# The Structure of a Conformationally Strained Macrocyclic Copper(II) Complex with Dimethyl 1-Thia-4,7-diaza-4,7-cyclononanediacetate 

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#### Abstract

Chloro(dimethyl 1-thia-4,7-diaza-4,7cyclononanediacetate)copper(II) perchlorate, [Cu$\left.\left(\mathrm{C}_{12} \mathrm{H}_{22} \mathrm{~N}_{2} \mathrm{O}_{4} \mathrm{~S}\right) \mathrm{Cl}\right] \mathrm{ClO}_{4}, \quad M_{r}=488 \cdot 83$, monoclinic, $P 2_{1}, a=13.808$ (2), $b=8.195$ (2), $c=8.702$ (2) $\AA, \beta$ $=104.68(2)^{\circ}, \quad V=952.5(7) \AA^{3}, \quad Z=2, \quad D_{x}=$ $1.70 \mathrm{~g} \mathrm{~cm}^{-3}, \lambda(\mathrm{Mo} \mathrm{K} \mathrm{\alpha})=0.71073 \AA, \mu=15.8 \mathrm{~cm}^{-1}$, $F(000)=502, \quad T=294 \mathrm{~K}, \quad R=0.043 \quad$ for 1757 observed reflections. The Cu ion has a Jahn-Teller distorted octahedral coordination geometry: the Cu ion is bound strongly to the two N atoms $[\mathrm{Cu}-\mathrm{N}(7)$ $2.010(5), \mathrm{Cu}-\mathrm{N}(4) 2.051(4) \AA]$, the Cl ion $[\mathrm{Cu}-$ $\mathrm{Cl}(1) 2 \cdot 241(2) \AA]$ and one of the ester O atoms $[\mathrm{Cu}-\mathrm{O}(17) 2.058(4) \AA$ ], but is only weakly bound to the S atom $[\mathrm{Cu}-\mathrm{S}(1) 2 \cdot 635(2) \AA]$ and to the carbonyl O atom $[\mathrm{Cu}-\mathrm{O}(12) 2.554$ (4) $\AA$ ] trans to $\mathrm{S}(1)$. The nine-membered macrocyclic ring adopts a rather strained conformation to accommodate the geometric and donor-atom preference of the copper(II)


 ion.Introduction. During the course of studies of the coordination chemistry of mixed donor atom medium-ring macrocyclic ligands, the 1-thia-4,7diazacyclononane derivative (1) was prepared. This ligand with five potential donors was complexed with copper(II) ions and the structure of the copper complex determined. Although there have been many structural determinations of transition-metal complexes with $9-\mathrm{N}_{3}, 9-\mathrm{S}_{3}$ and $9-\mathrm{N}_{2} \mathrm{~S}$ ligands, none has been reported for functionalized derivatives of the $9-\mathrm{N}_{2} \mathrm{~S}$ ring system (Boeyens \& Dobson, 1987). In the reported structures with 9 -ring macrocyclic ligands, the macrocyclic ring tends to adopt preferentially a low-energy [333] conformation (Dale, 1974; Boeyens \& Dobson, 1987).

(1)

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Experimental. The ligand was prepared by cyclization of ethylene glycol ditosylate with $N, N^{\prime}$-thiodiethylene bis(toluenesulfonamide) in dimethylformamide in the presence of caesium carbonate, followed by detosylation ( $\mathrm{HBr} / \mathrm{AcOH} / \mathrm{PhOH}$ ), alkylation ( $\mathrm{ClCH}_{2} \mathrm{CO}_{2} \mathrm{H} / \mathrm{LiOH}$ ) and esterification $\left(\mathrm{MeOH} / \mathrm{H}^{+}\right)$. Blue monoclinic crystals of the title complex were grown by slow evaporation of a methanol solution containing LiCl . Accurate cell dimensions and crystal orientation matrix were determined on a CAD-4 diffractometer by a leastsquares treatment of the setting angles of 25 reflections in the range $8<\theta<20^{\circ}$. Crystal dimensions


