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Bis(2-aminopyridine-κN^1)bis(*N*,*N*′-dimethylformamide-κO)(sulfato-$\kappa^2 O,O'$)-copper(II)

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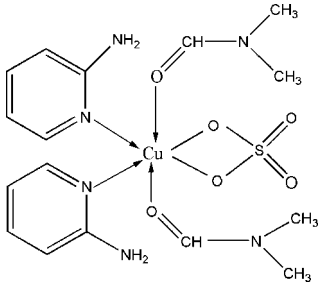
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.028; wR factor = 0.079; data-to-parameter ratio = 19.2.

In the title compound, $[Cu(SO_4)(C_5H_6N_2)_2(C_3H_7NO)_2]$, the coordination geometry around the Cu^{II} atom is distorted octahedral, involving two O atoms from a bidentate sulfate anion, two N atoms from two 2-aminopyridine ligands and two O atoms from two *N*,*N*′-dimethylformamide molecules. The Cu and S atoms are located on a crystallographic twofold rotation axis. A one-dimensional chain structure is built *via* hydrogen bonds.

Related literature

The bond distances are similar to those reported in the literature (Hagrman *et al.*, 1998).



Experimental

Crystal data

$[Cu(SO_4)(C_5H_6N_2)_2(C_3H_7NO)_2]$
M_r = 494.03
Monoclinic, $C2/c$
a = 11.528 (5) Å
b = 18.156 (5) Å
c = 10.828 (5) Å
β = 103.801 (5)°
V = 2200.9 (15) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.13 mm^{-1}
T = 293 (2) K
0.19 × 0.15 × 0.12 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.814, T_{max} = 0.877
6718 measured reflections
2666 independent reflections
2222 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.079
S = 1.05
2666 reflections
139 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—Cu1	1.9972 (14)	O3—Cu1	2.589 (2)
O1—Cu1	2.0056 (13)		
O3—Cu1—N1	86.89 (6)	N1^i—Cu1—N1	95.40 (8)
O3—Cu1—N1^i	93.10 (6)	N1^i—Cu1—O1	167.79 (5)
O3—Cu1—O1	87.47 (6)	N1—Cu1—O1	96.81 (6)
O3—Cu1—O1^i	92.47 (5)	O1—Cu1—O1^i	70.97 (7)
O3—Cu1—O3^i	179.99 (6)		

Symmetry code: (i) $-x, y, -z+\frac{3}{2}$.

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O1	0.86	2.55	3.087 (2)	121
N2—H2A···O3^i	0.86	2.28	2.955 (2)	135
N2—H2B···O2^{ii}	0.86	2.19	3.029 (2)	164

Symmetry codes: (i) $-x, y, -z+\frac{3}{2}$; (ii) $-x+\frac{1}{2}, -y+\frac{1}{2}, -z+2$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1990); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HY2071).

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supplementary materials

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Bis(2-aminopyridine-κN^1)bis(*N*,*N*'-dimethylformamide-κO)(sulfato-$\kappa^2 O,O'$)copper(II)

G.-J. Li, Y. Xing and S.-Y. Song

Comment

The title compound was obtained as the product of an attempted synthesis of an open-framework compound of copper(II) using 2-aminopyridine as structure-directing agent.

As shown in Fig. 1, the coordination polyhedron around the Cu^{II} atom is a distorted octahedron, comprising two O atoms from a bidentate sulfate anion, two N atoms from two 2-aminopyridine molecules and two O atoms from two *N*,*N*'-dimethylformamide (DMF) molecules. The bond distances are similar to those in the literature (Hagrman *et al.*, 1998). There is a weak Cu1—O3 interaction of 2.589 (2)Å (Table 1). The molecules are connected by intermolecular hydrogen-bonding interactions (Table 2), resulting in a one-dimentional supramolecular structure (Fig. 2).

Experimental

Typically, $CuSO_4{\cdot}5H_2O$ (0.125 g, 0.5 mmol) was dispersed into DMF (10 ml) with stirring, then 2-aminopyridine (0.285 g, 0.3 mmol) was added to the above reaction mixture with stirring. The mixture with pH value of 5 was transferred into a 18 ml Teflon-lined stainless steel autoclave and heated at 353 K for 3 d. After cooling to room temperature, green block-shaped crystals of the title compound were obtained; they were filtered and washed thoroughly with deionized water and dried at room temperature.

