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catena-Poly[copper(II)-di-μ_2-1,1-azido-copper(II)-μ_2-acetato-μ_2-1,1-azido-μ_2-(dimethyl sulfoxide)-copper(II)-di-μ_2-1,1-azido]

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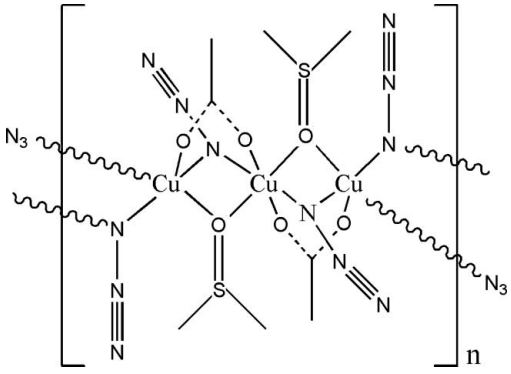
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.035; wR factor = 0.099; data-to-parameter ratio = 16.6.

The title crystal structure, $[Cu_3(C_2H_3O_2)_2(N_3)_4(C_2H_6OS)_2]_n$, consists of one-dimensional chains in which there are two independent Cu^{II} ions. One of the Cu^{II} ions lies on a crystallographic inversion center and is in a slightly distorted octahedral coordination environment while the other Cu^{II} ion is coordinated in a distorted square-pyramidal environment.

Related literature

For related literature, see: Goher *et al.* (1999, 2002); Liu *et al.* (2007); Song *et al.* (2007).



Experimental

Crystal data

$[Cu_3(C_2H_3O_2)_2(N_3)_4(C_2H_6OS)_2]$
M_r = 633.13
Triclinic, $P\overline{1}$
a = 8.5205 (9) Å
b = 8.6624 (9) Å
c = 9.4999 (10) Å
α = 90.386 (2)°
β = 112.687 (1)°
γ = 116.261 (1)°
V = 566.66 (10) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 3.02 mm^{-1}
T = 293 (2) K
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: none
3467 measured reflections
2404 independent reflections
2182 reflections with $I > 2\sigma(I)$
R_{int} = 0.060

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.099
S = 1.11
2404 reflections
145 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.60 e Å^{-3}
$\Delta\rho_{min}$ = −0.65 e Å^{-3}

Table 1
Selected bond lengths (Å).

Cu1—O1	1.933 (2)	Cu1—O3	2.351 (2)
Cu1—N4^{i}	1.970 (2)	Cu2—O2	1.936 (2)
Cu1—N1	1.986 (3)	Cu2—N4	1.995 (2)
Cu1—N1^{ii}	2.002 (3)	Cu2—O3	2.542 (2)

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2563).

References

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Liu, F. C., Zeng, Y. F., Zhao, J. P., Hu, B. W., Bu, X. H., Ribas, J. & Batten, S. R. (2007). *Inorg. Chem. Commun.* **10**, 129–132.
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supplementary materials

Acta Cryst. (2007). E**63**, m3156 [**doi:10.1107/S1600536807059247**]

catena-Poly[copper(II)-di-μ_2-1,1-azido-copper(II)-μ_2-acetato-μ_2-1,1-azido-μ_2-(dimethyl sulfoxide)-copper(II)-di-μ_2-1,1-azido]

K. Li, F. Luo, X. Song and Z. Hu

Comment

The crystal structure and some properties of Cu(II)-azido coordination polymers have been previously reported (Goher *et al.*, 1999; Goher *et al.*, 2002; Liu *et al.*, 2007; Song *et al.*, 2007). We report here the synthesis and crystal structure of the title one-dimensional Copper(II)-azido coordination polymer(I) (Fig. 1). In (I) one unique Cu^{II} atom is six-coordinated while the other is is five-coordinated. Crystallographically independent Cu^{II} atoms are bridged by two O atoms from acetate ligands, one μ_2-O atom from a DMSO ligand and one μ_2-N atom of an azido ion, while the symmetry related Cu^{II} atoms are bridged by two μ_2-N atoms from two azido ligands to from a one-dimensional polymer. In the absence of any direction specific interactions the the crystal structure is stabilized by Van der Waals interactions.

Experimental

To a solution of $Cu(OAc)_2$(0.14 g, 0.6 mmol) in Dimethyl sulphoxide (3 ml) a suspension of sodium azide(0.039 g, 0.6 mmol) in the ethanol (15 ml) was added and the mixture was stirred for 2 h at 333 K. The solution was filtered after cooled and allowed to stand at room temperature without disturbing, black crystals of of the title compound were obtained after about 3 weeks.

