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1,4-Dimethyl-1,4-diazoniabicyclo[2.2.2]octane tetrachlorocuprate(II)

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Key indicators

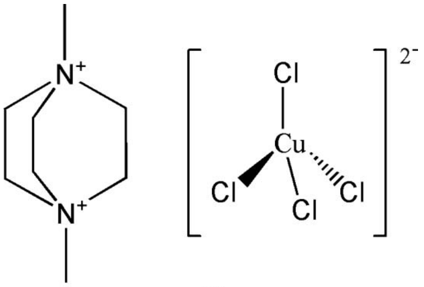
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.006 Å
R factor = 0.031
wR factor = 0.075
Data-to-parameter ratio = 16.3

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

The title compound, $(C_8H_{18}N_2)[CuCl_4]$, crystallizes with two cations and two anions in the asymmetric unit. The cation was obtained by methylation of 1,4-diazabicyclo[2.2.2]octane. C—H···Cl hydrogen-bonding interactions link cations and anions to form an infinite three-dimensional network.

Comment

Quaternary ammonium compounds (QAC) have been used widely in antimicrobial products, food production, and health care and domestic environments as antiseptics and preservatives to eliminate bacterial infections and contamination. Bis-QACs have stronger antibacterial and antifungal activities than the classical monomer type of QACs (Maeda *et al.*, 1998; Nagamune *et al.*, 2000). QACs also play an important role in cell systems as electrolytes. Here, we report the title compound, (I), a new bis-QAC, obtained by the reaction of cupric chloride with DABCO (1,4-diazabicyclo[2.2.2]-octane) in a mixture of methanol and chloroform at 423 K with pH < 1. The cation is the methylation product of DABCO.



(I)

The asymmetric unit of (I) comprises two bis-methyl DABCO cations and two $[CuCl_4]^{2-}$ anions. The two cations are each methylated at both N atoms. The two cations are ordered. One cation (N1/N2) is almost undistorted, as indicated by the N1—C—C—N2 torsion angles, which range from 0.6 (6) to 0.9 (5)°. The other cation (N3/N4) is distorted, as indicated by the N3—C—C—N4 torsion angles, which range from 5.5 (5) to 7.9 (5)°. Other DABCO cations have been observed to be ordered, for example, in DABCO–orcin (1:1) (Jin *et al.*, 2004), while disordered conformations have been observed in salts, such as in DABCO–biphenol (1:1) (Ferguson *et al.*, 1998), DABCO–perchloride acid (1:1) (Katrusiak, 2000), and DABCO–maleic acid (1:2) (Sun & Jin, 2002).

The $[CuCl_4]^{2-}$ anions exhibit approximately D_{2d} symmetry as a result of hydrogen-bonding interactions with the cations. The Cu—Cl bond lengths range from 2.2309 (14) to 2.2515 (14) Å and are close to the values observed in similar

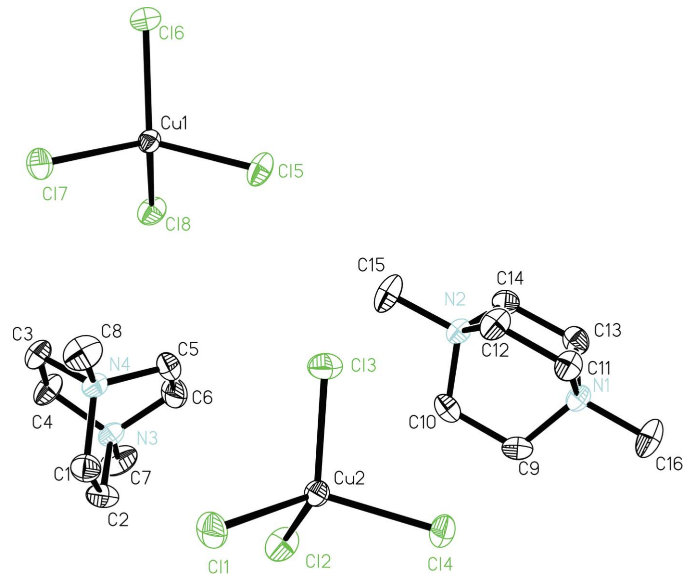


Figure 1
The asymmetric unit of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

complexes (Choi *et al.*, 2002). The Cl—Cu—Cl bond angles range from 97.59 (4) to 131.45 (5)°.

In the crystal structure of (I), there are numerous C—H···Cl hydrogen-bonding interactions between the cations and the anions (Fig. 2 and Table 2).

Experimental

$CuCl_2 \cdot 2H_2O$ (1 mmol, 170 mg) and DABCO (1,4-diazabicyclo[2.2.2]octane) (1 mmol, 112 mg) were dissolved in a methanol–chloroform (2:1) solution with pH < 1. The mixture was stirred for 20 min at room temperature to give a clear yellow solution, which was kept at 423 K for 12 h. After cooling, yellow crystals of (I) suitable for X-ray diffraction studies were formed at the bottom of the reaction vessel. The crystals were isolated, washed three times with ethanol and dried in a vacuum desiccator using anhydrous $CaCl_2$ (yield 42%). Analysis, calculated for $C_8H_{18}N_2CuCl_4$: C 27.64, H 5.22, N 8.06%; found: C 27.36, H 5.42, N 7.92%.

