N,N'-Dimethyl-1,4-diazoniabicyclo[2.2.2]-octane tetrachlorocobaltate(II)

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The title compound, $(C_8H_{18}N_2)[CoCl_4]$, crystallizes with two cations, one complete anion and two half-anions in the asymmetric unit, two anions having twofold rotation symmetry. The cation was obtained by methylation of DABCO (1,4-diazabicyclo[2.2.2]-octane). The geometry of the $[CoCl_4]^{2-}$ anion can be described as distorted tetrahedral. C—H···Cl hydrogen-bond interactions link cations and anions in the crystal structure.

Comment

Quaternary ammonium compounds (QAC), have been widely used in antimicrobial products, food production, health care and domestic environments as antiseptics and as 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. As reported previously, under acidic conditions in a mixture of methanol and chloroform, DABCO (1,4-diazabicyclo[2.2.2]octane) is methylated (Sun & Qu, 2005), We have now extended this work to include $CoCl_2$ in place of $CuCl_2$ in order to compare their antibacterial activities.

(I)
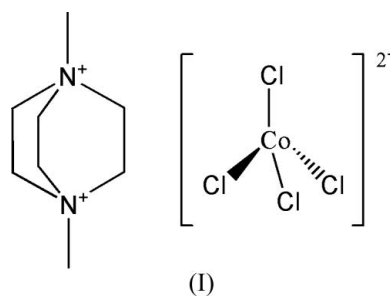
The title compound, (I), was obtained by the reaction of cobalt chloride with DABCO in a mixture of methanol and chloroform with pH < 1 and at 423 K. The cation is the methylation product of DABCO. The tetrachlorocobaltate(II) serves as a counter-ion to balance the positive charge on the quaternary ammonium centre.

The asymmetric unit of (I) comprises two dimethyl DABCO cations and one $[CoCl_4]^{2-}$ anion and two half-$[CoCl_4]^{2-}$ anions held together by C—H···Cl hydrogen bonds. Atoms Co1 and Co3 lie on twofold rotation axes. The two cations are methylated at atoms N1 and N2, and N3 and N4. Both cations are ordered and have slightly different torsion angles. The N—C—C—N torsion angles range from −3.0 (4) to 8.7 (4)°. In contrast to the present case, a disordered conformation is
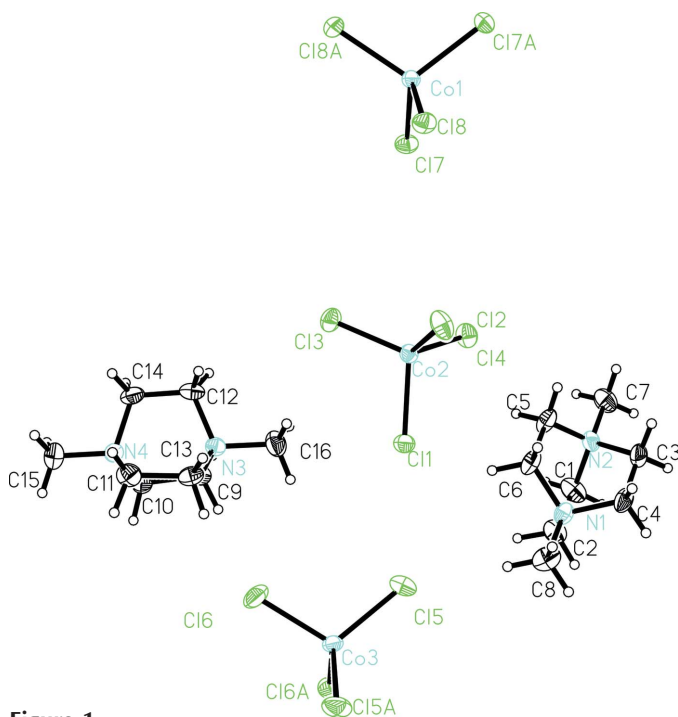
Figure 1
The ionic components of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. [Symmetry codes: (A) $-x, y, \frac{1}{2} - z$ for the Co1 anion; $1 - x, y, \frac{3}{2} - z$ for the Co3 anion.]

frequently observed in DABCO salts, such as in DABCO–biphenol (1:1) (Ferguson *et al.*, 1998), DABCO–perchloric acid (1:1) (Katrusiak, 2000), dimethyl–DABCO tetrachloro-cuprate(II) (Sun & Qu, 2005), and DABCO–maleic acid (1:2) (Sun & Jin, 2002).

The $[CoCl_4]^{2-}$ anions exhibit approximate D_{2d} symmetry. The Co1, Co2 and Co3 centres are each coordinated by four Cl atoms, at average distances of 2.2831 (8), 2.2716 (9) and 2.2612 (9) Å, respectively. The Cl—Co—Cl angles range from 104.77 (4) to 115.82 (4)°. These values are close to those observed in similar complexes (Mahmoudkhani & Langer, 2002). The $[CoCl_4]^{2-}$ anion is closer to regular tetrahedral geometry than the $[CuCl_4]^{2-}$ anion in our previous report (Sun & Qu, 2005).

