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Rerefinement of poly[[[μ-2,3-di-4-pyridylbutane-2,3-diol-κ^2N:N']-μ-iodido-copper(I)] dimethyl sulfoxide solvate] as an anhydrous honeycomb structure

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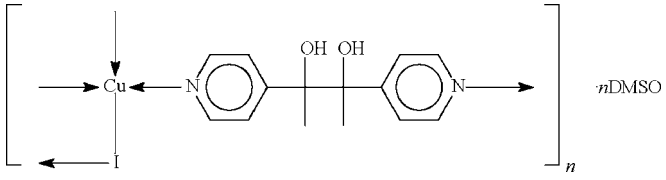
Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.009 Å; some non-H atoms missing; disorder in solvent or counterion; R factor = 0.046; wR factor = 0.184; data-to-parameter ratio = 23.2.

The crystal structure of the title compound, $\{[CuI(C_{14}H_{16}N_2O_2)]\cdot C_2H_6OS\}_n$, consists of (2,3-di-4-pyridylbutane-2,3-diol)iodidocopper(I) units that are connected through Cu—N and Cu—I bonds into a flat honeycomb motif. The dimethyl sulfoxide (DMSO) molecules occupy the spaces within the layer and are hydrogen-bonded to it. The iodide ligand lies on a special position of site symmetry *m*, the Cu atom on a special position of site symmetry 2, and the ligand about a centre of inversion. The DMSO solvent is disordered about a special position of site symmetry *m*. The structure has been re-refined as an anhydrous structure from the diffraction data of Niu, Song, Wang, Guo, Zhu & Hou [*Chem. Lett* (2006), pp. 650–651], who described the structure as a methanol and 0.25-water solvate.

Related literature

$[CuI(C_{14}H_{16}N_2O_2)]\cdot$DMSO was originally incorrectly refined as $[CuI(C_{14}H_{16}N_2O_2)]\cdot DMSO\cdot H_2O$; see the CIF deposited by Niu *et al.* (2006). Their communication misrepresented the compound as $[CuI(C_{14}H_{16}N_2O_2)]\cdot DMSO\cdot CH_3OH\cdot 0.25H_2O$.



Experimental

Crystal data

$[CuI(C_{14}H_{16}N_2O_2)]\cdot C_2H_6OS$
M_r = 512.86
Monoclinic, $C2/m$
a = 19.122 (1) Å
b = 18.499 (1) Å
c = 7.2538 (4) Å
β = 91.424 (1)°
V = 2565.1 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.15 mm^{-1}
T = 291 (2) K
0.37 × 0.25 × 0.22 mm

Data collection

Bruker APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.501, T_{max} = 0.649
7562 measured reflections
3012 independent reflections
2601 reflections with $I > 2\sigma(I)$
R_{int} = 0.014

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.184
S = 1.10
3012 reflections
130 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.52 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cu1—N1	2.055 (4)	Cu1—I1	2.6739 (6)
Cu1—I1—Cu1^{i}	62.97 (3)	N1—Cu1—I1^{i}	108.7 (1)
N1—Cu1—N1^{ii}	113.2 (3)	I1—Cu1—I1^{i}	117.03 (3)
N1—Cu1—I1	104.7 (1)		

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997) and *PLATON* (Spek, 2003); molecular graphics: *X-SEED* (Barbour, 2001) and *OLEX* (Dolomanov *et al.*, 2003); software used to prepare material for publication: *publCIF* (Westrip, 2007).

The author thanks Dr Yun-Yin Niu of Zhengzhou University for the diffraction measurements, and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2166).

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supplementary materials

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Rerefinement of poly[[[μ-2,3-di-4-pyridylbutane-2,3-diol-κ^2N:N']-μ-iodido-copper(I)] dimethyl sulfoxide solvate] as an anhydrous honeycomb structure

S. W. Ng

Comment

The crystal structure of $(C_{14}H_{16}N_2O_2)CuI\cdot DMSO$ was originally refined as a monohydrate; the water molecule is disordered over two positions, and each was refined with quarter site occupacy (Niu *et al.*, 2006). Further, the $(C_{14}H_{16}N_2O_2)CuI\cdot DMSO\cdot H_2O$ formula, as given in the CIF, differs from the $(C_{14}H_{16}N_2O_2)CuI\cdot DMSO\cdot CH_3OH\cdot(H_2O)_{0.25}$ given in the publication. Moreover, the study did not mention the use of methanol in the synthesis (Niu *et al.*, 2006). The discrepancy between the formula given in the cif and that presented in the communication promted the present rerefinement. When the structure was refined without water and methanol, the formula corresponded with the formula expected from the reported CH&N elemental percentages (Niu *et al.*, 2006).

The crystal structure has a void of 29% as calculated by *PLATON* (Spek, 2003). With the exclusion of the DMSO molecules from the calcuation, the void is increased to 53%, so that the compound can be described as being somewhat porous. The layer itself exhibits a flat honeycomb motif. Figure 2 depicts the motif for which the copper atoms are represented as nodes. The DMSO molecules occupy the spaces inside the layer as they interact through H atoms bonds [O···O 2.77 (1) Å].