Refinement

All H atoms were positioned geometrically and refined as riding atoms, with C—H = 0.93Å and U_{iso}(H) = 1.2U_{eq}(C,*N*), and U_{iso}(H) = 1.5U_{eq}(C) for CH_3 groups.

Figures

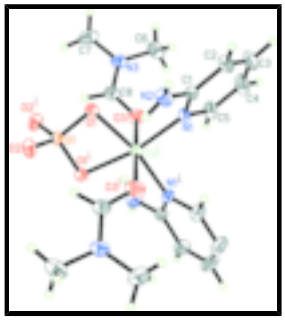

Fig. 1. The molecular structure of the title compound. Displacement ellipsoids are drawn at the 30% probability level. [Symmetry code: (i) −*x*, *y*, 3/2 − *z*.]

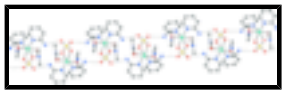

Fig. 2. One-dimensional structure built up *via* hydrogen-bonding interactions in the title compound.

Bis(2-aminopyridine-κN^1)bis(*N*,*N*'-dimethylformamide-κ*O*)(sulfato- κ^2*O*,*O*')copper(II)

Crystal data

$[Cu(SO_4)(C_5H_6N_2)_2(C_3H_7NO)_2]$	F_{000} = 1028
M_r = 494.03	D_x = 1.491 Mg m^{-3}
Monoclinic, *C*2/*c*	Melting point: not measured K
Hall symbol: -C 2yc	Mo *K*α radiation λ = 0.71073 Å
a = 11.528 (5) Å	Cell parameters from 2666 reflections
b = 18.156 (5) Å	θ = 2.1–28.3º
c = 10.828 (5) Å	μ = 1.13 mm^{-1}
β = 103.801 (5)º	*T* = 293 (2) K
V = 2200.9 (15) Å^3	Block, blue
Z = 4	0.19 × 0.15 × 0.12 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer	2666 independent reflections
Radiation source: fine-focus sealed tube	2222 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.023
T = 293(2) K	θ_{max} = 28.3º
φ and ω scans	θ_{min} = 2.1º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	*h* = −15→10
T_{min} = 0.814, T_{max} = 0.877	*k* = −23→24
6718 measured reflections	*l* = −14→13

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.028	H-atom parameters constrained
$wR(F^2)$ = 0.079	$w = 1/[\sigma^2(F_o^2) + (0.0417P)^2 + 0.588P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.05	$(\Delta/\sigma)_{max} < 0.001$
2666 reflections	$\Delta\rho_{max}$ = 0.24 e Å^{-3}
139 parameters	$\Delta\rho_{min}$ = −0.28 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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