Fig. 1. ORTEP diagram (Johnson, 1976) with the atomnumbering scheme and torsional angles ( ${ }^{\circ}$ ) for the ninemembered ring. Non-H-atom ellipsoids are at the $50 \%$ probability level.
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Table 1. Fractional coordinates and isotropic thermal parameters with their e.s.d.'s in parentheses

|  | $\boldsymbol{x}$ | $y$ | $z$ | $B_{\text {eq }}{ }^{*}\left(\AA^{2}\right)$ |
| :---: | :---: | :---: | :---: | :---: |
| $\mathrm{Cu}(1)$ | $0 \cdot 17385$ (5) | $0.25 \dagger$ | -0.02073 (8) | 2.43 (1) |
| $\mathrm{Cl}(1)$ | $0 \cdot 1514$ (1) | $0-5201$ (2) | -0.0514 (2) | $4 \cdot 12$ (4) |
| S(1) | $0 \cdot 1100$ (1) | $0 \cdot 2223$ (2) | 0.2389 (2) | $3 \cdot 28$ (3) |
| C(2) | 0.2310 (5) | 0.2819 (10) | $0 \cdot 3684$ (8) | 4.0 (2) |
| C(3) | 0.3045 (5) | 0.3503 (10) | $0 \cdot 2806$ (8) | $3 \cdot 8$ (2) |
| N(4) | $0 \cdot 3115$ (3) | $0 \cdot 2582$ (8) | $0 \cdot 1388$ (5) | $2 \cdot 8$ (1) |
| C(5) | $0 \cdot 3433$ (5) | 0.0858 (9) | $0 \cdot 1806$ (8) | $3 \cdot 4$ (1) |
| C(6) | $0 \cdot 2923$ (5) | -0.0376 (9) | 0.0571 (8) | $3 \cdot 4$ (1) |
| N(7) | 0.1854 (3) | 0.0054 (7) | -0.0168 (5) | $2 \cdot 4$ (1) |
| C(8) | $0 \cdot 1150$ (5) | -0.0657 (8) | 0.0710 (8) | $3 \cdot 1$ (1) |
| C(9) | 0.1224 (5) | -0.0012 (10) | 0.2337 (8) | $3 \cdot 7$ (2) |
| $\mathrm{C}(10)$ | 0.3836 (6) | 0.3427 (11) | 0.0668 (9) | $4 \cdot 2$ (2) |
| $\mathrm{C}(11)$ | 0.3770 (5) | 0.2846 (9) | -0.0982 (8) | $3 \cdot 6$ (2) |
| $\mathrm{O}(12)$ | $0 \cdot 3010$ (3) | 0.2343 (8) | -0.1883 (5) | $3 \cdot 8$ (1) |
| $\mathrm{O}(13)$ | 0.4640 (3) | 0.3011 (8) | -0.1342 (5) | $4 \cdot 6$ (1) |
| C(14) | 0.4655 (6) | 0.2566 (16) | -0.2966 (9) | $5 \cdot 8$ (2) |
| C(15) | 0.1532 (5) | -0.0483 (9) | -0.1846 (7) | $3 \cdot 1$ (1) |
| C(16) | 0.0723 (5) | 0.0644 (8) | -0.2707 (7) | $2 \cdot 8$ (1) |
| $\mathrm{O}(17)$ | 0.0591 (3) | $0 \cdot 1998$ (6) | -0.2175 (5) | $3 \cdot 2$ (1) |
| $\mathrm{O}(18)$ | 0.0201 (4) | 0.0080 (7) | -0.4040 (5) | $3 \cdot 6$ (1) |
| C(19) | -0.0624 (6) | $0 \cdot 1139$ (11) | -0.4929 (10) | $4 \cdot 8$ (2) |
| $\mathrm{Cl}(2)$ | 0.6502 (1) | $0 \cdot 2494$ (3) | 0.4212 (2) | 4.57 (4) |
| $\mathrm{O}(21)$ | 0.7045 (7) | $0 \cdot 2200$ (14) | 0.5821 (11) | 11.1 (3) |
| $\mathrm{O}(22)$ | 0.5555 (5) | $0 \cdot 2312$ (27) | $0 \cdot 4115$ (9) | 18.4 (6) |
| $\mathrm{O}(23)$ | 0.6778 (13) | 0.4070 (17) | $0 \cdot 4077$ (14) | 17.7 (5) |
| $\mathrm{O}(24)$ | 0.6817 (10) | $0 \cdot 1704$ (23) | $0 \cdot 3202$ (13) | 12.9 (5) |
| $\begin{aligned} & { }^{*} B_{\mathrm{eq}}=\frac{4}{3}\left[a^{2} B_{11}+b^{2} B_{22}+c^{2} B_{33}+a b(\cos \gamma) B_{12}+a c(\cos \beta) B_{13}+\right. \\ & \left.b c(\cos \alpha) B_{23}\right] . \\ & \dagger \text { The } y \text { coordinate was fixed at } 0.25 \text { to define the origin. } \end{aligned}$ |  |  |  |  |
|  |  |  |  |  |

