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Hexakis(dimethyl sulfoxide)copper(II) bis(hydrogen sulfate)

Susanne Bieller, Hans-Wolfram Lerner and Michael Bolte*

Institut für Anorganische Chemie, J. W. Goethe-Universität Frankfurt, Marie-Curie-Str. 11, 60439 Frankfurt/Main, Germany

Correspondence e-mail:
bolte@chemie.uni-frankfurt.de

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The title compound, $[Cu(C_2H_6OS)_6](HSO_4)_2$, is composed of discrete $[Cu(C_2H_6OS)_6]^{2+}$ cations and HSO_4^- anions. The Cu atom is located on a centre of inversion. Two HSO_4^- anions are connected *via* hydrogen bonds, forming a centrosymmetric dimer.

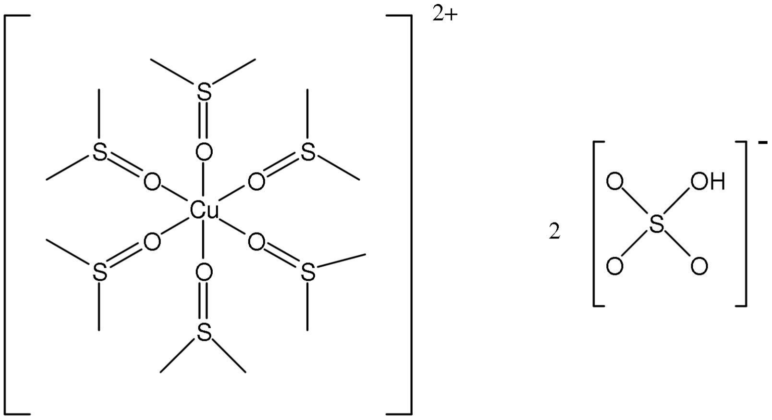
Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(S–C) = 0.002 Å
R factor = 0.023
wR factor = 0.058
Data-to-parameter ratio = 16.3

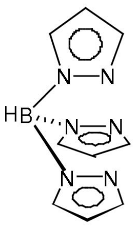
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

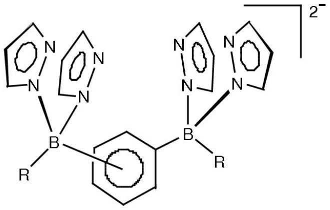
Tris(1-pyrazolyl)borates, (1) (‘scorpinates’), were reported by Trofimenko (1993) more than 30 years ago and are today well established as ligands in coordination chemistry, but only a limited number of transition metal complexes with the ditopic heteroscorpionate ligand, (2), have been structurally characterized (Zhang *et al.*, 2004, and references therein). Therefore, we became interested in the reaction of ligand (2) with Cu^{2+} ions. In an attempt to synthesize the Cu^{II} complex with ligand (2), crystals of the title compound, (I), were obtained.



(I)



(1)



(2)

A perspective view of (I) is shown in Fig. 1. The structure is composed of discrete $[Cu(C_2H_6OS]^{2+}$ cations and HSO_4^-

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anions. Bond lengths and angles can be regarded as normal (Cambridge Crystallographic Database, Version 1.7 plus one update; *MOGUL*, Version 1.0; Allen, 2002). The Cu atom is located on a centre of inversion. The Cu—O bond lengths (Table 1) show significant differences, a fact that can be attributed to the Jahn–Teller effect. The S—O bonds in the hydrogen sulfate anion also show differences. The bond to the O atom carrying the H atom is significantly longer than the other three S—O bonds. Two HSO_4^- anions are connected *via* hydrogen bonds to form a centrosymmetric dimer.

Experimental

Colourless crystals of the title compound were obtained from a solution of $CuCl_2$ (0.095 g, 0.71 mmol) and the $2K^+\cdot(2)$, where *R* is *tert*-butyl, in dimethyl sulfoxide (10 ml) in the presence of H_2SO_4 at room temperature.

Crystal data

$[Cu(C_2H_6OS)_6](HSO_4)_2$
$M_r = 726.44$
Triclinic, $P\bar{1}$
$a = 8.2271$ (7) Å
$b = 9.4663$ (8) Å
$c = 9.5945$ (9) Å
$\alpha = 97.559$ (7)°
$\beta = 90.415$ (7)°
$\gamma = 90.238$ (7)°
$V = 740.70$ (11) Å^3
$Z = 1$
$D_x = 1.629$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 21 283 reflections
$\theta = 3.8$–25.6°
$\mu = 1.36$ mm^{-1}
$T = 173$ (2) K
Block, colourless
0.42 × 0.35 × 0.33 mm

Data collection

Stoe IPDS-II two-circle diffractometer
ω scans
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
$T_{min} = 0.576$, $T_{max} = 0.636$
12 096 measured reflections
2693 independent reflections
2691 reflections with $I > 2\sigma(I)$
$R_{int} = 0.024$
$\theta_{max} = 25.6°$
$h = -10 \rightarrow 9$
$k = -11 \rightarrow 11$
$l = -11 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.023$
$wR(F^2) = 0.058$
$S = 1.11$
2693 reflections
165 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.025P)^2 + 0.6238P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.30$ e Å^{-3}
$\Delta\rho_{min} = -0.36$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.042 (4)

Table 1
Selected interatomic distances (Å).

Cu1—O1	1.9755 (11)	S4—O44	1.4442 (15)
Cu1—O3	2.0223 (11)	S4—O42	1.4791 (14)
Cu1—O2	2.3437 (12)	S4—O41	1.5806 (14)
S4—O43	1.4379 (16)		

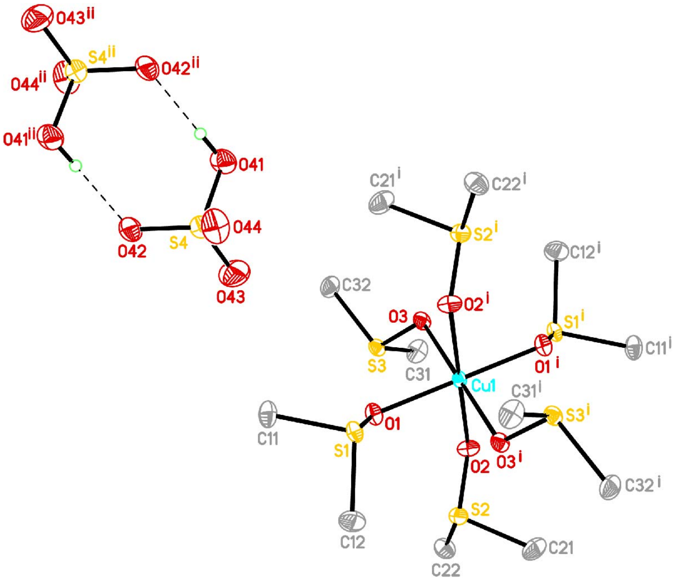

Figure 1
Perspective view of the title compound, with the atom numbering; displacement ellipsoids are at the 50% probability level and H atoms bonded to C atoms have been omitted for clarity. [Symmetry codes: (i) $1 - x, 1 - y, 1 - z$; (ii) $-x, 2 - y, -z$.]

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

D—H···A	D—H	H···A	D···A	D—H···A
O41—H41···O42^{ii}	0.80 (3)	1.82 (3)	2.620 (2)	179 (3)

Symmetry code: (ii) $-x, -y + 2, -z$.

All H atoms were located in a difference map. Methyl H atoms were refined with fixed individual displacement parameters [$U_{iso}(H) = 1.5U_{eq}(C)$] using a riding model, with C—H = 0.98 Å. The hydroxy H atom was freely refined.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

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