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Bis(tetraethylammonium) bis[cyanocuprate(I)]-tetrathiomolybdate(VI) hydrate

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The title complex, $(C_8H_{20}N)_2[MoS_4(CuCN)_2]\cdot H_2O$, contains an anion in which Cu atoms bridge two opposite S···S edges of the central MoS_4 unit. The Mo atom retains tetrahedral coordination geometry, while each Cu is trigonal planar coordinated by two bridging sulfides and a terminal cyano ligand. The anions are linked together into chains by hydrogen bonding to ordered water molecules.

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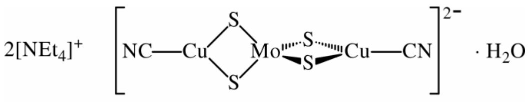
Key indicators

Single-crystal X-ray study
T = 160 K
Mean σ(C–C) = 0.003 Å
R factor = 0.024
wR factor = 0.056
Data-to-parameter ratio = 24.4

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

Comment

The anion of the title compound, (I), is one of the simplest complexes in which Cu^I is attached to the S atoms of a tetrathiomolybdate or tetrathiotungstate unit. Such complexes are convenient starting materials for the synthesis of other complexes with different ligands, by substitution of the cyanide ligands. Attempted use of (I) in this way for the generation of complexes with chelating and/or bridging nitrogen ligands led unexpectedly to the crystallization of this hydrate (Beheshti *et al.*, 2002).



(I)

The asymmetric unit of (I) consists of two independent $(NEt_4)^+$ cations, with unremarkable geometry, an $[MoS_4(CuCN)_2]^{2-}$ anion, and a water molecule, all in general positions (Fig. 1). In the anion, the coordination of the Cu and Mo atoms are essentially trigonal planar and tetrahedral, respectively, and each MoS_2Cu moiety is planar. Selected bond lengths and angles are given in Table 1.

The molecular geometry of (I) is very similar to that in the tetraphenylarsonium salt $(Ph_4As)_2[MoS_4(CuCN)_2]\cdot H_2O$, which is also a monohydrate (Gheller *et al.*, 1984). However, the space groups and packing arrangements of these two compounds are different, because of the very different sizes of the cations in the two structures. Unlike in the tetraphenylarsonium salt, the water molecule in (I) is ordered and engages in hydrogen bonding to the N atoms of the anionic cyanide ligands (Table 2), thus linking these anions together into chains (Fig. 2). The anhydrous tetramethylammonium salt, by contrast, is polymeric through formation of cyanide bridges between Cu atoms (Müller *et al.*, 1981).

Experimental

Crystals of the title compound were obtained from a solution of the anhydrous material in acetone during an attempt to replace the cyanide ligands by pyrazolylborate ligands (Beheshti *et al.*, 2002).

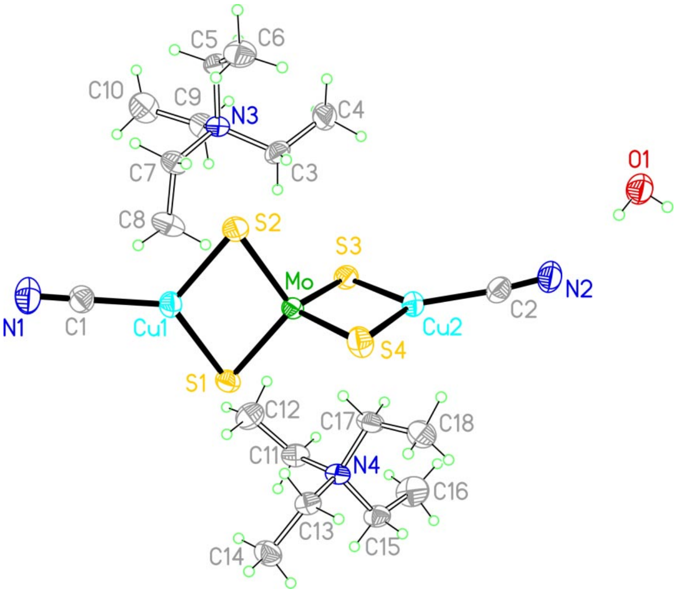


Figure 1

The complete asymmetric unit of (I), with atom labels and 50% probability ellipsoids for non-H atoms.