$0.15 \times 0.16 \times 0.56 \mathrm{~mm}$; intensities of reflections with indices $h-17$ to $17, k 0$ to $10, l 0$ to 11 , with $2<$ $2 \theta<54^{\circ}$ measured, $\omega-2 \theta$ scans; $\omega$ scan width ( $0.60+$ $0.35 \tan \theta)^{\circ}$, graphite-monochromatized Mo K $\alpha$ radiation. Intensities of three reflections were measured every two hours and showed no evidence of crystal decay. 2430 reflections were measured, 2228 were unique and 1757 with $I>3 \sigma(I)$ were labelled observed and used in structure solution and refinement; $R_{\text {int }}=0.026$. Data were corrected for Lorentz, polarization and absorption effects (max. and min. transmission factors $0.816,0.759$ ), Gaussian integration, grid $8 \times 10 \times 10$. The systematic absences ( $0 k 0$ absent if $k=2 n+1$ ) allow the space group to be either $P 2_{1}$ or $P 2_{1} / m$. With $Z=2$, the latter would require the molecule to have either mirror or inversion symmetry, and as this was unlikely, $P 2_{1}$ was chosen and confirmed by the analysis. The coordinates of the Cu atom were determined from an analysis of the three-dimensional Patterson function and the coordinates of the remaining non-H atoms found via the heavy-atom method. Refinement was by full-matrix least-squares calculations, initially with isotropic and then with anisotropic thermal parameters. At an intermediate stage of the refinement, difference maps showed maxima in positions consistent with the expected locations of the H atoms; in the final rounds of calculations the H atoms were positioned on geo-

Table 2. Bond distances $(\AA)$ and bond angles $\left({ }^{\circ}\right)$ with their e.s.d.'s in parentheses

