intermolecular hydrogen-bond interactions (dashed lines).

tion *in vacuo* yielded a red oil. Ethanol (100 ml) and  $\text{NaBH}_4$  (6.90 g, 0.18 mol) were added in small portions and the solution was stirred for 2 h. The reaction was quenched with water (70 ml) and the product was extracted with  $\text{CH}_2\text{Cl}_2$  ( $3 \times 100$  ml). The combined organic phases were dried over  $\text{MgSO}_4$ . Evaporation *in vacuo* yielded a bright yellow oil. Formaldehyde (37% in water, 30 ml, 0.38 mol) and formic acid (14 ml, 0.36 mol) were added to the oil, and the solution was stirred at 343 K for 12 h. The reaction flask was cooled in ice and  $\text{NaOH}$  (20% in water) was added until the pH was greater than 14. The solution was saturated with  $\text{NaCl}$ , extracted with diethyl ether ( $3 \times 100$  ml) and the combined ether phases were dried over  $\text{K}_2\text{CO}_3$ . Evaporation *in vacuo* gave a yellow oil which was distilled under reduced pressure from  $\text{CaH}_2$  (326–329 K,  $9.3 \times 10^{-3}$  mbar) to yield *N*-cyclohexyl-*N,N,N'*-trimethylethylenediamine (19.1 g, 57%) as a colourless oil.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  1.07 (1H, *m*,  $\text{C}_6\text{H}_{11}$ ), 1.20 (4H, *m*,  $\text{C}_6\text{H}_{11}$ ), 1.61 (1H, *m*,  $\text{C}_6\text{H}_{11}$ ), 1.78 (4H, *m*,  $\text{C}_6\text{H}_{11}$ ), 2.22 (6H, *s*,  $\text{CH}_3$ ), 2.26 (3H, *s*,  $\text{CH}_3$ ), 2.32 (1H, *m*,  $\text{C}_6\text{H}_{11}$ ), 2.36 (2H, *t*,  $\text{CH}_2$ ), 2.64 (2H, *t*,  $\text{CH}_2$ ).

For the synthesis of  $[\text{CuCl}_2(\text{cytrimedia})]$ , all manipulations were carried out under nitrogen, using standard Schlenk techniques. Dichloromethane was distilled from  $\text{CaH}_2$  and stored over 4 Å molecular sieves. Toluene was distilled from sodium/benzophenone prior to use. The dihydrate of copper(II) chloride (0.34 g, 2.0 mmol) was suspended in dichloromethane (1 ml) and *N*-cyclohexyl-*N,N,N'*-trimethylethylenediamine (0.37 g, 2.0 mmol) was added to yield a dark-blue solution. The solution was stirred for a few minutes and then toluene (4 ml) was layered over the solution. Blue single crystals suitable for X-ray analysis formed in 74% yield (0.47 g) after 12 h at ambient temperature.

Crystal data

[CuCl<sub>2</sub>(C<sub>11</sub>H<sub>24</sub>N<sub>2</sub>)]  
*M<sub>r</sub>* = 318.76  
 Monoclinic, *P*2<sub>1</sub>/*n*  
*a* = 7.8173 (19) Å  
*b* = 19.979 (4) Å  
*c* = 9.3411 (18) Å  
 β = 91.004 (9)°  
*V* = 1458.7 (5) Å<sup>3</sup>  
*Z* = 4

*D<sub>x</sub>* = 1.451 Mg m<sup>-3</sup>  
 Mo Kα radiation  
 Cell parameters from 13 113 reflections  
 θ = 2.4–31.9°  
 μ = 1.84 mm<sup>-1</sup>  
*T* = 293 (2) K  
 Needle, blue  
 0.5 × 0.2 × 0.2 mm

Data collection

Rigaku R-AXIS IIC image-plate diffractometer  
 φ scans  
 Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2000)  
*T<sub>min</sub>* = 0.493, *T<sub>max</sub>* = 0.692  
 13 113 measured reflections

4020 independent reflections  
 3372 reflections with *I* > 2σ(*I*)  
*R<sub>int</sub>* = 0.054  
 θ<sub>max</sub> = 31.9°  
*h* = -10 → 10  
*k* = -27 → 28  
*l* = -12 → 12

Refinement

Refinement on *F*<sup>2</sup>  
*R*[*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.043  
*wR*(*F*<sup>2</sup>) = 0.107  
*S* = 1.07  
 4020 reflections  
 145 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0486P)^2 + 0.3327P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 (Δ/σ)<sub>max</sub> = 0.001  
 Δρ<sub>max</sub> = 0.59 e Å<sup>-3</sup>  
 Δρ<sub>min</sub> = -0.45 e Å<sup>-3</sup>

Table 1

Selected geometric parameters (Å, °).

|            |             |             |            |
|------------|-------------|-------------|------------|
| Cu1—N1     | 2.0471 (19) | Cu1—Cl2     | 2.2239 (8) |
| Cu1—N2     | 2.0722 (18) | Cu1—Cl1     | 2.2472 (7) |
| N1—Cu1—N2  | 85.31 (8)   | N1—Cu1—Cl1  | 91.62 (6)  |
| N1—Cu1—Cl2 | 168.35 (6)  | N2—Cu1—Cl1  | 167.53 (6) |
| N2—Cu1—Cl2 | 92.84 (6)   | Cl2—Cu1—Cl1 | 92.54 (3)  |

All H atoms were included in calculated positions (C—H = 0.96–0.97 Å) and refined using a riding model, with *U*<sub>iso</sub>(H) = 1.2 or 1.5 times *U*<sub>eq</sub>(parent atom)

Data collection: *CrystalClear* (Rigaku, 2000); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *PLUTON* (Spek, 1990); software used to prepare material for publication: *SHELXL97*.

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