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Bis(4-acetylanilinium) tetrachlorocuprate(II)

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Key indicators

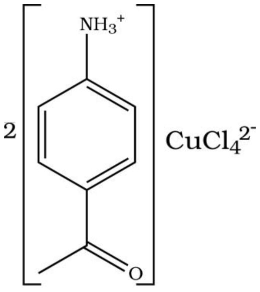
Single-crystal X-ray study
T = 293 K
Mean *σ*(C–C) = 0.005 Å
R factor = 0.040
wR factor = 0.111
Data-to-parameter ratio = 15.9

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

The title compound, $(C_8H_{10}NO)_2[CuCl_4]$, crystallizes with one 4-acetylanilinium cation and one half of a tetrachlorocuprate(II) anion in the asymmetric unit as the anion lies about a mirror plane with the Cu atom and two Cl^- ligands lying in the mirror plane. The geometry around the copper(II) ion is that of a discrete flattened tetrahedron. The cations are connected through N—H···O hydrogen bonds leading to a lamellar structure parallel to the *ab* plane. These sheets are also connected through the anions by N—H···Cl hydrogen bonds, leading to a three-dimensional hydrogen-bonded network.

Comment

Chlorocuprates occur in a variety of geometrical geometrical conformations including tetrahedral, square-pyramidal, square-bipyramidal, square-planar and trigonal–bipyramidal (Bhattacharya et al., 2004). In general, the size of the cation strongly influences the structure of the tetrachlorocuprate(II) anion. However, for very bulky organic cations, the anions exist as dimers or discrete monomers with strictly four-coordinate copper(II) (Ferguson, 1964). The geometry of the $[CuCl_4]^{2-}$ anions depends on many factors, including the Jahn–Teller effect, electrostatic repulsion between Cl atoms, crystal packing forces and hydrogen bonding (Haddad & Willett, 2001). Hence the title mononuclear Cu^{II} complex, (I), was prepared by treating copper(II) chloride with p-amino-acetophenone to investigate the hydrogen-bonding interactions in the resulting complex.



(I)

Compound (I) has a 4-acetylanilinium cation and one half of the tetrachlorocuprate(II) anion in the asymmetric unit, with the Cu^{II} atom and two of the Cl^- ligands lying in a mirror plane (Fig. 1). As described by Bhattacharya *et al.* (2004), the geometry of the $[CuCl_4]^{2-}$ species can typically be described by the average *trans* Cl—Cu—Cl angles or by the dihedral angle between $CuCl_2$ planes, which is 90° for the tetrahedral and 0° for the square-planar geometry. Here, the two inde-

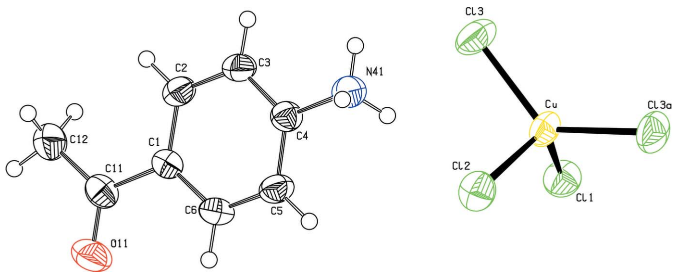


Figure 1
The molecular structure of one cation and the anion of (I), showing the atom-numbering scheme and with 50% probability displacement ellipsoids. The atom labelled Cl3a is related to Cl3 by the symmetry operation ($-x$, y, z).

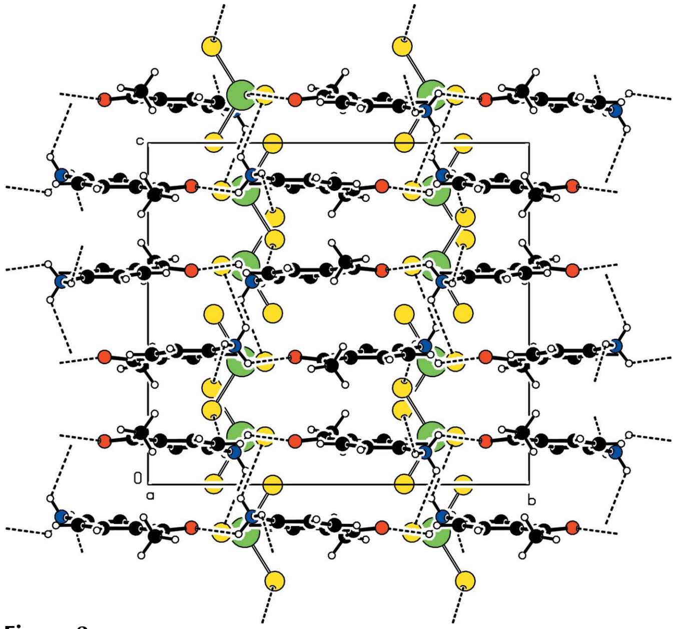


Figure 2
Packing diagram of the ions viewed down the *a* axis. Hydrogen bonds are shown as dashed lines.