Refinement

After being located in a difference map, all H-atoms were fixed geometrically at ideal positions and allowed to ride on the parent C atoms with C—H = 0.96Å and U_{iso}(H)= 1.5U_{eq}(C).

Figures

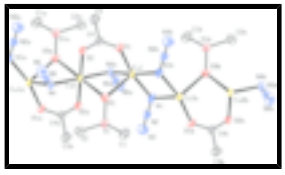


Fig. 1. Part of the one dimensional chain of the title compound showing 30% probability displacement ellipsoids. H atoms are not included [symmetry codes: (*a*) −*x* + 1, −*y* + 1, −*z*; (*b*) −*x* + 2, −*y* + 2, −*z*].

catena-Poly[copper(II)-di-μ_2-1,1-azido-copper(II)-μ_2-acetato- μ_2-1,1-azido-μ_2-(dimethyl sulfoxide)-copper(II)-di-μ_2-1,1-azido]

Crystal data

$[Cu_3(C_2H_3O_2)_2(N_3)_4(C_2H_6OS)_2]$ $Z = 1$

M_r = 633.13	F_{000} = 317
Triclinic, $P\bar{1}$	D_x = 1.855 Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 8.5205 (9) Å	Cell parameters from 2542 reflections
b = 8.6624 (9) Å	θ = 2.4–27.0º
c = 9.4999 (10) Å	μ = 3.02 mm^{-1}
α = 90.386 (2)º	T = 293 (2) K
β = 112.687 (1)º	Block, black
γ = 116.261 (1)º	0.30 × 0.20 × 0.20 mm
V = 566.66 (10) Å^3	

Data collection

Bruker SMART-CCD diffractometer	2182 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.060
Monochromator: graphite	θ_{max} = 27.0º
T = 293(2) K	θ_{min} = 2.4º
φ and ω scans	h = −10→7
Absorption correction: none	k = −7→11
3467 measured reflections	l = −10→12
2404 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.035	H-atom parameters constrained
$wR(F^2)$ = 0.099	$w = 1/[\sigma^2(F_o^2) + (0.052P)^2 + 0.3521P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.11	$(\Delta/\sigma)_{max} < 0.001$
2404 reflections	$\Delta\rho_{max}$ = 0.60 e Å^{-3}
145 parameters	$\Delta\rho_{min}$ = −0.65 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-

factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cu1	0.81687 (5)	0.81565 (4)	−0.06003 (4)	0.03025 (14)
Cu2	0.5000	0.5000	0.0000	0.03156 (15)
C1	0.7810 (8)	1.1353 (5)	0.2360 (6)	0.0673 (12)
H1A	0.8966	1.1839	0.2199	0.101*
H1B	0.8111	1.1893	0.3382	0.101*
H1C	0.6854	1.1577	0.1586	0.101*
C2	0.4761 (7)	0.8594 (7)	0.2395 (6)	0.0707 (13)
H2A	0.4049	0.9054	0.1643	0.106*
H2B	0.5099	0.9132	0.3429	0.106*
H2C	0.3974	0.7341	0.2218	0.106*
C3	0.2979 (6)	0.5365 (6)	−0.4771 (4)	0.0630 (12)
H3A	0.2879	0.4330	−0.5259	0.095*
H3B	0.3458	0.6318	−0.5264	0.095*
H3C	0.1722	0.5130	−0.4879	0.095*
C4	0.4350 (5)	0.5860 (4)	−0.3071 (4)	0.0361 (6)
N1	1.0712 (4)	0.9551 (4)	0.1239 (3)	0.0449 (7)
N2	1.1240 (4)	0.9351 (4)	0.2578 (3)	0.0421 (6)
N3	1.1756 (6)	0.9171 (6)	0.3806 (4)	0.0691 (11)
N4	0.2460 (4)	0.4183 (3)	0.0089 (3)	0.0328 (5)
N5	0.1232 (4)	0.4496 (4)	−0.0825 (3)	0.0379 (6)
N6	0.0095 (5)	0.4816 (5)	−0.1649 (5)	0.0624 (9)
O1	0.6009 (3)	0.7171 (3)	−0.2653 (3)	0.0432 (6)
O2	0.3755 (3)	0.4928 (3)	−0.2207 (3)	0.0406 (5)
O3	0.6177 (4)	0.8295 (3)	0.0499 (3)	0.0430 (5)
S1	0.68907 (12)	0.90584 (11)	0.22039 (10)	0.0401 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.0237 (2)	0.0250 (2)	0.0280 (2)	0.00378 (15)	0.00743 (15)	0.00848 (14)
Cu2	0.0225 (3)	0.0298 (3)	0.0332 (3)	0.0057 (2)	0.0115 (2)	0.0083 (2)
C1	0.080 (3)	0.0306 (19)	0.068 (3)	0.009 (2)	0.031 (2)	−0.0018 (18)
C2	0.057 (3)	0.079 (3)	0.062 (3)	0.014 (2)	0.035 (2)	−0.008 (2)
C3	0.046 (2)	0.071 (3)	0.0320 (18)	0.007 (2)	0.0030 (16)	0.0070 (17)
C4	0.0317 (15)	0.0350 (16)	0.0304 (14)	0.0103 (13)	0.0097 (12)	0.0041 (12)
N1	0.0338 (14)	0.0375 (15)	0.0345 (14)	0.0031 (12)	0.0040 (11)	0.0176 (11)
N2	0.0356 (14)	0.0370 (15)	0.0372 (15)	0.0090 (12)	0.0101 (12)	0.0122 (11)
N3	0.071 (2)	0.076 (3)	0.0393 (18)	0.026 (2)	0.0150 (17)	0.0256 (17)
N4	0.0251 (12)	0.0272 (12)	0.0428 (14)	0.0099 (10)	0.0148 (11)	0.0109 (10)
N5	0.0316 (13)	0.0317 (13)	0.0458 (15)	0.0109 (11)	0.0175 (12)	0.0118 (11)
N6	0.052 (2)	0.070 (2)	0.073 (2)	0.0381 (19)	0.0230 (18)	0.0342 (19)
O1	0.0344 (12)	0.0334 (11)	0.0305 (11)	−0.0018 (10)	0.0059 (9)	0.0056 (9)

