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Hexa-μ_2-chloro-tetrakis(2-ethylpyrazine-*N*)-μ_4-oxo-tetracopper(II)

Christian Näther* and Inke Jeß

Institut für Anorganische Chemie, Christian-Albrechts-Universität Kiel, Olshausenstr. 40, D-24098 Kiel, Germany

Correspondence e-mail: cnaether@ac.uni-kiel.de

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.036
wR factor = 0.100
Data-to-parameter ratio = 21.4

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

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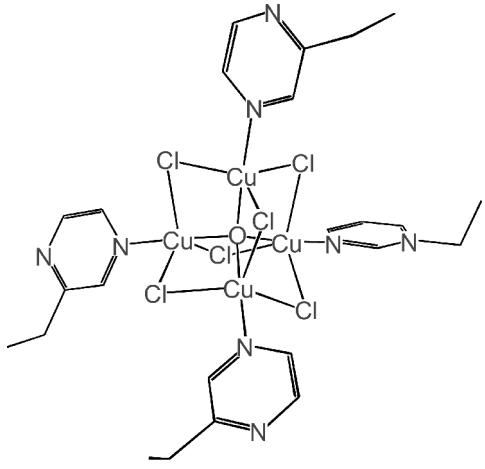
In the crystal structure of the title compound, $[Cu_4Cl_6O(C_6H_8N_2)_4]$, Cu_4 tetrahedra are found which are centred by an interstitial O atom. Each edge of the Cu_4 tetrahedron is bridged by a chloro ligand. The copper(II) cations are fourfold coordinated by one O atom, two chloro ligands and one N atom of the 2-ethylpyrazine ligand within a distorted tetrahedron. The $Cu_4Cl_6O(C_6H_8N_2)_4$ units are located in general positions.

Comment

This work is a part of a project dealing with the synthesis, structures and properties of coordination polymers based on copper halides and multidentate amino ligands (Näther *et al.*, 2001; Näther & Greve, 2001; Näther & Jess, 2001). Crystals of the title compound, (I), were obtained by the reaction of copper(I) chloride with 2-ethylpyrazine in acetonitrile.



(I)

In the crystal structure of the title compound, four copper(II) cations form a slightly distorted tetrahedron which is centred by an interstitial O atom. The Cu—O bond lengths are between 1.895 (2) and 1.907 (2) Å, and the Cu—O—Cu angles between 108.30 (8) and 110.22 (8)°. The edges of the Cu_4 tetrahedron are each bridged by μ_2-chloro ligands. The chloro bridges are not symmetrical, with Cu—Cl bond lengths between 2.3485 (8) and 2.4591 (8) Å. The copper coordination is completed by a 2-ethylpyrazine ligand which is coordinated by the N atom which is bonded through the ethyl group. The Cu—N bond lengths are between 1.977 (3) and 1.987 (2) Å. Presumably due to steric repulsion the second N atom of the 2-ethylpyrazine ligand is not involved in copper coordination.

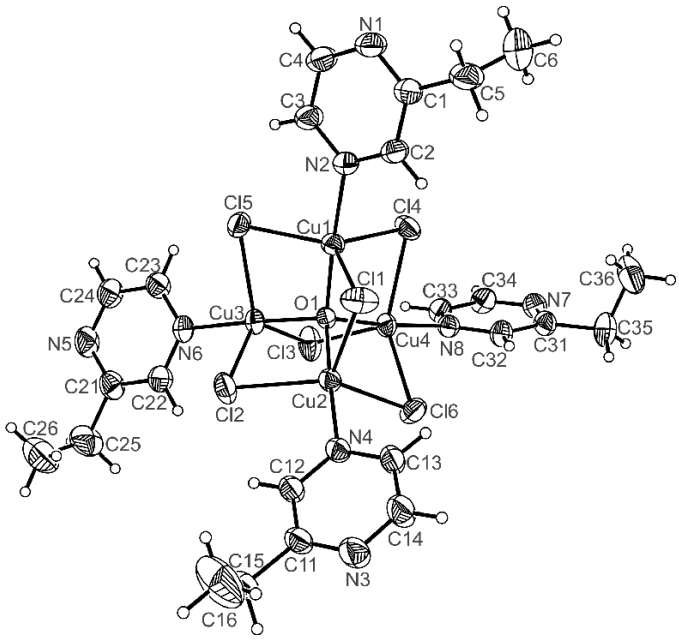


Figure 1
The crystal structure of the title compound with atom labelling and with isplacement ellipsoids drawn at the 50% probability level.

Bond lengths and angles are comparable to those in other amine-coordinated Cu_4Cl_6O clusters, such as μ_4-oxo-hexakis-(μ_2-chloro)tetrakis(nicotine-*N*)tetracopper(II) (Haendler, 1990), μ_4-oxo-hexakis(μ_2-chloro)tetrakis(acetonitrile-*N*)-tetracopper(II) acetonitrile solvate (Hiller *et al.*, 1990), μ_4-oxo-hexakis(μ_2-chloro)tetrakis(imidazole-*N*)tetracopper(II) (Atria *et al.*, 1999) and μ_4-oxo-hexakis(μ_2-chloro)tris(*N*-methyl-2-pyrrolidinone-*O*)aquatetracopper(II) (Churchill & Rotella, 1979).

