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Nitrato(1,4,7-trimethyl-1,4,7-triazacyclononane)-copper(II) perchlorate

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Key indicators

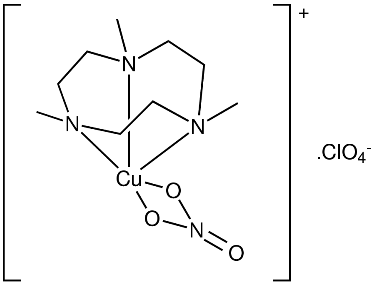
Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.005 Å
R factor = 0.037
wR factor = 0.092
Data-to-parameter ratio = 26.9

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

The Cu atom in the title compound, $[Cu(NO_3)(C_9H_{21}N_3)]ClO_4$, is coordinated facially by the tridentate triazacyclononane ligand, and also by a chelating nitrate anion. In addition, the copper makes a sixth much longer interaction [2.828 (3) Å] with an O atom of the perchlorate anion.

Comment

Recent work has reported the synthesis of $M_8(CN)_{12}$ cubes in which the formation of the cubes is directed by the occupation of three facial coordination sites of the octahedral metal ions by 'capping' ligands, such as cyclopentadiene or triazacyclononane derivatives (Heinrich *et al.*, 1998; Klausmeyer *et al.*, 1998, 1999). In such work, precursor complexes which contain one such tridentate 'capping' ligand and three further accessible coordination sites are important components in the reaction design. We report here the structure of one such complex.



(I)

The structure of the title complex, (I), is shown in Fig. 1. It consists of a Cu atom coordinated facially by the tridentate 1,4,7-trimethyl-1,4,7-triazacyclononane ligand, and also by a chelating nitrate anion (Table 1). Although the Cu atom is nominally five-coordinate, it also makes a weak interaction [2.828 (3) Å] with an O atom of the perchlorate anion. An unusual feature of this simple complex is the presence of two different types of counter-anion. This is likely due to crystal packing effects; both the nitrate and the perchlorate participate in a number of weak intermolecular C—H···O contacts with the organic ligands (Table 2).

Experimental

Blue crystals of the title complex were obtained by slow diffusion of a methanolic solution (15 ml) of sodium perchlorate (0.2 g) into a methanolic solution (15 ml) of copper nitrate (0.4 g) and 1,4,7-trimethyl-1,4,7-triazacyclononane (0.28 g).

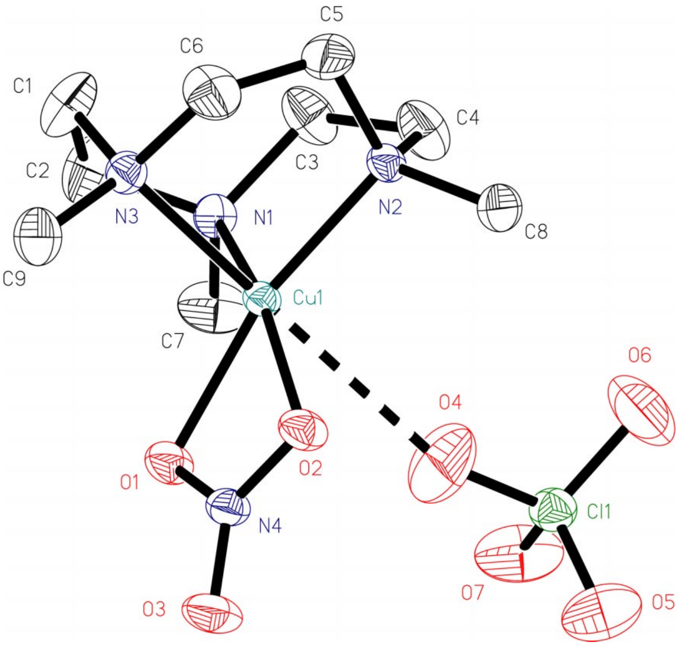


Figure 1
Atom-numbering scheme for (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms have been omitted.

Crystal data

$[Cu(NO_3)(C_9H_{21}N_3)]ClO_4$
$M_r = 396.29$
Orthorhombic, $P2_12_12_1$
$a = 8.5512\ (1)$ Å
$b = 11.8172\ (1)$ Å
$c = 15.0854\ (2)$ Å
$V = 1524.40\ (3)$ Å^3
$Z = 4$
$D_x = 1.727$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 18683 reflections
$\theta = 2.2$–$32.6°$
$\mu = 1.65$ mm^{-1}
$T = 173\ (2)$ K
Irregular, blue
0.58 × 0.50 × 0.38 mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
$T_{min} = 0.471$, $T_{max} = 0.535$
18 613 measured reflections
5357 independent reflections
5008 reflections with $I > 2\sigma(I)$
$R_{int} = 0.030$
$\theta_{max} = 32.6°$
$h = -11 \rightarrow 12$
$k = -17 \rightarrow 17$
$l = -22 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.092$
$S = 1.04$
5357 reflections
199 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0365P)^2 + 1.9698P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.26$ e Å^{-3}
$\Delta\rho_{min} = -0.52$ e Å^{-3}
Absolute structure: Flack (1983), 2287 Friedel pairs
Flack parameter = 0.022 (12)

Table 1
Selected geometric parameters (Å, °).

Cu1—N2	1.987 (2)	Cu1—N3	2.186 (2)
Cu1—N1	1.998 (2)	Cu1—N4	2.411 (2)
Cu1—O1	2.016 (2)	Cu1—O4	2.828 (3)
Cu1—O2	2.036 (2)		
N2—Cu1—N1	88.40 (9)	O1—Cu1—N3	103.91 (9)
N2—Cu1—O1	166.39 (8)	O2—Cu1—N3	105.57 (8)
N1—Cu1—O1	101.43 (8)	N2—Cu1—N4	135.96 (8)
N2—Cu1—O2	104.75 (8)	N1—Cu1—N4	132.77 (8)
N1—Cu1—O2	162.82 (8)	O1—Cu1—N4	32.11 (7)
O1—Cu1—O2	63.89 (7)	O2—Cu1—N4	31.81 (7)
N2—Cu1—N3	85.99 (8)	N3—Cu1—N4	108.32 (8)
N1—Cu1—N3	86.04 (8)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5A···O1^{i}	0.99	2.56	3.245 (3)	126
C4—H4B···O3^{ii}	0.99	2.63	3.145 (4)	113
C3—H3A···O3^{i}	0.99	2.59	3.562 (4)	166
C5—H5A···O3^{i}	0.99	2.69	3.672 (4)	172
C4—H4A···O5^{iii}	0.99	2.58	3.477 (4)	150
C2—H2A···O6^{iv}	0.99	2.56	3.506 (5)	160
C8—H8A···O7^{iii}	0.98	2.59	3.547 (4)	166
C9—H9A···O7^{i}	0.98	2.63	3.533 (5)	153
C6—H6A···O7^{v}	0.99	2.50	3.463 (5)	165

Symmetry codes: (i) $\frac{1}{2} - x, 1 - y, z - \frac{1}{2}$; (ii) $1 - x, y - \frac{1}{2}, \frac{3}{2} - z$; (iii) $\frac{3}{2} - x, 1 - y, z - \frac{1}{2}$; (iv) $x - 1, y, z$; (v) $1 - x, \frac{1}{2} + y, \frac{3}{2} - z$.

The largest residual electron-density peak is 0.84 Å from C4.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*III (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL*97.

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m266 Stuart R. Batten *et al.* • $[Cu(NO_3)(C_9H_{21}N_3)]ClO_4$ *Acta Cryst.* (2001). E**57**, m265–m266