O2	0.0282 (11)	0.0391 (12)	0.0337 (11)	0.0031 (10)	0.0094 (9)	0.0067 (9)
O3	0.0450 (13)	0.0400 (13)	0.0407 (12)	0.0177 (11)	0.0188 (11)	0.0008 (10)
S1	0.0395 (4)	0.0351 (4)	0.0384 (4)	0.0163 (4)	0.0118 (3)	0.0031 (3)

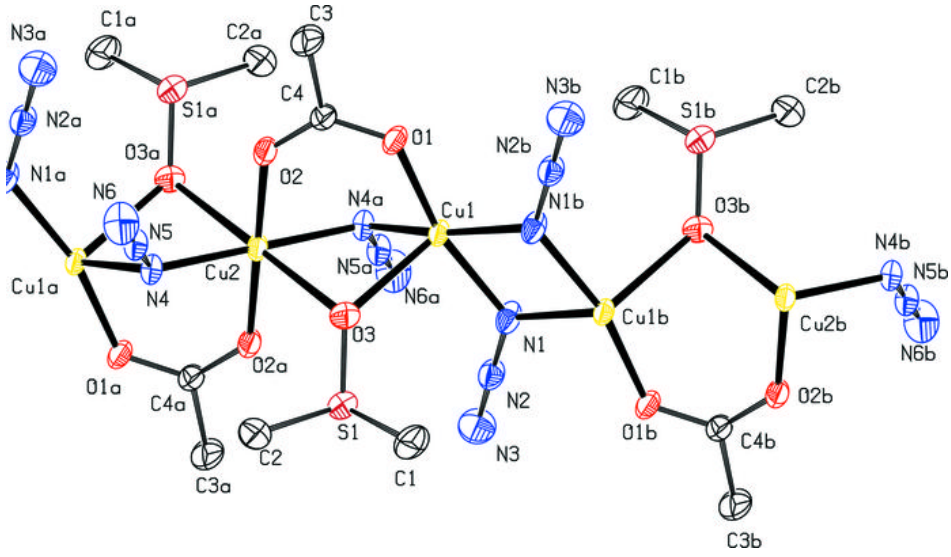
Geometric parameters (Å, °)