Experimental

Copper(I) chloride (98.0 mg, 1 mmol; freshly prepared according to Gmelin, 1958) and 2-ethylpyrazine (216.3 mg, 2 mmol; ACROS) were mixed in 2 ml acetonitrile in a glass container. After mixing the components, a red precipitate of CuCl(2-ethylpyrazine) was initially formed which transformed into large yellow crystals of the title compound over a period of two days.

Crystal data

$[Cu_4Cl_6O(C_6H_8N_2)_4]$
M_r = 915.44
Monoclinic, $P2_1/n$
a = 13.1203 (10) Å
b = 13.0461 (7) Å
c = 20.7233 (16) Å
β = 99.718 (9)°
V = 3496.3 (4) Å^3
Z = 4
D_x = 1.739 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8000 reflections
θ = 15–24°
μ = 2.89 mm^{-1}
T = 293 (2) K
Block, yellow
0.14 × 0.10 × 0.06 mm

Data collection

Stoe Imaging Plate Diffraction System diffractometer
φ scans
Absorption correction: numerical (*X-SHAPE*; Stoe & Cie, 1998)
T_{min} = 0.717, T_{max} = 0.849
30661 measured reflections
8309 independent reflections
6876 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 28.1°
$h = -17 \rightarrow 17$
$k = -16 \rightarrow 17$
$l = -27 \rightarrow 27$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.100
S = 1.03
8309 reflections
389 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.066P)^2 + 1.0923P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.57 e Å^{-3}
$\Delta\rho_{min}$ = −0.56 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0064 (5)

Table 1
Selected geometric parameters (Å, °).

Cu1—O1	1.9073 (18)	Cu3—O1	1.8974 (16)
Cu1—N2	1.986 (2)	Cu3—N6	1.978 (2)
Cu1—Cl1	2.3555 (8)	Cu3—Cl3	2.3485 (8)
Cu1—Cl5	2.4005 (7)	Cu3—Cl5	2.4196 (8)
Cu1—Cl4	2.4438 (7)	Cu3—Cl2	2.4591 (8)
Cu2—O1	1.8946 (17)	Cu4—O1	1.9010 (17)
Cu2—N4	1.987 (2)	Cu4—N8	1.977 (2)
Cu2—Cl2	2.3765 (7)	Cu4—Cl4	2.3835 (8)
Cu2—Cl6	2.4199 (8)	Cu4—Cl6	2.3968 (8)
Cu2—Cl1	2.4314 (9)	Cu4—Cl3	2.4325 (8)
O1—Cu1—N2	175.83 (8)	O1—Cu3—Cl2	82.84 (5)
O1—Cu1—Cl1	86.05 (5)	N6—Cu3—Cl2	95.05 (7)
N2—Cu1—Cl1	95.29 (7)	Cl3—Cu3—Cl2	126.99 (4)
O1—Cu1—Cl5	84.77 (5)	Cl5—Cu3—Cl2	104.57 (3)
N2—Cu1—Cl5	97.31 (7)	O1—Cu4—N8	174.92 (9)
Cl1—Cu1—Cl5	130.20 (3)	O1—Cu4—Cl4	85.33 (6)
O1—Cu1—Cl4	83.51 (5)	N8—Cu4—Cl4	89.72 (7)
N2—Cu1—Cl4	92.37 (7)	O1—Cu4—Cl6	84.87 (5)
Cl1—Cu1—Cl4	118.00 (3)	N8—Cu4—Cl6	97.38 (7)
Cl5—Cu1—Cl4	109.37 (3)	Cl4—Cu4—Cl6	128.47 (3)
O1—Cu2—N4	177.53 (8)	O1—Cu4—Cl3	83.70 (5)
O1—Cu2—Cl2	85.20 (5)	N8—Cu4—Cl3	99.66 (7)
N4—Cu2—Cl2	97.03 (7)	Cl4—Cu4—Cl3	118.08 (3)
O1—Cu2—Cl6	84.36 (5)	Cl6—Cu4—Cl3	110.87 (4)
N4—Cu2—Cl6	93.34 (7)	Cu1—Cl1—Cu2	80.13 (2)
Cl2—Cu2—Cl6	132.44 (3)	Cu2—Cl2—Cu3	79.59 (2)
O1—Cu2—Cl1	84.18 (6)	Cu3—Cl3—Cu4	80.54 (2)
N4—Cu2—Cl1	95.83 (8)	Cu4—Cl4—Cu1	80.39 (2)
Cl2—Cu2—Cl1	114.02 (3)	Cu1—Cl5—Cu3	80.64 (2)
Cl6—Cu2—Cl1	110.85 (3)	Cu4—Cl6—Cu2	80.53 (2)
O1—Cu3—N6	177.38 (10)	Cu2—O1—Cu3	109.45 (8)
O1—Cu3—Cl3	86.14 (5)	Cu2—O1—Cu4	110.22 (8)
N6—Cu3—Cl3	93.91 (7)	Cu3—O1—Cu4	108.94 (8)
O1—Cu3—Cl5	84.44 (6)	Cu2—O1—Cu1	108.30 (8)
N6—Cu3—Cl5	97.62 (8)	Cu3—O1—Cu1	110.12 (9)
Cl3—Cu3—Cl5	125.72 (3)	Cu4—O1—Cu1	109.80 (8)

H atoms were positioned with idealized geometry and refined with fixed isotropic displacement parameters [U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(O or C_{methyl})] using a riding model.

Data collection: *IPDS Program Package* (Stoe & Cie, 1998); cell refinement: *IPDS Program Package*; data reduction: *IPDS Program Package*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *CIFTAB* in *SHELXL*97.

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