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Diaquabis[(2-nitrophenylsulfinyl)acetato-κO]copper(II) tetrahydrate

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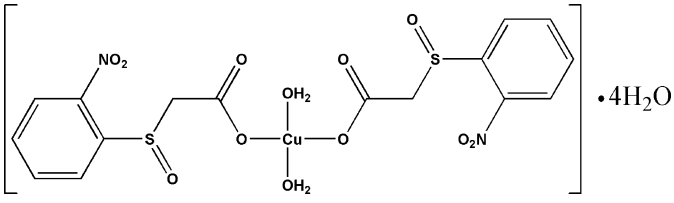
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.039; wR factor = 0.115; data-to-parameter ratio = 16.3.

The centrosymmetric title compound, $[Cu(C_8H_6NO_5S)_2(H_2O)_2]\cdot 4H_2O$, has a four-coordinate Cu^{II} ion in a square-planar geometry defined by two carboxylate O atoms from two (2-nitrophenylsulfinyl)acetate groups and two O atoms from two water molecules. The molecules are linked together by intermolecular hydrogen bonds involving the water molecules, resulting in a layer network.

Related literature

For the structure of the parent carboxylic acid, see: Ma (2007). For synthesis of the parent carboxylic acid, see: Nobles & Thompson (1965).



Experimental

Crystal data

$[Cu(C_8H_6NO_5S)_2(H_2O)_2]\cdot 4H_2O$
M_r = 628.03
Monoclinic, $P2_1/c$
a = 15.577 (3) Å
b = 5.3724 (11) Å
c = 14.740 (3) Å
β = 99.79 (3)°
V = 1215.6 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.15 mm^{-1}
T = 293 (2) K
0.28 × 0.25 × 0.22 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.741, T_{max} = 0.783
10697 measured reflections
2762 independent reflections
2319 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.115
S = 1.13
2762 reflections
169 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.56 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O7—H9···O5^{i}	0.85	1.85	2.699 (3)	175
O7—H10···O3^{ii}	0.85	2.00	2.779 (3)	152
O8—H11···O4^{iii}	0.85	2.15	2.982 (3)	166
O8—H12···O3	0.85	2.10	2.933 (3)	167
O6—H7···O8	0.85	1.84	2.686 (3)	172
O6—H8···O7^{iv}	0.85	1.85	2.680 (3)	165

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

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); 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: NG2272).

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supplementary materials

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Diaquabis[(2-nitrophenylsulfinyl)acetato-*κO*]copper(II) tetrahydrate

X.-F. Zhu, L.-M. Zhao, G.-F. Hou and J.-S. Gao

Comment

The crystal structure of (2-nitrophenylsulfinyl)acetic acid has been reported (Ma, 2007). Here, we report the structure of the copper derivative. The compound has the copper center being coordinated to two (2-nitrophenylsulfinyl)acetic acid groups and two water molecules in a square-planar geometry. The structure is stabilized by hydrogen bonding interactions (Table 1) that link the molecules into a layer structure (Fig. 2).

Experimental

(2-Nitrophenylsulfanyl)acetic acid was prepared by nucleophilic reaction of chloroacetic acid and 2-nitrothiophenol under basic conditions (Nobles & Thompson, 1965). It was then oxidized using 30% aqueous hydrogen peroxide in acetic anhydride solution to produce (2-nitrophenylsulfinyl)acetic acid. Copper(II) nitrate trihydrate (0.482 g, 2 mmol) and (2-nitrophenylsulfinyl)acetic acid (0.458 g, 2 mmol) were dissolved in water and the pH was adjusted to 6 with 0.01 *M* sodium hydroxide; green crystals separated from the filtered solution after several days.

Refinement

H atoms bound to C atoms were placed in calculated positions and treated as riding on their parent atoms, with C—H = 0.93 Å (aromatic C) or C—H = 0.97 Å (methylene C), and with U_{iso}(H) = 1.2Ueq(C). Water H atoms were initially located in a difference Fourier map but they were treated as riding on their parent atoms with O—H = 0.85 Å and with U_{iso}(H) = 1.5*U*eq(O).