Crystal data

$(C_8H_{18}N_2)[CuCl_4]$
$M_r = 347.58$
Orthorhombic, $Pca2_1$
$a = 19.6572$ (16) Å
$b = 8.0741$ (7) Å
$c = 17.6413$ (15) Å
$V = 2799.9$ (4) Å^3
$Z = 8$
$D_x = 1.649$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 3800 reflections
$\theta = 2.0–28.3°$
$\mu = 2.30$ mm^{-1}
$T = 298$ (2) K
Block, yellow
0.44 × 0.34 × 0.26 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.412$, $T_{max} = 0.560$
13376 measured reflections
4444 independent reflections
4074 reflections with $I > 2\sigma(I)$
$R_{int} = 0.039$
$\theta_{max} = 25.0°$
$h = -22 \rightarrow 23$
$k = -9 \rightarrow 6$
$l = -17 \rightarrow 20$

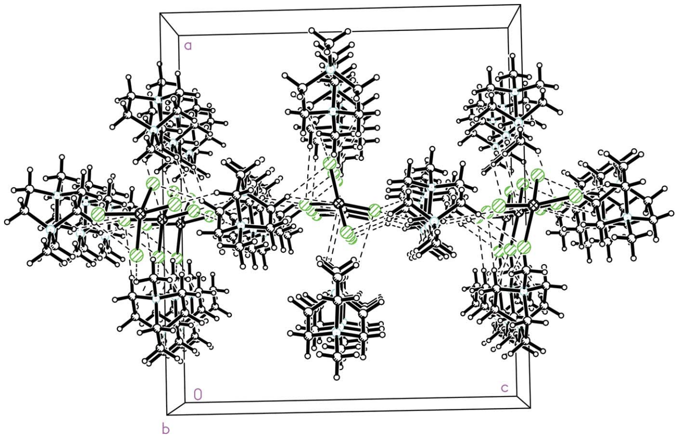


Figure 2
The crystal packing of (I), viewed along the *b* axis. Hydrogen-bonding interactions are shown as dashed lines.

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.075$
$S = 1.03$
4444 reflections
273 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0434P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.012$
$\Delta\rho_{max} = 0.32$ e Å^{-3}
$\Delta\rho_{min} = -0.34$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997a)
Extinction coefficient: 0.0088 (3)
Absolute structure: Flack (1983), 2362 Friedel pairs
Flack parameter: 0.422 (13)

Table 1
Selected geometric parameters (Å, °).

Cu1—Cl7	2.2309 (14)	Cu2—Cl3	2.2364 (14)
Cu1—Cl8	2.2407 (10)	Cu2—Cl1	2.2367 (13)
Cu1—Cl5	2.2448 (9)	Cu2—Cl2	2.2382 (10)
Cu1—Cl6	2.2515 (14)	Cu2—Cl4	2.2434 (10)
Cl7—Cu1—Cl8	104.36 (5)	Cl3—Cu2—Cl1	97.59 (4)
Cl7—Cu1—Cl5	125.81 (5)	Cl3—Cu2—Cl2	131.45 (5)
Cl8—Cu1—Cl5	98.22 (4)	Cl1—Cu2—Cl2	100.28 (5)
Cl7—Cu1—Cl6	98.11 (4)	Cl3—Cu2—Cl4	102.36 (5)
Cl8—Cu1—Cl6	130.39 (5)	Cl1—Cu2—Cl4	131.28 (5)
Cl5—Cu1—Cl6	103.42 (5)	Cl2—Cu2—Cl4	98.81 (4)
N4—C1—C2—N3	7.9 (5)	N1—C9—C10—N2	−0.9 (5)
N4—C3—C4—N3	6.5 (5)	N1—C11—C12—N2	−0.6 (6)
N4—C5—C6—N3	5.5 (5)	N1—C13—C14—N2	−0.6 (5)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2*B*···Cl2	0.97	2.77	3.604 (5)	144
C3—H3*B*···Cl7	0.97	2.68	3.556 (5)	151
C12—H12*A*···Cl3	0.97	2.71	3.626 (5)	157
C6—H6*A*···Cl7^{i}	0.97	2.77	3.446 (4)	127
C2—H2*A*···Cl5^{ii}	0.97	2.60	3.552 (5)	167
C3—H3*A*···Cl6^{iii}	0.97	2.77	3.695 (5)	160
C12—H12*B*···Cl1^{iv}	0.97	2.78	3.688 (5)	157
C13—H13*B*···Cl1^{v}	0.97	2.79	3.534 (4)	134

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

Although *PLATON* (Spek, 2003) indicates the presence of a pseudo-centre of symmetry with an 80% fit, all attempts to resolve the structure in the centrosymmetric space group *Pbcm* failed and the structure was refined as an inversion twin in the space group $Pca2_1$. All H atoms were placed in calculated positions and allowed to ride on their parent atoms at distances of 0.97 Å, with U_{iso}(H) values equal to $1.2U_{eq}$ of their parent atoms.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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