Crystal data

$(C_8H_{20}N)_2[Cu_2MoS_4(CN)_2]\cdot H_2O$
$M_r = 681.82$
Monoclinic, $P2_1/n$
$a = 14.1202$ (5) Å
$b = 7.1989$ (3) Å
$c = 29.3581$ (10) Å
$\beta = 99.592$ (2)°
$V = 2942.53$ (19) Å^3
$Z = 4$

$D_x = 1.539$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 19634 reflections
$\theta = 2.3$–28.6°
$\mu = 2.15$ mm^{-1}
$T = 160$ (2) K
Block, orange
0.36 × 0.26 × 0.16 mm

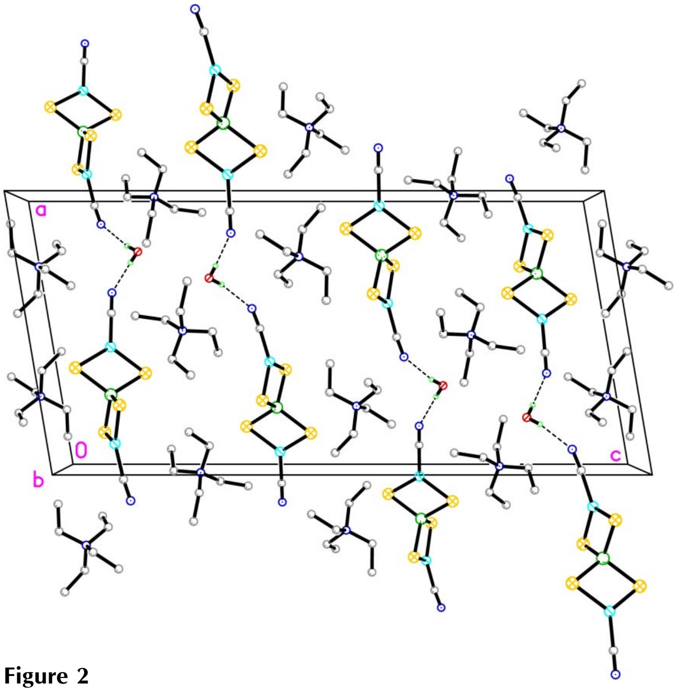


Figure 2

A packing diagram, viewed down the *b* axis, showing the hydrogen bonds as dashed lines.

Data collection

Bruker SMART CCD diffractometer
ω rotation with narrow frames
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997*a*)
$T_{min} = 0.511$, $T_{max} = 0.725$
24398 measured reflections

7028 independent reflections
6142 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 28.6°$
$h = -18 \rightarrow 18$
$k = -9 \rightarrow 9$
$l = -38 \rightarrow 39$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.024$
$wR(F^2) = 0.056$
$S = 1.06$
7028 reflections
288 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0244P)^2 + 1.151P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.003$
$\Delta\rho_{max} = 0.42$ e Å^{-3}
$\Delta\rho_{min} = -0.36$ e Å^{-3}
Extinction correction: *SHELXTL*
Extinction coefficient: 0.00051 (8)

Table 1

Selected geometric parameters (Å, °).

Mo—S1	2.2222 (5)	S3—Cu2	2.2156 (5)
Mo—S2	2.2076 (5)	S4—Cu2	2.2101 (5)
Mo—S3	2.2089 (5)	Cu1—C1	1.889 (2)
Mo—S4	2.2143 (5)	C1—N1	1.137 (3)
S1—Cu1	2.2136 (5)	Cu2—C2	1.890 (2)
S2—Cu1	2.2111 (5)	C2—N2	1.136 (3)
S1—Mo—S2	107.103 (19)	Mo—S4—Cu2	72.496 (17)
S1—Mo—S3	111.174 (19)	S1—Cu1—S2	107.285 (19)
S1—Mo—S4	111.78 (2)	S1—Cu1—C1	127.66 (6)
S2—Mo—S3	109.561 (19)	S2—Cu1—C1	125.04 (6)
S2—Mo—S4	109.73 (2)	Cu1—C1—N1	179.4 (2)
S3—Mo—S4	107.483 (19)	S3—Cu2—S4	107.40 (2)
Mo—S1—Cu1	72.640 (15)	S3—Cu2—C2	126.55 (6)
Mo—S2—Cu1	72.968 (16)	S4—Cu2—C2	125.59 (6)
Mo—S3—Cu2	72.494 (16)	Cu2—C2—N2	174.46 (19)

Table 2

Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*A*···N1^{i}	0.79 (3)	2.10 (3)	2.889 (3)	178 (3)
O1—H1*B*···N2	0.79 (3)	2.12 (3)	2.897 (3)	170 (3)

Symmetry code: (i) $x - 1, y - 1, z$.

H atoms attached to C atoms were placed geometrically and refined with a riding model (including free rotation about C—C bonds), and with U_{iso} constrained to be 1.2 (1.5 for methyl groups) times U_{eq} of the carrier atom. H atoms of the water molecule were refined freely, with individual U_{iso} values.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997*b*); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and local programs.

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