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Key indicators

Single-crystal X-ray study
$T = 293$ K
Mean σ(C–C) = 0.004 Å
R factor = 0.029
wR factor = 0.079
Data-to-parameter ratio = 14.2

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

Diazido(1,4,7-tribenzyl-1,4,7-triazacyclo-nonane-κ^3N)copper(II)

In the title compound, [Cu(N$_3$)$_2$(C$_{27}$H$_{33}$N$_3$)], the copper(II) atom is coordinated by five N atoms from the 1,4,7-tribenzyl-1,4,7-triazacyclononane ligand and the azide ions in a slightly distorted square-pyramidal geometry.

Comment

1,4,7-Triazacyclononane and its derivatives have attracted a great deal of attention in recent years due to their biological activities and their strong coordination abilities as multi-dentate ligands (Daly & Martin, 2002). One of the derivatives, 1,4,7-tribenzyl-1,4,7-triazacyclononane (L), was synthesized by Thomas *et al.* (1990). We report here a copper(II) complex with L, namely Cu(L)(N$_3$)$_2$, (I).

(I)
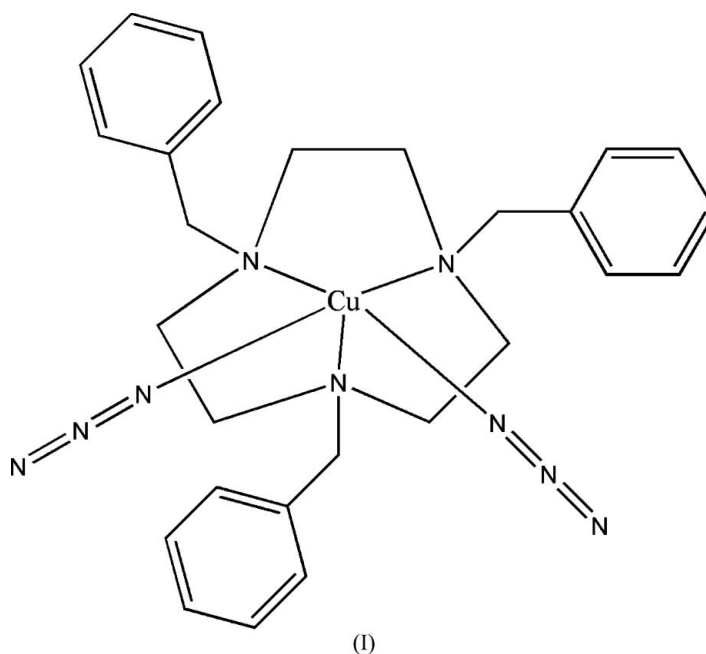
As shown in Fig. 1, the Cu atom adopts a slightly distorted square-pyramidal geometry. The basal plane is composed of atoms N1, N3, N4, N7 and atom N2 atom occupies the apical position. The Cu1 atom is displaced out of the basal plane by 0.105 L ligand are longer than those to the azide ions (Table 1). The value of τ as defined by Addison et al. (1984) is 0.134 [$\tau = (\beta-\alpha)/60 = 0$ and 1 for perfectly square-pyramidal and trigonal-bipyramidal geometries, respectively]; $\beta = $ N7—Cu1—N1 = 176.47 (7) and $\alpha = $ N4—Cu1—N3 = 168.40 (8), indicating a slight distortion from a square-pyramidal geometry. The azide ions are almost linear.

metal-organic papers

Experimental

A solution of $Cu(ClO_4)_2$ (0.047 g, 0.1 mmol) in CH_3CN (5 ml) was added to a solution of *L* (0.040 g, 0.1 mmol) in CH_3CN (15 ml). The reaction mixture was stirred at room temperature for 30 min to afford a clear deep-blue solution. A solution of NaN_3 (0.013 g, 0.2 mmol) in water (5 ml) was then slowly added dropwise. The mixture was continuously stirred for 2 h and filtered to remove any insoluble particles. Deep blue crystals suitable for X-ray analysis were obtained by slow evaporation of the filtrate. Analysis, calculated for $C_{27}H_{33}CuN_9$: C 59.27, H 6.08, N 23.04%; found: C 59.52, H 6.25, N 22.96%. IR (KBr, cm^{-1}): 3421 (*m*), 2053 (*s*), 1636 (*w*), 1355 (*w*).