	x	*y*	*z*	U_{iso}*/U_{eq}

C1	0.23305 (15)	0.10448 (9)	0.86610 (17)	0.0350 (4)
C2	0.32604 (18)	0.05410 (12)	0.8644 (2)	0.0531 (5)
H2	0.3957	0.0550	0.9289	0.064*
C3	0.3138 (2)	0.00471 (13)	0.7693 (3)	0.0659 (7)
H3	0.3755	−0.0278	0.7670	0.079*
C4	0.2086 (2)	0.00265 (12)	0.6749 (2)	0.0636 (6)
H4	0.1983	−0.0316	0.6093	0.076*
C5	0.12116 (19)	0.05163 (10)	0.68049 (18)	0.0461 (4)
H5	0.0505	0.0499	0.6175	0.055*
C6	0.1553 (2)	0.15450 (15)	0.3507 (2)	0.0672 (6)
H6A	0.1303	0.1484	0.2630	0.101*
H6B	0.1240	0.1169	0.3912	0.101*
H6C	0.2383	0.1530	0.3750	0.101*
C7	0.1560 (2)	0.29009 (15)	0.3324 (2)	0.0623 (6)
H7A	0.1346	0.2874	0.2441	0.093*
H7B	0.2387	0.2933	0.3600	0.093*
H7C	0.1213	0.3316	0.3592	0.093*
C8	0.04146 (17)	0.22969 (12)	0.46322 (18)	0.0437 (4)
H8	0.0197	0.2768	0.4830	0.052*
N1	0.13161 (12)	0.10318 (7)	0.77361 (13)	0.0326 (3)
N2	0.24550 (13)	0.15442 (9)	0.95966 (16)	0.0451 (4)
H2A	0.1890	0.1852	0.9602	0.054*
H2B	0.3100	0.1556	1.0191	0.054*
N3	0.11426 (14)	0.22427 (10)	0.38583 (15)	0.0461 (4)
O1	0.10372 (10)	0.26717 (6)	0.76849 (12)	0.0368 (3)
O2	0.01014 (11)	0.36464 (7)	0.86254 (12)	0.0442 (3)
O3	0.00034 (14)	0.17725 (8)	0.51108 (14)	0.0532 (4)
S1	0.0000	0.31992 (3)	0.7500	0.02985 (13)
Cu1	0.0000	0.177216 (14)	0.7500	0.03053 (10)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0330 (8)	0.0314 (8)	0.0415 (9)	0.0056 (7)	0.0110 (7)	0.0099 (7)
C2	0.0419 (10)	0.0495 (12)	0.0673 (14)	0.0179 (9)	0.0121 (10)	0.0141 (10)
C3	0.0659 (15)	0.0486 (12)	0.0899 (18)	0.0290 (11)	0.0316 (13)	0.0091 (12)
C4	0.0897 (17)	0.0407 (11)	0.0663 (15)	0.0180 (12)	0.0302 (14)	−0.0085 (10)
C5	0.0590 (11)	0.0357 (9)	0.0436 (10)	0.0062 (8)	0.0124 (9)	−0.0049 (8)
C6	0.0670 (15)	0.0732 (16)	0.0676 (16)	0.0154 (13)	0.0286 (13)	−0.0051 (12)
C7	0.0502 (12)	0.0835 (17)	0.0540 (13)	−0.0139 (12)	0.0139 (10)	0.0161 (12)
C8	0.0419 (10)	0.0525 (11)	0.0377 (10)	0.0039 (9)	0.0117 (8)	0.0025 (8)
N1	0.0354 (7)	0.0274 (7)	0.0354 (7)	0.0047 (5)	0.0090 (6)	0.0014 (5)
N2	0.0373 (8)	0.0498 (9)	0.0421 (8)	0.0074 (7)	−0.0023 (7)	−0.0039 (7)
N3	0.0438 (9)	0.0569 (10)	0.0407 (9)	0.0010 (8)	0.0165 (7)	0.0055 (7)
O1	0.0250 (5)	0.0303 (6)	0.0537 (8)	0.0004 (4)	0.0064 (5)	−0.0001 (5)
O2	0.0506 (7)	0.0354 (7)	0.0422 (7)	0.0043 (6)	0.0022 (6)	−0.0077 (5)
O3	0.0549 (8)	0.0605 (9)	0.0497 (8)	−0.0010 (7)	0.0234 (7)	0.0091 (6)
S1	0.0272 (3)	0.0246 (3)	0.0349 (3)	0.000	0.0019 (2)	0.000

Cu1	0.02611 (15)	0.02407 (15)	0.03997 (18)	0.000	0.00507 (12)	0.000

Geometric parameters (Å, °)