| $\mathrm{Cu}(1)$ | $\mathrm{Cl}(1)$ | 2.241 |  | N(7) | C(15) | 1.482 (7) |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| $\mathrm{Cu}(1)$ | S(1) | 2.635 |  | C(8) | C(9) | 1.489 |  |
| $\mathrm{Cu}(1)$ | N(4) | 2.051 |  | C(10) | C(11) | 1.494 |  |
| $\mathrm{Cu}(1)$ | N(7) | 2.010 |  | C(11) | $\mathrm{O}(12)$ | $1 \cdot 21$ |  |
| $\mathrm{Cu}(1)$ | $\mathrm{O}(12)$ | 2.554 |  | C(11) | O(13) | $1 \cdot 323$ |  |
| $\mathrm{Cu}(1)$ | $\mathrm{O}(17)$ | 2.058 |  | O(13) | C(14) | 1.465 |  |
| S(1) | C(2) | 1.829 |  | C(15) | C(16) | 1.495 |  |
| S(1) | C(9) | 1.841 |  | C(16) | O(17) | 1.23 |  |
| C(2) | C(3) | 1.523 |  | C(16) | $\mathrm{O}(18)$ | 1.287 |  |
| C(3) | N(4) | 1.471 |  | O(18) | C(19) | 1.484 |  |
| N(4) | C(5) | 1.496 |  | $\mathrm{Cl}(2)$ | $\mathrm{O}(21)$ | 1.431 |  |
| N(4) | C(10) | 1.476 |  | $\mathrm{Cl}(2)$ | $\mathrm{O}(22)$ | 1.297 |  |
| C(5) | C(6) | 1.513 |  | $\mathrm{Cl}(2)$ | O(23) | 1.360 |  |
| C(6) | $\mathrm{N}(7)$ | 1.495 |  | $\mathrm{Cl}(2)$ | O (24) | $1 \cdot 255$ |  |
| $\mathrm{N}(7)$ | C(8) | 1.498 |  |  |  |  |  |
| $\mathrm{Cl}(1)$ | $\mathrm{Cu}(1)$ | S(1) | 96.94 (7) | C(5) | C(6) | N(7) | $112 \cdot 3$ (5) |
| $\mathrm{Cl}(1)$ | $\mathrm{Cu}(1)$ | $\mathrm{N}(4)$ | 97.2 (2) | $\mathrm{Cu}(1)$ | $\mathrm{N}(7)$ | C(6) | 107.9 (4) |
| $\mathrm{Cl}(1)$ | $\mathrm{Cu}(1)$ | $\mathrm{N}(7)$ | 174.0 (1) | $\mathrm{Cu}(1)$ | N(7) | C(8) | $109 \cdot 7$ (4) |
| $\mathrm{Cl}(1)$ | $\mathrm{Cu}(1)$ | $\mathrm{O}(12)$ | $94 \cdot 4$ (2) | $\mathrm{Cu}(1)$ | N(7) | C(15) | $106 \cdot 1$ (4) |
| $\mathrm{Cl}(1)$ | $\mathrm{Cu}(1)$ | $\mathrm{O}(17)$ | 92.5 (1) | C(6) | $\mathrm{N}(7)$ | C(8) | 112.7 (5) |
| S(1) | $\mathrm{Cu}(1)$ | N(4) | 82.9 (1) | C(6) | N(7) | C(15) | 111.7 (5) |
| S(1) | $\mathrm{Cu}(1)$ | $\mathrm{N}(7)$ | 86.6 (1) | C(8) | N(7) | C(15) | 108.4 (5) |
| S(1) | $\mathrm{Cu}(1)$ | $\mathrm{O}(12)$ | 155.9 (1) | N(7) | C(8) | C(9) | 116.7 (5) |
| S(1) | $\mathrm{Cu}(1)$ | O(17) | 110.0 (1) | S(1) | C(9) | C(8) | 113.1 (5) |
| N(4) | $\mathrm{Cu}(1)$ | $\mathrm{N}(7)$ | 88.0 (2) | N(4) | C(10) | C(11) | 112.0 (5) |
| N(4) | $\mathrm{Cu}(1)$ | $\mathrm{O}(12)$ | 74.6 (2) | C(10) | C(11) | $\mathrm{O}(12)$ | 124.2 (6) |
| N(4) | $\mathrm{Cu}(1)$ | $\mathrm{O}(17)$ | $162 \cdot 8$ (2) | C(10) | C(11) | O(13) | 110.9 (6) |
| N(7) | $\mathrm{Cu}(1)$ | $\mathrm{O}(12)$ | $84 \cdot 1$ (2) | O(12) | C(11) | $\mathrm{O}(13)$ | $124 \cdot 8$ (6) |
| $\mathrm{N}(7)$ | $\mathrm{Cu}(1)$ | O(17) | 81.7 (2) | $\mathrm{Cu}(1)$ | $\mathrm{O}(12)$ | C(11) | $102 \cdot 8$ (4) |
| $\mathrm{O}(12)$ | $\mathrm{Cu}(1)$ | $\mathrm{O}(17)$ | $90 \cdot 6$ (2) | C(11) | $\mathrm{O}(13)$ | C(14) | 116.0 (5) |
| $\mathrm{Cu}(1)$ | S(1) | C(2) | $93 \cdot 1$ (2) | N(7) | C(15) | C(16) | 108.0 (5) |
| $\mathrm{Cu}(1)$ | S(1) | C(9) | 90.6 (2) | C(15) | C(16) | $\mathrm{O}(17)$ | 122.0 (5) |
| C(2) | S(1) | C(9) | 101.8 (3) | C(15) | C(16) | $\mathrm{O}(18)$ | 114.0 (5) |
| S(1) | C(2) | C(3) | 114.2 (5) | O(17) | C(16) | $\mathrm{O}(18)$ | 124.0 (6) |
| C(2) | C(3) | N(4) | 115.2 (6) | $\mathrm{Cu}(1)$ | $\mathrm{O}(17)$ | C(16) | 109.6 (4) |
| $\mathrm{Cu}(1)$ | N(4) | C(3) | 109.7 (4) | C(16) | O(18) | C(19) | 116.1 (5) |
| $\mathrm{Cu}(1)$ | $\mathrm{N}(4)$ | C(5) | 107.4 (4) | O(21) | $\mathrm{Cl}(2)$ | $\mathrm{O}(22)$ | 108.3 (6) |
| $\mathrm{Cu}(1)$ | $\mathrm{N}(4)$ | C(10) | 109.7 (4) | O(21) | $\mathrm{Cl}(2)$ | O(23) | 99.0 (7) |
| C(3) | N(4) | C(5) | 111.0 (5) | O(21) | $\mathrm{Cl}(2)$ | O(24) | 114. (1) |
| C(3) | N(4) | C(10) | 108.1 (6) | O(22) | $\mathrm{Cl}(2)$ | O(23) | 114. (1) |
| C(5) | N(4) | C(10) | 111.0 (5) | O (22) | $\mathrm{Cl}(2)$ | $\mathrm{O}(24)$ | 115.(1) |
| N(4) | C(5) | C(6) | 113.8 (5) | O(23) | $\mathrm{Cl}(2)$ | $\mathrm{O}(24)$ | 106. (1) |