Crystal data

$[Cu(N_3)_2(C_{27}H_{33}N_3)]$	$Z = 4$
$M_r = 547.16$	$D_x = 1.348$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
$a = 14.105\ (7)$ Å	$\mu = 0.84$ mm^{-1}
$b = 8.999\ (5)$ Å	$T = 293\ (2)$ K
$c = 21.603\ (11)$ Å	Block, blue
$\beta = 100.470\ (7)°$	$0.28 \times 0.22 \times 0.20$ mm
$V = 2696\ (2)$ Å^3	

Data collection

Bruker SMART APEX area-detector diffractometer	14088 measured reflections
φ and ω scan	4753 independent reflections
Absorption correction: multi-scan	3883 reflections with $I > 2\sigma(I)$
SADABS (Sheldrick, 1996)	$R_{int} = 0.023$
$T_{min} = 0.728$, $T_{max} = 0.845$	$\theta_{max} = 25.0°$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0434P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.029$	$+ 0.5508P]$
$wR(F^2) = 0.079$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{max} = 0.002$
4753 reflections	$\Delta\rho_{max} = 0.37$ e Å^{-3}
334 parameters	$\Delta\rho_{min} = -0.23$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Cu1—N4	1.9810 (19)	Cu1—N3	2.1165 (16)
Cu1—N7	1.9844 (19)	Cu1—N2	2.2490 (18)
Cu1—N1	2.1045 (17)		
N4—Cu1—N7	93.18 (9)	N4—Cu1—N2	106.05 (8)
N4—Cu1—N1	89.63 (8)	N7—Cu1—N2	97.56 (8)
N7—Cu1—N1	176.47 (7)	N1—Cu1—N2	83.69 (6)
N4—Cu1—N3	168.40 (8)	N3—Cu1—N2	83.02 (6)
N7—Cu1—N3	92.75 (7)	N6—N5—N4	177.0 (2)
N1—Cu1—N3	84.11 (7)	N7—N8—N9	174.6 (2)

H atoms were positioned geometrically and refined as riding with C—H = 0.93 Å (CH) and 0.97 Å (CH$_2$) and U_{iso}(H) = 1.2U_{eq}(C).
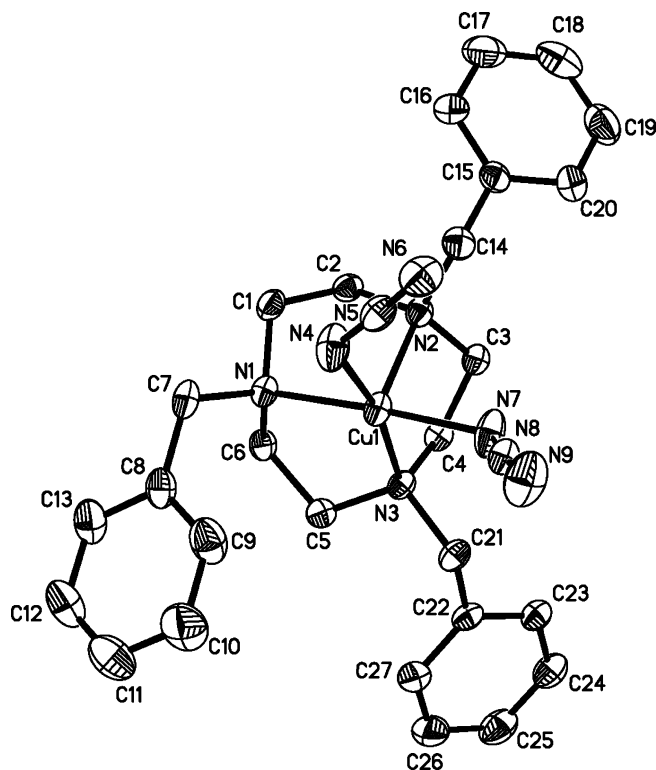

Figure 1
The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL* (Bruker, 2001).

References

Addison, A. W., Rao, T. N., Reedijk, J., van Rijn, J. & Verschoor, G. C. (1984). *J. Chem. Soc. Dalton Trans.* pp. 1349–1356.
Bruker (2001). *SAINT*, *SMART* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Daly, R. I. & Martin, L. L. (2002). *Inorg. Chem. Commun.* **5**, 777–781.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Thomas, B., Della, V., Wieghardt, K. & Roland, B. (1990). *Inorg. Chem.* **29**, 1736–1741.