C1—N2	1.342 (2)	C7—H7B	0.9300
C1—N1	1.346 (2)	C7—H7C	0.9300
C1—C2	1.413 (2)	C8—O3	1.232 (2)
C2—C3	1.347 (3)	C8—N3	1.324 (2)
C2—H2	0.9300	C8—H8	0.9300
C3—C4	1.388 (4)	N1—Cu1	1.9972 (14)
C3—H3	0.9300	N2—H2A	0.8600
C4—C5	1.356 (3)	N2—H2B	0.8600
C4—H4	0.9300	O1—S1	1.5076 (13)
C5—N1	1.360 (2)	O1—Cu1	2.0056 (13)
C5—H5	0.9300	O2—S1	1.4457 (13)
C6—N3	1.435 (3)	O3—Cu1	2.589 (2)
C6—H6A	0.9300	S1—O2^{i}	1.4457 (13)
C6—H6B	0.9300	S1—O1^{i}	1.5076 (13)
C6—H6C	0.9300	Cu1—N1^{i}	1.9972 (14)
C7—N3	1.458 (3)	Cu1—O1^{i}	2.0056 (13)
C7—H7A	0.9300	Cu1—O3^{i}	2.589 (2)
N2—C1—N1	119.40 (15)	O3^{i}—Cu1—N1	93.10 (6)
N2—C1—C2	120.18 (17)	O3^{i}—Cu1—N1^{i}	86.89 (6)
N1—C1—C2	120.42 (17)	O3—Cu1—O1	87.47 (6)
C3—C2—C1	120.0 (2)	O3^{i}—Cu1—O1	92.47 (5)
C3—C2—H2	120.0	O3—Cu1—O1^{i}	92.47 (5)
C1—C2—H2	120.0	O3—Cu1—O3^{i}	179.99 (6)
C2—C3—C4	119.6 (2)	O3^{i}—Cu1—O1^{i}	87.47 (6)
C2—C3—H3	120.2	N3—C8—H8	117.5
C4—C3—H3	120.2	C1—N1—C5	118.11 (15)
C5—C4—C3	118.5 (2)	C1—N1—Cu1	125.59 (11)
C5—C4—H4	120.7	C5—N1—Cu1	115.99 (12)
C3—C4—H4	120.7	C1—N2—H2A	120.0
N1—C5—C4	123.3 (2)	C1—N2—H2B	120.0
N1—C5—H5	118.4	H2A—N2—H2B	120.0
C4—C5—H5	118.4	C8—N3—C6	122.20 (19)
N3—C6—H6A	109.5	C8—N3—C7	120.6 (2)
N3—C6—H6B	109.5	C6—N3—C7	117.20 (19)
H6A—C6—H6B	109.5	S1—O1—Cu1	93.96 (6)
N3—C6—H6C	109.5	O2—S1—O2^{i}	111.66 (11)
H6A—C6—H6C	109.5	O2—S1—O1	110.05 (7)
H6B—C6—H6C	109.5	O2^{i}—S1—O1	111.77 (7)
N3—C7—H7A	109.5	O2—S1—O1^{i}	111.77 (7)
N3—C7—H7B	109.5	O2^{i}—S1—O1^{i}	110.05 (7)
H7A—C7—H7B	109.5	O1—S1—O1^{i}	101.11 (10)

N3—C7—H7C	109.5	$N1^{i}$—Cu1—N1	95.40 (8)
H7A—C7—H7C	109.5	$N1^{i}$—Cu1—O1	167.79 (5)
H7B—C7—H7C	109.5	N1—Cu1—O1	96.81 (6)
O3—C8—N3	125.1 (2)	$N1^{i}$—Cu1—$O1^{i}$	96.81 (6)
O3—C8—H8	117.5	N1—Cu1—$O1^{i}$	167.79 (5)
O3—Cu1—N1	86.89 (6)	O1—Cu1—$O1^{i}$	70.97 (7)
O3—Cu1—$N1^{i}$	93.10 (6)		
N2—C1—C2—C3	−179.1 (2)	C5—N1—Cu1—$O1^{i}$	−126.5 (2)
N1—C1—C2—C3	0.5 (3)	C1—N1—Cu1—S1	47.13 (15)
C1—C2—C3—C4	−1.4 (4)	C5—N1—Cu1—S1	−126.28 (12)
C2—C3—C4—C5	0.9 (4)	S1—O1—Cu1—$N1^{i}$	0.2 (3)
C3—C4—C5—N1	0.5 (3)	S1—O1—Cu1—N1	−179.95 (6)
N2—C1—N1—C5	−179.56 (16)	S1—O1—Cu1—$O1^{i}$	0.0
C2—C1—N1—C5	0.9 (2)	O2—S1—Cu1—$N1^{i}$	91.32 (8)
N2—C1—N1—Cu1	7.2 (2)	$O2^{i}$—S1—Cu1—$N1^{i}$	−88.68 (8)
C2—C1—N1—Cu1	−172.40 (13)	O1—S1—Cu1—$N1^{i}$	−179.94 (8)
C4—C5—N1—C1	−1.4 (3)	$O1^{i}$—S1—Cu1—$N1^{i}$	0.06 (8)
C4—C5—N1—Cu1	172.55 (17)	O2—S1—Cu1—N1	−88.68 (8)
O3—C8—N3—C6	−1.1 (3)	$O2^{i}$—S1—Cu1—N1	91.32 (8)
O3—C8—N3—C7	178.8 (2)	O1—S1—Cu1—N1	0.06 (8)
Cu1—O1—S1—O2	118.29 (7)	$O1^{i}$—S1—Cu1—N1	−179.94 (8)
Cu1—O1—S1—$O2^{i}$	−117.04 (7)	O2—S1—Cu1—O1	−88.75 (9)
Cu1—O1—S1—$O1^{i}$	0.0	$O2^{i}$—S1—Cu1—O1	91.25 (9)
C1—N1—Cu1—$N1^{i}$	−132.87 (15)	$O1^{i}$—S1—Cu1—O1	180.0
C5—N1—Cu1—$N1^{i}$	53.72 (12)	O2—S1—Cu1—$O1^{i}$	91.25 (9)
C1—N1—Cu1—O1	47.16 (14)	$O2^{i}$—S1—Cu1—$O1^{i}$	−88.75 (9)
C5—N1—Cu1—O1	−126.24 (13)	O1—S1—Cu1—$O1^{i}$	180.0
C1—N1—Cu1—$O1^{i}$	47.0 (3)		

