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[(4*RS*,7*SR*)-1-(1*H*-Benzimidazol-2-ylmethyl-κN^3)-1,4,7-triazacyclononane-$\kappa^3 N$]bromidocopper(II) bromide

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In the title complex, [CuBr($C_{14}H_{21}N_5$)]Br, the Cu atom is located at the center of a distorted square pyramid with three N and one Br atoms in the square plane and one N atom at the apex. The Cu atom is displaced by 0.2430 (2) Å from the square plane.

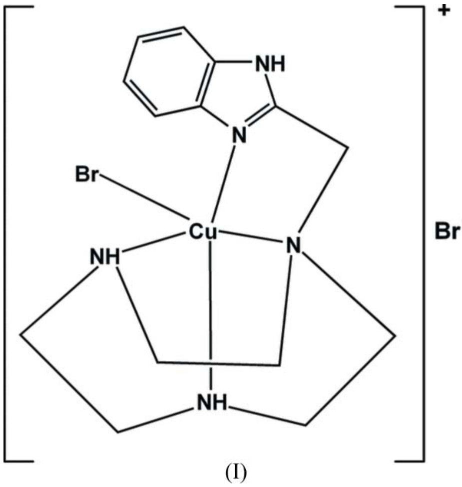
Key indicators

Single-crystal X-ray study
T = 297 K
Mean σ(C–C) = 0.007 Å
R factor = 0.035
wR factor = 0.080
Data-to-parameter ratio = 19.1

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

Comment

N-Functionalized 1,4,7-triazacyclononanes (tacn) have attracted attention as they are versatile and efficient ligands. Some metal complexes of such ligands have potential applications including the modeling of enzymes (Wainwright *et al.*, 1997), as radiotherapeutic agents and time-resolved luminescence labels (Charbonnieŕe *et al.*, 2001). Derivatives of tacn with pendant pyridyls (Tamura *et al.*, 2000), anilines (Fallis *et al.*, 2000), imidazoles, pyrazoles (Vaira *et al.*, 2000) *etc.* have been reported. Our group has reported the structure of a copper(II) complex of tacn with a benzimidazolyl ligand, [1-(benzimidazol-2-ylmethyl)-1,4,7-triazacyclononane]chloridocopper(II) perchlorate (Li *et al.*, 2003). Here we report the structure of a new complex (I).



(I)

In this complex, the Cu atom is located at the center of a distorted square pyramid with N2 at the apex and N1, N3, N4, Br1 in the square plane. The Cu atom is displaced 0.2430 (2) Å from the least-squares plane composed of N1, N3, N4 and Br1, the largest deviation from the plane being 0.0536 (4) Å for N1. The N1—Cu1—N2, N3—Cu1—N2, N4—Cu1—N2 and Br1—Cu1—N2 angles are 83.24 (14), 82.83 (16), 104.71 (15) and 110.49 (11)° respectively. At 1.973 (4) Å, Cu—N4 is the shortest of the Cu—N bonds. It indicates that the N atom of the benzimidazole group has a stronger coordination affinity for Cu(II) than those of triazacyclononane. The chiral atoms

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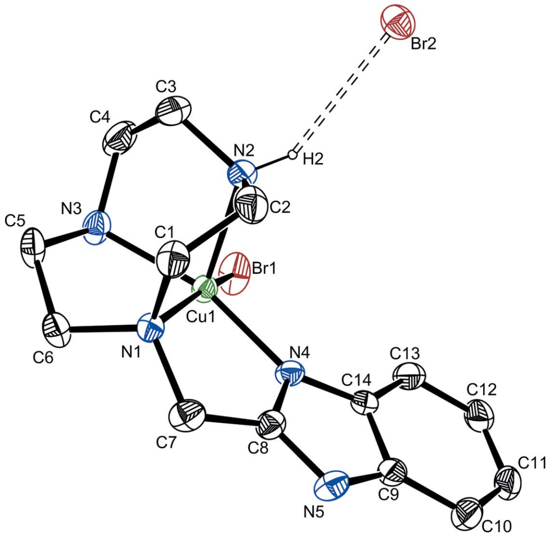

Figure 1
The molecular structure of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms not involved in the hydrogen-bonding interaction (double dashed line) have been omitted for the sake of clarity.

N2 and N3 possess different configurations; as the space group possesses glide planes the two diastereoisomers *R*,*S* and *R*,*S* are present in the cell. There are three weak N—H···Br hydrogen interactions which stabilize the structure (Table 1).

Experimental

The ligand 1-(benzimidazolyl-2-methyl)-1,4,7-triazacyclononane was prepared by modified literature methods (Li *et al.*, 2005). Ligand 1 (0.50 mmol) was added to a methanol solution (25 ml) of $CuBr_2$ (0.50 mmol) with stirring at reflux temperature. After the mixture had been stirred continuously for 2 h, it was cooled and filtered. Evaporation of the solution give a blue crystalline compound. Crystals suitable for X-ray analysis were obtained by diffusion of diethyl ether into an MeCN solution after three days.

Crystal data

$[CuBr(C_{14}H_{21}N_5)]Br$
$M_r = 482.72$
Orthorhombic, $Pna2_1$
$a = 19.525$ (2) Å
$b = 7.9390$ (9) Å
$c = 11.2702$ (12) Å
$V = 1747.0$ (3) $Å^3$
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 5.83$ mm^{-1}
$T = 297$ (2) K
0.20 × 0.10 × 0.10 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
$T_{min} = 0.389$, $T_{max} = 0.593$ (expected range = 0.366–0.558)
12313 measured reflections
3796 independent reflections
3268 reflections with $I > 2\sigma(I)$
$R_{int} = 0.036$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.080$
$S = 1.00$
3796 reflections
199 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max} = 0.74$ e $Å^{-3}$
$\Delta\rho_{min} = -0.28$ e $Å^{-3}$
Absolute structure: Flack (1983), 1789 Friedel pairs
Flack parameter: 0.017 (12)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···Br2	0.91	2.64	3.395 (4)	141
N3—H3···Br2^{i}	0.91	2.64	3.456 (4)	149
N5—H5···Br2^{ii}	0.86	2.58	3.269 (3)	138

Symmetry codes: (i) $-x+\frac{1}{2}, y-\frac{1}{2}, z-\frac{1}{2}$; (ii) $x-\frac{1}{2}, -y+\frac{1}{2}, z$.

All H atoms attached to C atoms and N atom were positioned geometrically and treated as riding, with C—H = 0.93 Å (aromatic), 0.97 Å (methylene) and N—H = 0.91 Å (sp^3) or 0.86 Å (sp^2), and with $U_{iso}(H) = 1.2U_{eq}(C,N)$.

Data collection: *SMART* (Bruker, 2001; cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *PLATON* (Spek, 2003).

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