Cu1—O1	1.933 (2)	C2—H2B	0.9600
Cu1—N4^{i}	1.970 (2)	C2—H2C	0.9600
Cu1—N1	1.986 (3)	C3—C4	1.500 (4)
Cu1—N1^{ii}	2.002 (3)	C3—H3A	0.9600
Cu1—O3	2.351 (2)	C3—H3B	0.9600
Cu2—O2	1.936 (2)	C3—H3C	0.9600
Cu2—O2^{i}	1.936 (2)	C4—O2	1.245 (4)
Cu2—N4^{i}	1.995 (2)	C4—O1	1.259 (4)
Cu2—N4	1.995 (2)	N1—N2	1.217 (4)
Cu2—O3	2.542 (2)	N1—Cu1^{ii}	2.002 (3)
C1—S1	1.765 (4)	N2—N3	1.116 (4)
C1—H1A	0.9600	N4—N5	1.215 (4)
C1—H1B	0.9600	N4—Cu1^{i}	1.970 (2)
C1—H1C	0.9600	N5—N6	1.133 (4)
C2—S1	1.760 (5)	O3—S1	1.517 (2)
C2—H2A	0.9600		
O1—Cu1—N4^{i}	92.49 (11)	H2A—C2—H2C	109.5
O1—Cu1—N1	166.25 (11)	H2B—C2—H2C	109.5
N4^{i}—Cu1—N1	98.17 (11)	C4—C3—H3A	109.5
O1—Cu1—N1^{ii}	89.72 (10)	C4—C3—H3B	109.5
N4^{i}—Cu1—N1^{ii}	165.71 (13)	H3A—C3—H3B	109.5
N1—Cu1—N1^{ii}	77.84 (12)	C4—C3—H3C	109.5
O1—Cu1—O3	91.09 (10)	H3A—C3—H3C	109.5
N4^{i}—Cu1—O3	87.19 (10)	H3B—C3—H3C	109.5
N1—Cu1—O3	98.06 (12)	O2—C4—O1	126.0 (3)
N1^{ii}—Cu1—O3	106.90 (12)	O2—C4—C3	117.2 (3)
O2—Cu2—O2^{i}	180.00 (15)	O1—C4—C3	116.8 (3)
O2—Cu2—N4^{i}	90.66 (10)	N2—N1—Cu1	129.6 (2)
O2^{i}—Cu2—N4^{i}	89.34 (10)	N2—N1—Cu1^{ii}	124.8 (2)
O2—Cu2—N4	89.34 (10)	Cu1—N1—Cu1^{ii}	102.16 (11)
O2^{i}—Cu2—N4	90.66 (10)	N3—N2—N1	178.7 (4)
N4^{i}—Cu2—N4	180.0	N5—N4—Cu1^{i}	122.3 (2)
S1—C1—H1A	109.5	N5—N4—Cu2	120.7 (2)
S1—C1—H1B	109.5	Cu1^{i}—N4—Cu2	104.04 (11)
H1A—C1—H1B	109.5	N6—N5—N4	178.3 (4)
S1—C1—H1C	109.5	C4—O1—Cu1	128.8 (2)
H1A—C1—H1C	109.5	C4—O2—Cu2	131.9 (2)
H1B—C1—H1C	109.5	S1—O3—Cu1	124.36 (14)
S1—C2—H2A	109.5	O3—S1—C2	104.30 (19)

S1—C2—H2B	109.5	O3—S1—C1	105.62 (19)
H2A—C2—H2B	109.5	C2—S1—C1	97.6 (3)
S1—C2—H2C	109.5		
O1—Cu1—N1—N2	−175.2 (4)	$N4^{i}$—Cu1—O1—C4	35.2 (3)
$N4^{i}$—Cu1—N1—N2	−34.7 (4)	N1—Cu1—O1—C4	176.1 (5)
$N1^{ii}$—Cu1—N1—N2	159.2 (5)	$N1^{ii}$—Cu1—O1—C4	−158.9 (3)
O3—Cu1—N1—N2	53.6 (4)	O3—Cu1—O1—C4	−52.0 (3)
O1—Cu1—N1—$Cu1^{ii}$	25.6 (6)	O1—C4—O2—Cu2	−1.5 (6)
$N4^{i}$—Cu1—N1—$Cu1^{ii}$	166.06 (14)	C3—C4—O2—Cu2	177.6 (3)
$N1^{ii}$—Cu1—N1—$Cu1^{ii}$	0.0	$N4^{i}$—Cu2—O2—C4	−34.0 (3)
O3—Cu1—N1—$Cu1^{ii}$	−105.65 (14)	N4—Cu2—O2—C4	146.0 (3)
O2—Cu2—N4—N5	−27.1 (3)	O1—Cu1—O3—S1	−177.11 (17)
$O2^{i}$—Cu2—N4—N5	152.9 (3)	$N4^{i}$—Cu1—O3—S1	90.44 (18)
O2—Cu2—N4—$Cu1^{i}$	114.98 (12)	N1—Cu1—O3—S1	−7.41 (19)
$O2^{i}$—Cu2—N4—$Cu1^{i}$	−65.02 (12)	$N1^{ii}$—Cu1—O3—S1	−87.08 (18)
O2—C4—O1—Cu1	1.1 (5)	Cu1—O3—S1—C2	−173.7 (2)
C3—C4—O1—Cu1	−178.1 (3)	Cu1—O3—S1—C1	83.9 (2)

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*; (ii) −*x*+2, −*y*+2, −*z*.

Fig. 1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C2a
C3
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O3a
O2
O1
N3b
C1b
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N1a
N6
N4a
Cu1
N2b
N1b
O3b
N5
N5a
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N5b
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Cu2
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N6a
O3
N1
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C1
C3b