Refinement

Refinement on F
$R = 0.037$
$wR = 0.044$
$S = 1.009$
2010 reflections
125 parameters
H-atom parameters not refined

$w = 1/[\sigma^2(F_o) + 0.002777(F_o^2)]$
$(\Delta/\sigma)_{\max} = 0.01$
$\Delta\rho_{\max} = 0.32$ e Å^{-3}
$\Delta\rho_{\min} = -0.52$ e Å^{-3}
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)

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

	x	y	z	$U_{\rm eq}$
Cr	0.29760 (3)	−0.01745 (4)	0.09236 (3)	0.0330 (1)
C(1)	0.2443 (2)	−0.2647 (3)	0.1510 (2)	0.0465 (7)
C(2)	0.3554 (2)	−0.2746 (3)	0.1325 (3)	0.0553 (9)
C(3)	0.4219 (2)	−0.1592 (4)	0.2163 (3)	0.0620 (9)
C(4)	0.3518 (2)	−0.0781 (4)	0.2859 (2)	0.0551 (8)
C(5)	0.2422 (2)	−0.1434 (3)	0.2461 (2)	0.0447 (7)
C(6)	0.1433 (3)	−0.0932 (5)	0.2960 (3)	0.072 (1)
C(7)	0.3150 (2)	0.0589 (3)	−0.0864 (2)	0.0475 (8)
C(8)	0.2001 (2)	0.0749 (3)	−0.0819 (2)	0.0471 (7)
C(9)	0.3763 (2)	0.1732 (3)	0.0034 (2)	0.0470 (7)
C(10)	0.2994 (2)	0.2584 (3)	0.0616 (2)	0.0458 (7)
C(11)	0.1904 (2)	0.1979 (3)	0.0085 (2)	0.0452 (7)
C(12)	0.0850 (2)	0.2563 (4)	0.0444 (3)	0.070 (1)

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

Cp(1) and *Cp*(2) are the centroids of the cyclopentadienyl rings.

Cr—C(1)	2.167 (2)	Cr—C(2)	2.124 (3)
Cr—C(3)	2.125 (3)	Cr—C(4)	2.167 (2)
Cr—C(5)	2.206 (3)	Cr—C(7)	2.135 (3)
Cr—C(8)	2.168 (2)	Cr—C(9)	2.125 (3)
Cr—C(10)	2.160 (2)	Cr—C(11)	2.199 (2)
Cr—*Cp*(1)	1.788 (2)	Cr—*Cp*(2)	1.788 (2)
C(1)—C(2)	1.426 (4)	C(1)—C(5)	1.420 (3)
C(2)—C(3)	1.414 (4)	C(3)—C(4)	1.426 (4)
C(4)—C(5)	1.414 (4)	C(5)—C(6)	1.494 (5)
C(7)—C(8)	1.428 (4)	C(7)—C(9)	1.421 (3)
C(8)—C(11)	1.410 (3)	C(9)—C(10)	1.421 (4)
C(10)—C(11)	1.419 (3)	C(11)—C(12)	1.507 (4)
C(2)—C(1)—C(5)	108.6 (2)	C(1)—C(2)—C(3)	107.6 (2)
C(2)—C(3)—C(4)	107.9 (3)	C(3)—C(4)—C(5)	108.6 (2)
C(1)—C(5)—C(4)	107.3 (3)	C(8)—C(7)—C(9)	107.2 (2)
C(7)—C(8)—C(11)	108.8 (2)	C(7)—C(9)—C(10)	107.9 (2)
C(9)—C(10)—C(11)	108.5 (2)	C(8)—C(11)—C(10)	107.5 (2)
C(4)—C(5)—C(6)	125.8 (2)	C(1)—C(5)—C(6)	126.8 (2)
C(10)—C(11)—C(12)	125.3 (2)	C(8)—C(11)—C(12)	127.2 (2)
Cp(1)—Cr—*Cp*(2)	178.1 (1)		

An air-sensitive crystal was lodged in a Lindemann glass capillary and centred on the Philips PW1100 diffractometer. The orientation matrix and preliminary unit-cell dimensions were determined from reflections found by mounting the crystal at random, and varying each of the orientation angles χ and φ over a range of 120°, with $7 < \theta < 10°$. There were no significant fluctuations of intensities other than those expected from Poisson statistics. Intensity data were corrected for Lp effects. The structure was solved by Patterson and Fourier techniques and refined by full-matrix least squares; anisotropic displacement parameters were assigned to all non-H atoms. H atoms were introduced at calculated positions and allowed to ride on the parent C atoms during the least-squares refinement (C—H = 0.98 Å and $U = 0.07$ Å^2). Data processing and computations were carried out using *SHELX76* (Sheldrick, 1976). Molecular graphics were obtained using *ORTEPII* (Johnson, 1976).