Figures

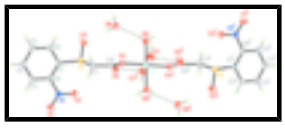

Fig. 1. **Figure 1**. The molecular structure of (I), showing displacement ellipsoids at the 30% probability level for non-H atoms·Dashed lines indicate the hydrogen bonding interactions.

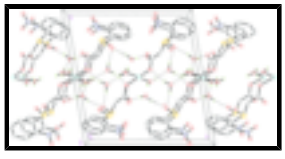

Fig. 2. **Figure 2**. A partial packing view,showing the two-dimensional hydrogen-bonding plan·Dashed lines indicate the hydrogen-bonding interactions. H atoms not involved in hydrogen bonds have been omitted.

Diaquabis[(2-nitrophenylsulfinyl)acetato-*κO*]copper(II) tetrahydrate

Crystal data

$[Cu(C_8H_6NO_5S)_2(H_2O)_2]\cdot 4H_2O$ $F_{000} = 646$

M_r = 628.03	D_x = 1.716 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 8753 reflections
a = 15.577 (3) Å	θ = 6.4–54.9º
b = 5.3724 (11) Å	μ = 1.15 mm^{-1}
c = 14.740 (3) Å	T = 293 (2) K
β = 99.79 (3)º	Block, green
V = 1215.6 (4) Å^3	0.28 × 0.25 × 0.22 mm
Z = 2	

Data collection

Rigaku R-AXIS RAPID diffractometer	2762 independent reflections
Radiation source: fine-focus sealed tube	2319 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.034
T = 293(2) K	θ_{max} = 27.5º
ω scans	θ_{min} = 3.3º
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	h = −20→16
T_{min} = 0.741, T_{max} = 0.783	k = −6→6
10697 measured reflections	l = −19→19

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.039	H-atom parameters constrained
$wR(F^2)$ = 0.115	$w = 1/[\sigma^2(F_o^2) + (0.0607P)^2 + 0.3938P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.13	$(\Delta/\sigma)_{max}$ = 0.001
2762 reflections	$\Delta\rho_{max}$ = 0.56 e Å^{-3}
169 parameters	$\Delta\rho_{min}$ = −0.32 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}
O7	0.64039 (15)	0.3858 (5)	0.97093 (15)	0.0651 (7)
H9	0.6487	0.2770	1.0134	0.098*
H10	0.6754	0.3607	0.9335	0.098*
C1	0.17597 (14)	0.3739 (4)	0.65632 (15)	0.0305 (5)
C2	0.19283 (17)	0.3735 (5)	0.56705 (17)	0.0403 (6)
H1	0.2324	0.2600	0.5503	0.048*
C3	0.1514 (2)	0.5401 (6)	0.50240 (18)	0.0460 (7)
H2	0.1642	0.5400	0.4430	0.055*
C4	0.09128 (18)	0.7062 (6)	0.52569 (17)	0.0453 (6)
H3	0.0630	0.8159	0.4817	0.054*
C5	0.07302 (16)	0.7100 (5)	0.61371 (17)	0.0398 (6)
H4	0.0324	0.8216	0.6296	0.048*
C6	0.11567 (14)	0.5465 (4)	0.67806 (15)	0.0296 (5)
C7	0.31452 (16)	0.3077 (5)	0.79487 (17)	0.0359 (5)
H5	0.3533	0.3608	0.7536	0.043*
H6	0.2927	0.4545	0.8219	0.043*
C8	0.36275 (16)	0.1415 (5)	0.86970 (16)	0.0360 (5)
Cu1	0.5000	0.0000	1.0000	0.03945 (17)
N1	0.09619 (14)	0.5590 (4)	0.77139 (14)	0.0347 (4)
O1	0.03918 (14)	0.6992 (4)	0.78782 (13)	0.0532 (5)
O2	0.13813 (14)	0.4272 (4)	0.83009 (13)	0.0521 (5)
O3	0.26962 (14)	−0.0378 (4)	0.67413 (15)	0.0488 (5)
O4	0.44160 (12)	0.2006 (3)	0.89878 (11)	0.0410 (4)
O6	0.53552 (14)	−0.2293 (4)	0.91383 (13)	0.0541 (6)
H7	0.5101	−0.2419	0.8582	0.081*
H8	0.5606	−0.3657	0.9315	0.081*
O5	0.32407 (15)	−0.0344 (4)	0.89713 (17)	0.0638 (7)
S1	0.22542 (4)	0.13019 (11)	0.73286 (4)	0.03311 (17)
O8	0.44357 (13)	−0.2394 (4)	0.74232 (13)	0.0526 (5)
H11	0.4682	−0.2654	0.6960	0.079*
H12	0.3902	−0.2035	0.7249	0.079*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O7	0.0726 (15)	0.0724 (15)	0.0554 (13)	0.0386 (13)	0.0251 (11)	0.0294 (12)
C1	0.0249 (11)	0.0369 (12)	0.0283 (11)	−0.0008 (9)	0.0010 (8)	0.0029 (9)
C2	0.0381 (14)	0.0523 (16)	0.0314 (12)	0.0075 (11)	0.0085 (10)	−0.0001 (11)
C3	0.0506 (17)	0.0626 (18)	0.0251 (12)	0.0017 (13)	0.0077 (11)	0.0043 (12)
C4	0.0445 (15)	0.0592 (17)	0.0307 (12)	0.0109 (12)	0.0021 (10)	0.0119 (12)
C5	0.0352 (13)	0.0493 (15)	0.0344 (12)	0.0090 (11)	0.0046 (10)	0.0049 (11)
C6	0.0250 (11)	0.0377 (12)	0.0256 (10)	−0.0025 (9)	0.0035 (8)	0.0003 (9)