Symmetry codes: (i) −*x*, *y*, −*z*+3/2.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2A···O1	0.86	2.55	3.087 (2)	121
N2—H2A···$O3^{i}$	0.86	2.28	2.955 (2)	135
N2—H2B···$O2^{ii}$	0.86	2.19	3.029 (2)	164

Symmetry codes: (i) −*x*, *y*, −*z*+3/2; (ii) −*x*+1/2, −*y*+1/2, −*z*+2.

Fig. 1

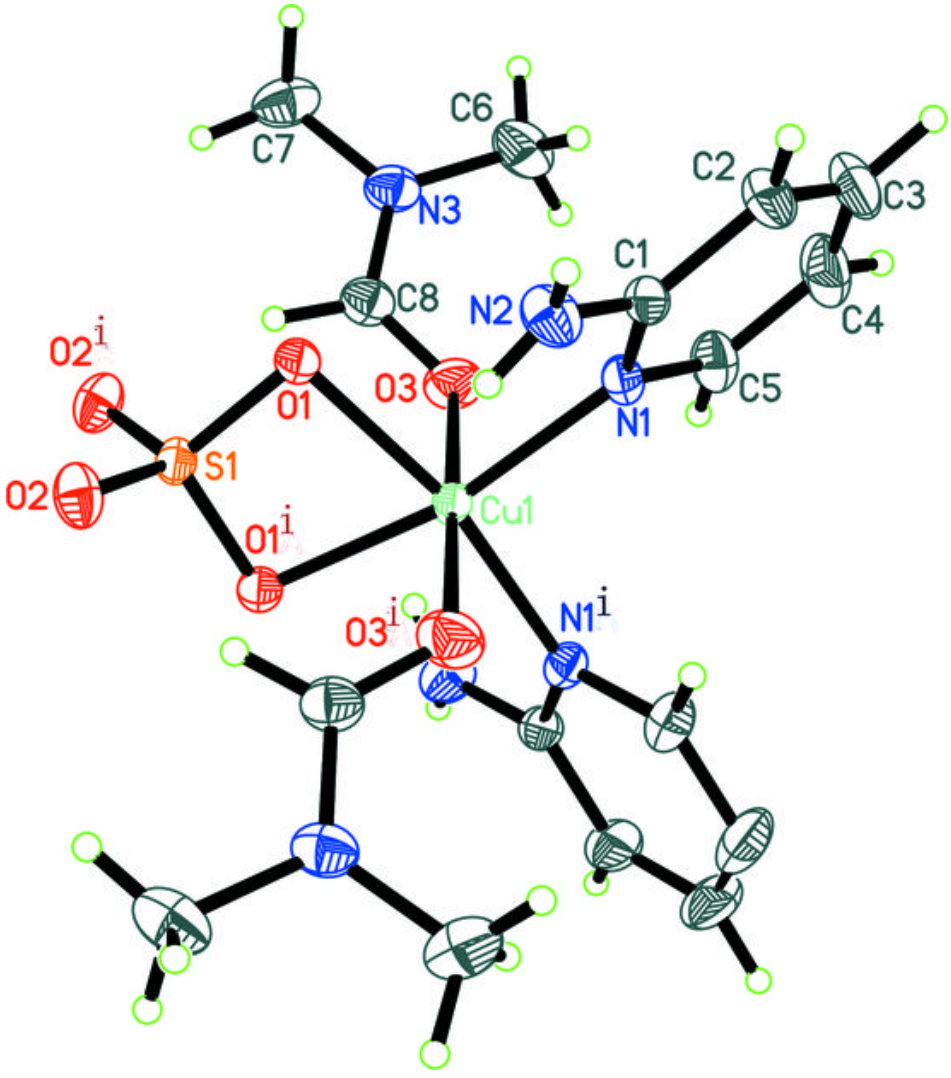

Fig. 2