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71837 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: NA1054]

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(4,7-Dimethyl-4,7-diazadecanediamide)-diisothiocyanatocopper(II)

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Abstract

The Cu$^{\rm II}$ ion of the title complex, [Cu(NCS)$_2$(C$_{10}$H$_{22}$O$_2$N$_4$)], is six-coordinated in a tetragonally elongated octahedral geometry with the two amine N atoms of the quadridentate ligand and the two N atoms of the isothiocyanato groups equatorial and the two amide O atoms axial. The two *N*-methyl groups of the tetradentate ligand are *trans* to each other with respect to the equatorial plane. The complex has a *cis-α* conformation. The central five-membered ring is in a stable skew form and both of the terminal

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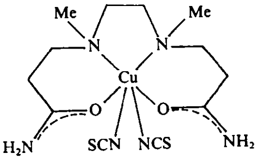
six-membered rings are in unstable twist forms. Both isothiocyanato groups are disordered.

Comment

Metal–thiocyanato complexes have been studied extensively because the coordination behaviour of the ambidentate ligand, the thiocyanate ion, involves many dramatically different bonding modes (Burmeister, 1990; Ferrer *et al.*, 1992). In order to expand the knowledge of this area, we report the crystal structure of the title complex (I).



(I)

The ligand 4,7-dimethyl-4,7-diazadecanediamide was prepared by a previously described procedure (Lu, Shan, Chao & Chung, 1987). The green complex was prepared by the literature method using this ligand instead of triethylenetetraamine (Barbucci, Cialdi, Ponticelli & Paoletti, 1969).

The crystal consists of discrete mononuclear copper(II) units linked by hydrogen bonds. As shown in Fig. 1, the Cu^{II} ion is six-coordinated in a tetragonally elongated octahedral geometry with the two amine N atoms of the tetradentate ligand and the two N atoms of the isothiocyanato groups in equatorial positions and the two amide O atoms in axial positions. The four equatorial atoms form a plane with a maximum deviation of 0.089 (4) Å. The Cu^{II} ion is displaced 0.005 (1) Å out of the equatorial plane towards O(2). The two Cu—N(amine) distances are slightly longer than the two Cu—N(isothiocyanato) distances. The two *N*-methyl groups of the tetradentate ligand are *trans* to each other with respect to the equatorial plane. The complex has a *cis-α* conformation. The central five-membered ring is in a stable *gauche* form and both terminal six-membered rings are in unstable twist forms. The two isothiocyanato groups are disordered.

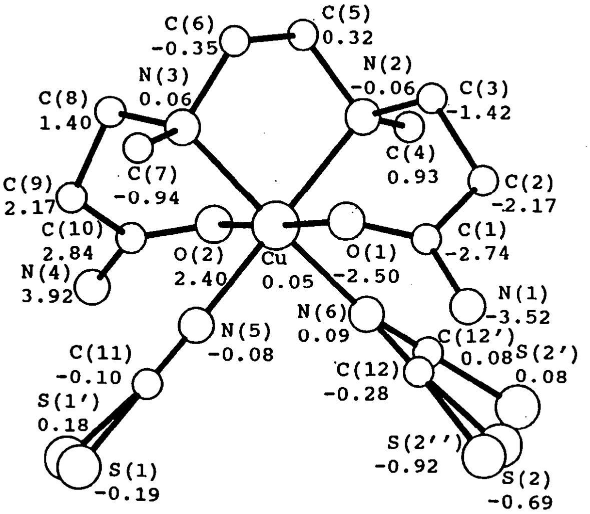


Fig. 1. A perspective view with the atom-numbering scheme of the title molecule, excluding the H atoms. The displacements from the equatorial N_4 plane are indicated (e.s.d.'s within 0.01 Å).

Experimental

Crystal data

$[Cu(NCS)_2(C_{10}H_{22}O_2N_4)]$
$M_r = 410.01$
Monoclinic
$C2/c$
$a = 16.980$ (5) Å
$b = 15.160$ (4) Å
$c = 14.30$ (1) Å
$\beta = 93.48$ (4)°
$V = 3674$ (3) Å^3
$Z = 8$

$D_x = 1.482$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 24 reflections
$\theta = 7.25$–16.2°
$\mu = 1.42$ mm^{-1}
$T = 298$ (3) K
Parallelepiped
0.53 × 0.47 × 0.44 mm
Blue

Data collection

Enraf-Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: empirical (North, Phillips & Mathews, 1968)
$T_{\min} = 0.835$, $T_{\max} = 1.000$
3337 measured reflections
3224 independent reflections