C7	0.0338 (13)	0.0361 (12)	0.0355 (12)	0.0009 (9)	−0.0004 (9)	0.0074 (10)
C8	0.0390 (14)	0.0407 (13)	0.0289 (11)	0.0124 (10)	0.0077 (10)	0.0051 (10)
Cu1	0.0463 (3)	0.0408 (3)	0.0267 (2)	0.01786 (18)	−0.00671 (18)	−0.00255 (17)
N1	0.0349 (11)	0.0406 (11)	0.0294 (10)	−0.0004 (9)	0.0077 (8)	0.0003 (9)
O1	0.0613 (13)	0.0607 (12)	0.0415 (10)	0.0239 (10)	0.0198 (9)	0.0025 (10)
O2	0.0545 (12)	0.0721 (13)	0.0304 (9)	0.0210 (11)	0.0093 (8)	0.0120 (10)
O3	0.0531 (13)	0.0426 (10)	0.0509 (12)	0.0120 (8)	0.0092 (9)	−0.0051 (9)
O4	0.0421 (10)	0.0449 (10)	0.0319 (9)	0.0126 (8)	−0.0055 (7)	0.0016 (8)
O6	0.0738 (15)	0.0493 (11)	0.0339 (9)	0.0295 (10)	−0.0057 (9)	−0.0029 (9)
O5	0.0549 (14)	0.0691 (14)	0.0693 (15)	0.0095 (11)	0.0161 (11)	0.0429 (12)
S1	0.0312 (3)	0.0326 (3)	0.0356 (3)	−0.0001 (2)	0.0057 (2)	0.0047 (2)
O8	0.0520 (12)	0.0708 (14)	0.0353 (9)	0.0081 (10)	0.0080 (8)	−0.0014 (10)

Geometric parameters (Å, °)

