

Bis(2-methylbenzimidazole- κN^1)copper(I) dichlorocuprate(I)

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Key indicators

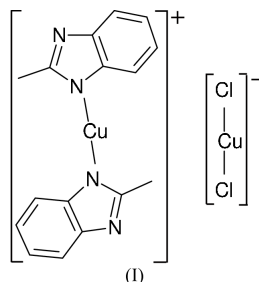
Single-crystal X-ray study
T = 295 K
Mean $\sigma(\text{C}-\text{C}) = 0.005 \text{ \AA}$
R factor = 0.044
wR factor = 0.124
Data-to-parameter ratio = 15.6

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

By using alternating-current electrochemical synthesis, crystals of the copper(I) ionic compound composed of $[\text{Cu}(\text{C}_8\text{H}_8\text{N}_2)_2]^+$ and $[\text{CuCl}_2]^-$ ions have been obtained and structurally investigated. Both crystallographically independent Cu atoms lie on centres of inversion and exhibit the less-common coordination number 2. A linear arrangement of the metal atoms includes two N atoms of the different organic moieties in the $[\text{Cu}(\text{2-methylbenzimidazole})_2]^+$ cation and two Cl atoms in the case of the inorganic anion.

Comment

Complexes of copper(I) halogenides with small neutral ligands usually contain large inorganic fragments (for example, Massaux & Le Bihan, 1976). On the other hand, such compounds with more voluminous bicyclic aromatic units frequently appear with small isolated inorganic fragments (Goreschnik, 1999; Goreschnik *et al.*, 2002). In these complexes, the aromatic moieties show an ability for stacking and play an important role in the structure formation, especially in compounds without direct metal–ligand bonding (Goreschnik *et al.*, 1999, 2000). To estimate the influence of ligand packing on structure formation, the title compound $[\text{Cu}(\text{C}_8\text{H}_8\text{N}_2)_2]^+[\text{CuCl}_2]^-$, (I), has been synthesized and structurally investigated.



The title compound is an ionic copper(I) σ -complex and consists of metal–organic $[\text{Cu}(\text{2-methylbenzimidazole})_2]^+$ cations and $[\text{CuCl}_2]^-$ anions. The metal atoms in both the cation and anion lie on inversion centres. In each cation, a pair of symmetrically equivalent ligands coordinates to a Cu atom. All the benzimidazole ligands are oriented strictly parallel, with typical stacking ring–ring distances of 3.474 (3) \AA . The linear $[\text{CuCl}_2]^-$ anions are oriented parallel to the benzimidazole planes and are connected to the metal–organic cations *via* especially strong $[d(\text{H}\cdots\text{Cl})/(r_{\text{H}} + r_{\text{Cl}}) = 0.78$; Brammer *et al.*, 2002] N–H \cdots Cl hydrogen bonds. The Cu–Cl distance of 2.0967 (10) \AA is comparable with previously observed values of 2.093 (4) \AA in the copper(I) chloride complex with a dimer of *trans*-1-cyano-1,2-butadiene, *viz.*

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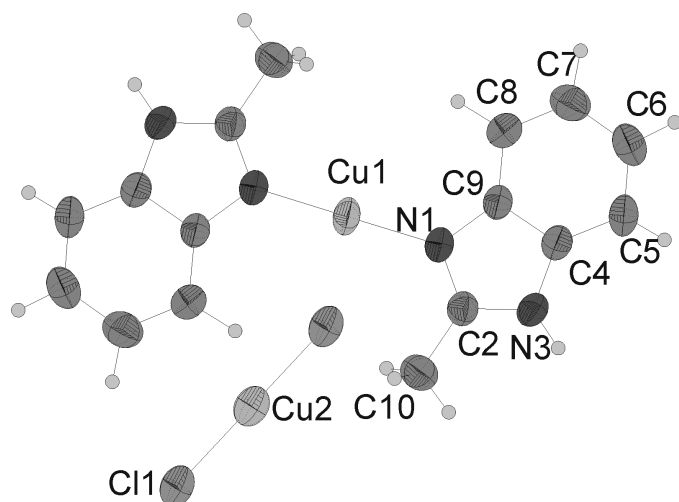


Figure 1
The structure of (I), shown with 50% probability displacement ellipsoids.

$[\text{Cu}(\text{C}_{10}\text{H}_{10}\text{N}_2)_2]\text{CuCl}_3$ (Olijnyk *et al.*, 1985), and 2.09 (1) Å in the π -complex $[\text{Cu}_2\text{Cl}_3(\text{CH}_2=\text{CH}-\text{H}_2-\text{NH}_3)_2]\text{CuCl}_2$ (Olijnyk, 1999).

Experimental

2-Methylbenzimidazole was obtained from *o*-phenylenediamine and acetic acid. Good quality crystals of the title compound were obtained using the alternating-current electrochemical technique (Mykhalichko & Mys'kiv, 1998), starting from 2-methylbenzimidazole and copper(II) chloride. To 2 ml of an ethanol solution of $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ (1 mmol), 2 ml of an ethanol solution of 2-methylbenzimidazole (1.2 mmol) was added. The resulting solution was placed in a small test-tube and copper-wire electrodes in cork were inserted. After applying 0.30 V alternating current (frequency 50 Hz) for some days, colourless crystals of the title compound appeared on the copper electrodes.