2594 observed reflections [$I > 2.5\sigma(I)$]
$R_{\rm int} = 0.021$
$\theta_{\max} = 24.9$°
$h = -20 \rightarrow 20$
$k = 0 \rightarrow 18$
$l = 0 \rightarrow 16$
3 standard reflections
frequency: 60 min
intensity variation: ±1.0%

Refinement

Refinement on F
$R = 0.035$
$wR = 0.056$
$S = 1.09$
2594 reflections
267 parameters
Only H-atom U's refined
$w = 1/[\sigma^2(F_o) + 0.002|F_o|^2]$
$(\Delta/\sigma)_{\max} = 0.032$

$\Delta\rho_{\max} = 0.60$ (7) e Å^{-3}
$\Delta\rho_{\min} = -0.59$ (7) e Å^{-3}
Extinction correction: secondary
Extinction coefficient: 0.42 (6) mm
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)

$$B_{\rm eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$
Cu	0.250112 (7)	0.421947 (21)	0.188198 (22)	2.515 (13)
S(1)	0.05408 (6)	0.62409 (7)	0.06602 (8)	4.36 (4)
S(1′)	0.04935 (21)	0.6235 (3)	0.0926 (3)	3.97 (17)
S(2)	0.43879 (12)	0.62414 (14)	0.09319 (19)	7.29 (11)
S(2′)	0.46931 (25)	0.6074 (3)	0.1605 (5)	8.3 (3)
S(2′′)	0.4191 (4)	0.6277 (4)	0.0709 (4)	7.0 (3)

O(1)	0.27183 (11)	0.36472 (15)	0.02334 (15)	3.57 (9)
O(2)	0.23308 (11)	0.46996 (17)	0.34209 (16)	3.98 (9)
N(1)	0.36420 (16)	0.42941 (18)	−0.05929 (21)	3.91 (11)
N(2)	0.33394 (14)	0.32598 (16)	0.22833 (16)	2.99 (9)
N(3)	0.16663 (13)	0.32497 (15)	0.20953 (16)	2.94 (9)
N(4)	0.14399 (15)	0.55346 (19)	0.41008 (20)	3.94 (11)
N(5)	0.16956 (14)	0.50539 (17)	0.13551 (19)	3.58 (11)
N(6)	0.33237 (14)	0.51144 (17)	0.17289 (20)	3.75 (11)
C(1)	0.34168 (16)	0.38159 (19)	0.01128 (20)	2.97 (11)
C(2)	0.40668 (16)	0.34532 (19)	0.07769 (21)	3.06 (11)
C(3)	0.37540 (18)	0.28303 (20)	0.14991 (22)	3.43 (12)
C(4)	0.39381 (18)	0.36101 (24)	0.29873 (23)	4.05 (13)
C(5)	0.28763 (20)	0.25550 (21)	0.27374 (24)	3.98 (14)
C(6)	0.21157 (19)	0.24072 (20)	0.21664 (24)	3.72 (14)
C(7)	0.10714 (17)	0.31764 (22)	0.12839 (23)	3.71 (12)
C(8)	0.12357 (18)	0.33585 (22)	0.29751 (23)	3.83 (13)
C(9)	0.09762 (17)	0.42938 (22)	0.31660 (22)	3.77 (13)
C(10)	0.16309 (16)	0.48617 (21)	0.35703 (19)	3.14 (12)
C(11)	0.12152 (17)	0.55491 (20)	0.10840 (21)	3.12 (11)
C(12)	0.3726 (3)	0.5599 (3)	0.1354 (3)	3.93 (19)
C(12′)	0.3881 (5)	0.5509 (6)	0.1673 (8)	2.3 (4)