metrical grounds ( $\mathrm{C}-\mathrm{H} 0.95 \AA$ ) and included (as riding atoms) in the structure-factor calculations with an overall $B_{\text {iso }}$ of $5 \cdot 0 \AA^{2}$. The final cycle of refinement on $F$ included 234 variable parameters, $R$ $=0.043, \quad w R=0.054$, goodness-of-fit 1.37, $w=$ $1 /\left[\sigma^{2}\left(F_{o}\right)+0.040\left(F_{o}\right)^{2}\right] . \quad$ Max. $\quad$ shift/e.s.d. $=0 \cdot 11$; extreme values in final difference map $\pm 0.95 \mathrm{e} \AA^{-3}$ adjacent to the markedly anisotropic perchlorate counterion. As the copper complex of (1) is chiral, refinement calculations were performed with the opposite hand. The model corresponding to the absolute stereochemistry indicated had $R$ factors that were lower than that of the enantiomer ( $R=0.053$, $w R=0.065$ ). The coordinates and molecular diagram refer to this absolute stereochemistry. Scattering factors and anomalous-dispersion corrections were taken from International Tables for X-ray Crystallography (1974). All calculations were performed on a PDP11/73 computer using SDP-Plus (B. A. Frenz \& Associates, Inc., 1983). Atomic coordinates and details of molecular geometry are given in

Tables 1 and 2.* Fig. 1, prepared using ORTEPII (Johnson, 1976), shows details of the cation structure and copper coordination.