pendent *trans* Cl—Cu—Cl angles are 130.14 (6) and 117.68 (5)° and the $CuCl_2$ planes are exactly perpendicular to each other by symmetry. This confirms the flattened tetrahedral geometry of the $[CuCl_4]^{2-}$ anions (Table 1).

In the cation, the acetyl group is slightly twisted from the aromatic plane by an angle of 5.3 (1)°. Protonation on the N site is well confirmed by the C—N bond distance, which is significantly longer than in the non-protonated molecule [1.335 (3) Å; Goswami *et al.*, 1999]. An N—H⋯O hydrogen bond to the O atom of the acetyl group gives a layered structure parallel to the *ab* plane. Tetrachlorocuprate(II) anions are sandwiched between these cationic sheets and form a three-dimensional N—H⋯Cl hydrogen-bonded network (Fig. 2 and Table 2).

Experimental

A solution of 4-aminoacetophenone (20 mmol) in 2 ml of hydrochloric acid (10 *M*) and deionized water (10 ml) was added to a solution (10 ml) of $CuCl_2 \cdot 2H_2O$ (10 mmol) in deionized water. The resulting solution was concentrated and dark-brown block-shaped single crystals of (I) were obtained within three days at ambient temperature.

Crystal data

$(C_8H_{10}NO)_2[CuCl_4]$
$M_r = 477.68$
Orthorhombic, *Cmca*
$a = 19.7316$ (11) Å
$b = 15.3962$ (9) Å
$c = 13.8017$ (8) Å
$V = 4193$ (1) Å^3
$Z = 8$
$D_x = 1.514$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 1.56$ mm^{-1}
$T = 293$ (2) K
Block, dark brown
0.25 × 0.23 × 0.22 mm

Data collection

Nonius MACH3 diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.671$, $T_{\max} = 0.739$
4121 measured reflections
1904 independent reflections
1464 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.049$
$\theta_{\max} = 25.0°$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.111$
$S = 1.03$
1904 reflections
120 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0554P)^2 + 5.7534P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.55$ e Å^{-3}
$\Delta\rho_{\min} = -0.38$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.00068 (11)

Table 1
Selected geometric parameters (Å, °).

C4—N41	1.466 (4)	Cu—Cl2	2.3139 (14)
Cu—Cl1	2.2223 (13)		
$Cl3^{i}$—Cu—Cl3	130.14 (6)	Cl1—Cu—Cl2	117.68 (5)
C6—C1—C11—O11	−4.2 (4)	C2—C1—C11—C12	−5.6 (4)

Symmetry code: (i) $-x, y, z$.

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N41—H41A⋯$Cl2^{ii}$	0.89	2.29	3.173 (3)	172
N41—H41B⋯$O11^{iii}$	0.89	1.90	2.787 (3)	171
N41—H41C⋯$Cl3^{iv}$	0.89	2.46	3.317 (3)	163

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

All the H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93 or 0.96 Å and N—H=0.89 Å with $U_{\rm iso}$(H) = 1.2 or $1.5U_{\rm eq}$(parent atom).

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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References

Bhattacharya, R., Chanda, S., Bocelli, G., Cantoni, A. & Ghosh, A. (2004). *J. Chem. Crystallogr.* **34**, 393–400.

Enraf–Nonius (1994). *CAD-4 EXPRESS*. Version 5.1/1.2. Enraf–Nonius, Delft, The Netherlands.

Ferguson, J. (1964). *J. Chem. Phys.* **40**, 11–19.

Goswami, S., Mahapatra, A. K., Ghosh, K., Nigam, G. D., Chinnakali, K. & Fun, H.-K. (1999). *Acta Cryst.* C**55**, 87–89.

Haddad, S. & Willett, R. D. (2001). *Inorg. Chem.* **40**, 2457–2460.

Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

Sheldrick, G. M. (1997). *SHELXS97 and SHELXL97*. University of Göttingen, Germany.

Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

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