O7—H9	0.8500	C7—H5	0.9700
O7—H10	0.8500	C7—H6	0.9700
C1—C2	1.385 (3)	C8—O5	1.226 (3)
C1—C6	1.396 (3)	C8—O4	1.271 (3)
C1—S1	1.812 (2)	Cu1—O6^{i}	1.9173 (19)
C2—C3	1.384 (4)	Cu1—O6	1.9173 (19)
C2—H1	0.9300	Cu1—O4	1.9373 (17)
C3—C4	1.379 (4)	Cu1—O4^{i}	1.9373 (17)
C3—H2	0.9300	N1—O2	1.218 (3)
C4—C5	1.375 (4)	N1—O1	1.219 (3)
C4—H3	0.9300	O3—S1	1.497 (2)
C5—C6	1.378 (3)	O6—H7	0.8499
C5—H4	0.9300	O6—H8	0.8500
C6—N1	1.460 (3)	O8—H11	0.8500
C7—C8	1.515 (3)	O8—H12	0.8501
C7—S1	1.800 (2)		
H9—O7—H10	109.6	S1—C7—H6	110.2
C2—C1—C6	117.6 (2)	H5—C7—H6	108.5
C2—C1—S1	117.47 (19)	O5—C8—O4	125.6 (2)
C6—C1—S1	124.64 (17)	O5—C8—C7	118.9 (2)
C3—C2—C1	120.8 (2)	O4—C8—C7	115.4 (2)
C3—C2—H1	119.6	O6^{i}—Cu1—O6	180.000 (1)
C1—C2—H1	119.6	O6^{i}—Cu1—O4	90.12 (8)
C4—C3—C2	120.2 (2)	O6—Cu1—O4	89.88 (8)
C4—C3—H2	119.9	O6^{i}—Cu1—O4^{i}	89.88 (8)
C2—C3—H2	119.9	O6—Cu1—O4^{i}	90.12 (8)
C5—C4—C3	120.2 (2)	O4—Cu1—O4^{i}	180.00 (7)
C5—C4—H3	119.9	O2—N1—O1	122.6 (2)
C3—C4—H3	119.9	O2—N1—C6	118.0 (2)
C4—C5—C6	119.1 (2)	O1—N1—C6	119.4 (2)
C4—C5—H4	120.4	C8—O4—Cu1	114.91 (16)
C6—C5—H4	120.4	Cu1—O6—H7	123.4

C5—C6—C1	122.0 (2)	Cu1—O6—H8	121.4
C5—C6—N1	117.8 (2)	H7—O6—H8	109.9
C1—C6—N1	120.2 (2)	O3—S1—C7	102.84 (12)
C8—C7—S1	107.53 (17)	O3—S1—C1	105.31 (11)
C8—C7—H5	110.2	C7—S1—C1	98.20 (11)
S1—C7—H5	110.2	H11—O8—H12	110.3
C8—C7—H6	110.2		

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O7—H9···O5^{i}	0.85	1.85	2.699 (3)	175
O7—H10···O3^{ii}	0.85	2.00	2.779 (3)	152
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O6—H8···O7^{iv}	0.85	1.85	2.680 (3)	165

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

Fig. 1

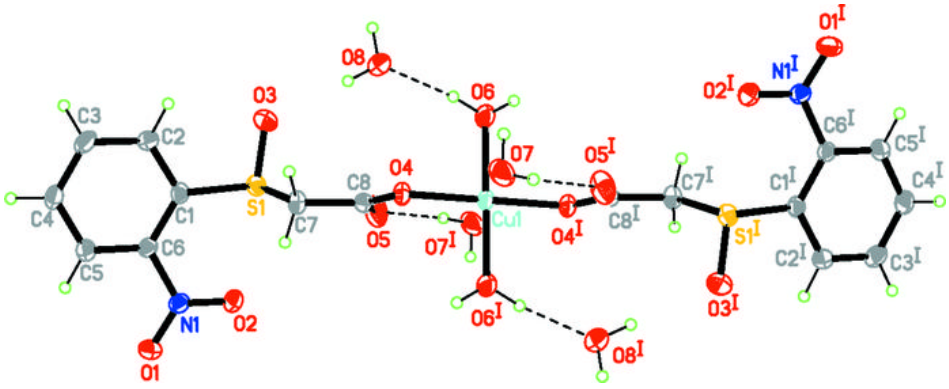
O8
O6
O3
C3
C2
C4
C1
S1
C7
C8
O4
O5
O7
Cu1
O7I
O5I
O4I
C8I
C7I
S1I
O3I
O1I
N1I
O2I
C6I
C5I
C4I
C1I
C2I
C3I
C6
C5
N1
O2
O1
O6I
O8I

Fig. 2