Discussion. The copper coordination geometry is distorted octahedral with longer bonds to the axial $\mathrm{O}(12)$ and $\mathrm{S}(1)$ atoms. The macrocyclic ring adopts a rather strained [333] conformation (Dale, 1973, 1974; Boeyens \& Dobson, 1987) as evidenced in the 'nonideal' torsional angles about C(6) (Fig. 1). The alternative [234] conformation is energetically less stable by about $12-20 \mathrm{~kJ} \mathrm{~mol}^{-1}$ (Dobson, 1986) but may be preferred by those metal ions which do not accept facial symmetrical coordination - such as $\mathrm{Cu}^{\mathrm{II}}$ and $\mathrm{Pt}^{\mathrm{II}}$. Related macrocyclic $\mathrm{Cu}^{\mathrm{II}}$ complexes with the unfunctionalized nine-membered $\mathrm{N}_{2} \mathrm{~S}$ ligand have been reported (Dobson, 1986; Boeyens, Dobson \& Hancock, 1985) in both of which the macrocycle adopts a [234] conformation. This [234] con-

[^0]formation is common for mixed-donor ninemembered macrocyclic ligands that may interact asymmetrically with metal ions that cannot accept symmetrical facial coordination.

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# Structures of trans-Cyanobis(dimethylglyoximato)pyridinecobalt(III) Hemihydrate and trans-Cyclohexylbis(dimethylglyoximato)(1,5,6-trimethylbenzimidazole)cobalt(III) 

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#### Abstract

I) $\left[\mathrm{Co}(\mathrm{CN})\left(\mathrm{C}_{4} \mathrm{H}_{7} \mathrm{~N}_{2} \mathrm{O}_{2}\right)_{2}\left(\mathrm{C}_{5} \mathrm{H}_{5} \mathrm{~N}\right)\right] \cdot 0 \cdot 5 \mathrm{H}_{2} \mathrm{O}$, $M_{r}=403 \cdot 3$, monoclinic, $P 2_{1} / m, a=7 \cdot 564$ (1), $b=$ $13.950(1), \quad c=8.653(1) \AA, \quad \beta=98.20(1)^{\circ}, \quad V=$ 903.7 (3) $\AA^{3}, \quad Z=2, \quad D_{x}=1.48 \mathrm{~g} \mathrm{~cm}^{-3}, \quad \lambda($ Mo $K \alpha)$ $=0.7107 \AA, \quad \mu=9.2 \mathrm{~cm}^{-1}, \quad F(000)=418, \quad T=$ 295 (1) K, $R=0.027$ for 1815 unique observed

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reflections. (II) $\left[\mathrm{Co}\left(\mathrm{C}_{6} \mathrm{H}_{11}\right)\left(\mathrm{C}_{4} \mathrm{H}_{7} \mathrm{~N}_{2} \mathrm{O}_{2}\right)_{2}\left(\mathrm{C}_{10} \mathrm{H}_{12} \mathrm{~N}_{2}\right)\right]$, $M_{r}=532 \cdot 5$, monoclinic, $P 2_{1} / n, a=17 \cdot 133$ (2), $b=$ 8.566 (1),$\quad c=19.369$ (3) $\AA, \quad \beta=115.36$ (3) ${ }^{\circ}, \quad V=$ 2568.6 (9) $\AA^{3}, Z=4, \quad D_{x}=1.37 \mathrm{~g} \mathrm{~cm}^{-3}, \quad \lambda($ Mo $K \alpha)$ $=0.7107 \AA, \quad \mu=7.0 \mathrm{~cm}^{-1}, \quad F(000)=1128, \quad T=$ 295 (1) K, $R=0.044$ for 3290 unique observed reflections. The axial fragment of the distorted octahedral polyhedron of Co is characterized by $\mathrm{Co}-\mathrm{C}$ and $\mathrm{Co}-\mathrm{N}$ distances of 1.995 (2) and 1.937 (2) $\AA$ in © 1989 International Union of Crystallography


[^0]:    * Lists of structure factors, thermal parameters, calculated H-atom coordinates and all torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51978 ( 22 pp .). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