Crystal data

$[\text{Cu}(\text{C}_8\text{H}_8\text{N}_2)_2][\text{CuCl}_2]$
 $M_r = 462.31$
 Triclinic, $P\bar{1}$
 $a = 7.2510$ (10) Å
 $b = 7.4873$ (10) Å
 $c = 9.7058$ (13) Å
 $\alpha = 112.628$ (10)°
 $\beta = 98.786$ (11)°
 $\gamma = 103.832$ (11)°
 $V = 454.32$ (11) Å³
 $Z = 1$
 $D_x = 1.690$ Mg m⁻³

$D_m = 1.7$ Mg m⁻³
 D_m measured by flotation in chloroform–bromoform
 Cu $K\alpha$ radiation
 Cell parameters from 25 reflections
 $\theta = 65\text{--}73^\circ$
 $\mu = 5.63$ mm⁻¹
 $T = 295$ (2) K
 Block, colourless
 0.40 × 0.20 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
 $\omega/2\theta$ scans
 Absorption correction: numerical (de Meulenaar & Tompa, 1965)
 $T_{\min} = 0.212$, $T_{\max} = 0.399$
 1986 measured reflections
 1835 independent reflections
 1676 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.051$
 $\theta_{\text{max}} = 74.0^\circ$
 $h = -9 \rightarrow 0$
 $k = -9 \rightarrow 9$
 $l = -11 \rightarrow 12$
 3 standard reflections
 frequency: 60 min
 intensity decay: 3%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.044$
 $wR(F^2) = 0.125$
 $S = 1.13$
 1835 reflections
 118 parameters
 Only H-atom U 's refined

$$w = 1/[\sigma^2(F_o^2) + (0.0731P)^2 + 0.1751P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.54 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.48 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (Å, °).

Cu1–N1 ⁱ	1.874 (2)	N3–C4	1.373 (3)
Cu1–N1	1.874 (2)	C4–C5	1.385 (4)
Cu2–Cl1	2.0967 (10)	C4–C9	1.393 (3)
Cu2–Cl1 ⁱⁱ	2.0967 (10)	C5–C6	1.373 (4)
N1–C2	1.321 (3)	C6–C7	1.405 (5)
N1–C9	1.391 (3)	C7–C8	1.372 (5)
C2–N3	1.349 (3)	C8–C9	1.393 (4)
C2–C10	1.486 (4)		
N1 ⁱ –Cu1–N1	180.0	C5–C4–C9	122.8 (2)
Cl1–Cu2–Cl1 ⁱⁱ	180.0	C6–C5–C4	116.7 (3)
C2–N1–C9	106.0 (2)	C5–C6–C7	121.2 (3)
N1–C2–N3	111.4 (2)	C8–C7–C6	121.8 (3)
N1–C2–C10	125.8 (2)	C7–C8–C9	117.6 (3)
N3–C2–C10	122.8 (2)	N1–C9–C4	108.9 (2)
C2–N3–C4	108.4 (2)	N1–C9–C8	131.1 (2)
N3–C4–C5	131.9 (2)	C4–C9–C8	120.0 (2)
N3–C4–C9	105.3 (2)		
C9–N1–C2–N3	−0.2 (3)	C5–C6–C7–C8	−0.5 (5)
C9–N1–C2–C10	179.0 (2)	C6–C7–C8–C9	0.5 (5)
N1–C2–N3–C4	0.3 (3)	C2–N1–C9–C4	0.0 (3)
C10–C2–N3–C4	−178.9 (2)	N3–C4–C9–N1	0.2 (3)
C2–N3–C4–C5	179.7 (3)	C5–C4–C9–N1	−179.8 (2)
C2–N3–C4–C9	−0.3 (3)	N3–C4–C9–C8	−179.5 (2)
N3–C4–C5–C6	179.5 (3)	C5–C4–C9–C8	0.5 (4)
C9–C4–C5–C6	−0.4 (4)	C7–C8–C9–N1	179.9 (3)
C4–C5–C6–C7	0.4 (4)	C7–C8–C9–C4	−0.5 (4)

Symmetry codes: (i) $-x, 1-y, 1-z$; (ii) $-x, 2-y, -z$.

Table 2

Hydrogen-bonding geometry (Å, °).

$D\cdots H\cdots A$	$D\cdots H$	$H\cdots A$	$D\cdots A$	$D\cdots H\cdots A$
N3–H3 \cdots Cl1	0.91	2.29	3.199 (2)	173

H atoms were placed at calculated positions and refined with a riding-motion model.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *CORINC* (Dräger & Gattow, 1971); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Bergerhoff, 1996); software used to prepare material for publication: *enCIFer* (CCDC, 2002).

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