In the crystal structure, there are numerous C—H⋯Cl hydrogen-bond interactions between the cations and the anions (Table 2). As a result, the $(C_8H_{18}N_2)^{2+}$ cations and $[CoCl_4]^{2-}$ anions are held together by hydrogen-bonding interactions to form an infinite three-dimensional network.

Experimental

$CoCl_2 \cdot 6H_2O$ (1 mmol, 238 mg) and DABCO (1 mmol, 112 mg) were dissolved in a methanol and chloroform (2:1) solution with the pH adjusted to < 1 with HCl. The mixture was stirred for 20 min at room temperature, then the resulting clear yellow solution was kept at 423 K for 12 h. After cooling, yellow crystals of (I) were formed at the bottom of the vessel. The crystals were isolated and washed three times with ethanol and dried in a vacuum desiccator using anhydrous $CaCl_2$ (yield = 42%). Large yellow crystals suitable for X-ray

diffraction were obtained. Analysis calculated for $C_8H_{18}CoCl_4N_2$: C 28.01, H 5.29, N 8.17%; found: C 28.36, H 5.41, N 7.82%.

Crystal data

$(C_8H_{18}N_2)[CoCl_4]$	Mo $K\alpha$ radiation
$M_r = 342.97$	Cell parameters from 2000
Orthorhombic, $Pbcn$	reflections
$a = 19.5053$ (16) Å	$\theta = 3.0–29.3°$
$b = 14.0326$ (12) Å	$\mu = 1.98$ mm^{-1}
$c = 20.2636$ (17) Å	$T = 295$ (2) K
$V = 5546.4$ (8) Å^3	Block, yellow
$Z = 16$	$0.42 \times 0.31 \times 0.23$ mm
$D_x = 1.643$ Mg m^{-3}	

Data collection

Bruker SMART CCD area-detector diffractometer	4894 independent reflections
	3928 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.042$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$\theta_{max} = 25.0°$
	$h = -23 \rightarrow 19$
$T_{min} = 0.486, T_{max} = 0.644$	$k = -16 \rightarrow 15$
26656 measured reflections	$l = -24 \rightarrow 20$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0424P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.035$	$+ 2.3343P]$
$wR(F^2) = 0.088$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} = 0.002$
4894 reflections	$\Delta\rho_{max} = 0.45$ e Å^{-3}
272 parameters	$\Delta\rho_{min} = -0.30$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Co1—Cl8	2.2757 (8)	Co2—Cl1	2.2828 (9)
Co1—Cl7	2.2905 (8)	Co2—Cl4	2.2847 (10)
Co2—Cl2	2.2496 (9)	Co3—Cl6	2.2576 (10)
Co2—Cl3	2.2693 (9)	Co3—Cl5	2.2648 (9)
Cl8^i—Co1—Cl8	113.80 (5)	Cl2—Co2—Cl4	109.74 (4)
Cl8^i—Co1—Cl7^i	108.13 (3)	Cl3—Co2—Cl4	108.07 (4)
Cl8—Co1—Cl7^i	108.64 (3)	Cl1—Co2—Cl4	104.77 (4)
Cl8^i—Co1—Cl7	108.64 (3)	Cl6^{ii}—Co3—Cl6	105.95 (7)
Cl8—Co1—Cl7	108.13 (3)	Cl6^{ii}—Co3—Cl5^{ii}	108.67 (4)
Cl7^i—Co1—Cl7	109.42 (5)	Cl6—Co3—Cl5^{ii}	109.78 (4)
Cl2—Co2—Cl3	115.82 (4)	Cl6^{ii}—Co3—Cl5	109.78 (4)
Cl2—Co2—Cl1	107.99 (4)	Cl6—Co3—Cl5	108.67 (4)
Cl3—Co2—Cl1	109.88 (4)	Cl5^{ii}—Co3—Cl5	113.69 (5)
N2—C1—C2—N1	−4.7 (4)	N3—C9—C10—N4	8.7 (4)
N2—C3—C4—N1	−3.0 (4)	N4—C11—C13—N3	8.0 (4)
N2—C5—C6—N1	−4.0 (4)	N3—C12—C14—N4	7.2 (4)

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

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C6—H6B⋯Cl1	0.97	2.71	3.530 (3)	143
C9—H9A⋯Cl6	0.97	2.70	3.562 (3)	148
C13—H13A⋯Cl6	0.97	2.67	3.561 (3)	152
C14—H14A⋯Cl5^{iii}	0.97	2.72	3.656 (3)	162
C15—H15A⋯Cl2^{iv}	0.96	2.79	3.602 (4)	143
C5—H5B⋯Cl6^v	0.97	2.78	3.599 (4)	142
C8—H8B⋯Cl4^{vi}	0.96	2.62	3.573 (4)	170
C9—H9B⋯Cl8^{vii}	0.97	2.79	3.742 (3)	167
C12—H12B⋯Cl1^{iii}	0.97	2.82	3.615 (4)	140

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

All H atoms were placed in calculated positions and allowed to ride on their parent atoms at distances of 0.96 or 0.97 Å, with $U_{iso}(H)$ values set at $1.2U_{eq}$ 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, 1997a); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997a); molecular graphics: *SHELXTL* (Sheldrick, 1997b); software used to prepare material for publication: *SHELXTL*.

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