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

Cu—O(1)	2.560 (3)	N(2)—C(4)	1.485 (4)
Cu—O(2)	2.352 (3)	N(2)—C(5)	1.498 (4)
Cu—N(2)	2.091 (2)	N(3)—C(6)	1.488 (4)
Cu—N(3)	2.078 (2)	N(3)—C(7)	1.496 (4)
Cu—N(5)	1.978 (3)	N(3)—C(8)	1.502 (4)
Cu—N(6)	1.969 (3)	N(4)—C(10)	1.323 (4)
S(1)—C(11)	1.641 (3)	N(5)—C(11)	1.158 (4)
S(1′)—C(11)	1.612 (5)	N(6)—C(12)	1.156 (5)
S(2)—C(12)	1.631 (5)	N(6)—C(12′)	1.13 (1)
S(2′)—C(12′)	1.63 (1)	C(1)—C(2)	1.517 (4)
S(2″)—C(12)	1.620 (6)	C(2)—C(3)	1.520 (4)
O(1)—C(1)	1.236 (3)	C(5)—C(6)	1.502 (5)
O(2)—C(10)	1.245 (3)	C(8)—C(9)	1.515 (5)
N(1)—C(1)	1.318 (4)	C(9)—C(10)	1.494 (4)
N(2)—C(3)	1.508 (4)		
O(1)—Cu—O(2)	177.79 (8)	Cu—N(3)—C(8)	114.6 (2)
O(1)—Cu—N(2)	83.29 (9)	C(6)—N(3)—C(7)	108.0 (2)
O(1)—Cu—N(3)	91.89 (9)	C(6)—N(3)—C(8)	108.1 (2)
O(1)—Cu—N(5)	90.1 (1)	C(7)—N(3)—C(8)	108.5 (2)
O(1)—Cu—N(6)	89.3 (1)	Cu—N(5)—C(11)	177.2 (3)
O(2)—Cu—N(2)	94.52 (9)	Cu—N(6)—C(12)	158.7 (3)
O(2)—Cu—N(3)	87.59 (9)	Cu—N(6)—C(12′)	168.1 (5)
O(2)—Cu—N(5)	92.1 (1)	O(1)—C(1)—N(1)	123.1 (3)
O(2)—Cu—N(6)	91.1 (1)	O(1)—C(1)—C(2)	120.5 (3)
N(2)—Cu—N(3)	85.78 (9)	N(1)—C(1)—C(2)	116.4 (3)
N(2)—Cu—N(5)	173.1 (1)	C(1)—C(2)—C(3)	112.1 (2)
N(2)—Cu—N(6)	92.0 (1)	N(2)—C(3)—C(2)	115.9 (2)
N(3)—Cu—N(5)	92.7 (1)	N(2)—C(5)—C(6)	109.1 (2)
N(3)—Cu—N(6)	177.3 (1)	N(3)—C(6)—C(5)	109.2 (3)
N(5)—Cu—N(6)	89.8 (1)	N(3)—C(8)—C(9)	114.5 (3)
Cu—O(1)—C(1)	104.6 (2)	C(8)—C(9)—C(10)	113.0 (3)
Cu—O(2)—C(10)	113.2 (2)	O(2)—C(10)—N(4)	121.4 (3)
Cu—N(2)—C(3)	115.9 (2)	O(2)—C(10)—C(9)	121.0 (3)
Cu—N(2)—C(4)	111.3 (2)	N(4)—C(10)—C(9)	117.6 (3)
Cu—N(2)—C(5)	104.4 (2)	S(1)—C(11)—N(5)	177.9 (3)
C(3)—N(2)—C(4)	109.1 (2)	S(1′)—C(11)—N(5)	168.2 (3)
C(3)—N(2)—C(5)	107.2 (2)	S(2)—C(12)—N(6)	172.2 (5)
C(4)—N(2)—C(5)	108.5 (2)	S(2″)—C(12)—N(6)	171.8 (5)
Cu—N(3)—C(6)	105.4 (2)	S(2′)—C(12′)—N(6)	179 (1)
Cu—N(3)—C(7)	111.9 (2)		

The structure was solved by direct and Fourier methods, and refined by full-matrix least squares. H atoms were solved by difference Fourier methods. Program used: *NRCVAX* (Gabe, Le Page, White & Lee, 1987).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and possible hydrogen bonds have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71842 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS1076]

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Acta Cryst. (1994). C**50**, 1005–1007

Bis(μ-4-methylphenoxido-1:2κ^2O)tris(triphenylphosphine)-1κ^2P,2κP-dicopper(I)

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Abstract

$(Ph_3P)_2Cu(\mu\text{-}OC_6H_4Me\text{-}4)_2Cu(PPh_3)$ has one Cu atom, Cu(1), in a distorted tetrahedral coordination environment and the other, Cu(2), in a distorted trigonal planar environment. The P(1)—Cu(1)—P(2) angle [127.0 (1)°] involving the phosphines is significantly larger than the O(1)—Cu(1)—O(2) angle [79.3 (2)°] involving the bridging 4-methylphenoxido ligands. The P—Cu(1)—O angles fall within the range 106.6 (2)–112.9 (2)°. The dihedral angle between the plane defined by the Cu atom and its two coordinated P atoms and that defined by the Cu atom and the two O atoms is 89.2°. The sum of the three *L*—Cu—*L* angles around Cu(2) is 360.0°. However, the triangular environment around Cu(2) is not symmetric: O(2)—Cu(2)—P(3) 142.9 (2), O(1)—Cu(2)—P(3) 132.0 (2) and O(1)—Cu(2)—O(2) 85.0 (2)°.