Experimental

The raw diffraction measurements of the original study by Niu *et al.* (2006) were kindly provided by the senior author. In the rerefinement, the raw data were processed by using a multi-scan absorption correction program (Sheldrick, 1996) in which a model with heavy atoms was assumed.

Refinement

The DMSO is disordered about a mirror plane; the two S—C distances were restrained to be within 0.01 Å of each other.

All H atoms were generated geometrically (O—H 0.82 and C—H 0.93 to 0.97 Å), and were included in the refinement in the riding model approximation, with U(H) set to 1.2–1.5U_{eq}(C,*O*).

The final difference Fourier map had only one peak larger than 1 eA^{-3}, at 3.5 Å from H1, but was otherwise featureless. This peak at (0.482, 0.167, 0.145) is 2.2 Å from its symmetry-related peak.

There is no solvent in the solvent-accessible voids other than the disordered DMSO. The original paper by Niu *et al.* (2006) had formulated the compound $(C_{14}H_{16}N_2O_2)CuI\cdot DMSO\cdot CH_3OH\cdot H_2O_{0.25}$ although the authors did not mention the use of methanol in the synthesis. The present methanol- and water-free formulation is supported by the calculated CH&N percentages (compared with found percentages given in the study) of C 37.26 (37.47), H 4.44 (4.19) and N 5.5 (5.30).

Furthermore, the authors probably used *racemic* 2,3-di(4-pyridyl)-2,3-butanediol instead of the *meso* compound in their synthesis as the ligand lies about a center-of-inversion instead of a mirror plane in the crystal structure.

Figures

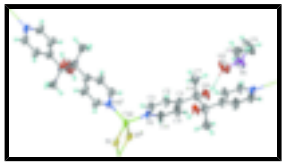

Fig. 1. Thermal ellipsoid plot of a portion of the layer structure of $(C_{14}H_{16}N_2O_2)CuI\cdot DMSO$; displacement ellipsoids are drawn at the 50% probability level. [Symmetry codes are given in Table 1.]

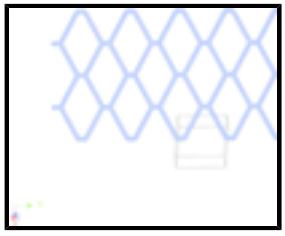

Fig. 2. *OLEX* (Dolomanov *et al.*, 2003) depiction of the (6,3) honeycomb topology, shown projected against the unit cell.

poly[[[μ-2,3-di-4-pyridylbutane-2,3-diol-κ^2*N*:*N*']-μ-\ iodido-copper(I)] dimethyl sulfoxide solvate]

Crystal data

$[CuI(C_{14}H_{16}N_2O_2)]\cdot C_2H_6OS$	F_{000} = 1016
M_r = 512.86	D_x = 1.328 Mg m^{-3}
Monoclinic, *C*2/*m*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -C 2y	Cell parameters from 4414 reflections
a = 19.122 (1) Å	θ = 2.6–28.4º
b = 18.499 (1) Å	μ = 2.15 mm^{-1}
c = 7.2538 (4) Å	*T* = 291 (2) K
β = 91.424 (1)º	Block, light yellow
V = 2565.1 (2) Å^3	0.37 × 0.25 × 0.22 mm
Z = 4	

Data collection

Bruker APEX2 diffractometer	3012 independent reflections
Radiation source: fine-focus sealed tube	2601 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.014
T = 291(2) K	θ_{max} = 27.5º
φ and ω scans	θ_{min} = 2.8º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	*h* = −24→22
T_{min} = 0.501, T_{max} = 0.649	*k* = −23→16
7562 measured reflections	*l* = −9→8

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.046$	H-atom parameters constrained
$wR(F^2) = 0.184$	$w = 1/[\sigma^2(F_o^2) + (0.1259P)^2 + 4.989P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.10$	$(\Delta/\sigma)_{max} = 0.001$
3012 reflections	$\Delta\rho_{max} = 1.27$ e Å^{-3}
130 parameters	$\Delta\rho_{min} = -0.52$ e Å^{-3}
1 restraint	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
I1	0.42163 (2)	0.5000	1.23186 (6)	0.0491 (2)	
Cu1	0.5000	0.42451 (5)	1.0000	0.0501 (3)	
S1	0.2536 (3)	0.0590 (2)	0.0681 (8)	0.104 (1)	0.50
O1	0.3145 (3)	0.2268 (3)	0.3303 (6)	0.079 (1)	
O2	0.3108 (7)	0.0914 (8)	0.167 (2)	0.132 (5)	0.50
N1	0.4296 (2)	0.3634 (2)	0.8486 (5)	0.051 (1)	
C1	0.3357 (3)	0.2772 (4)	0.6314 (8)	0.064 (1)	
C2	0.3343 (4)	0.2783 (4)	0.8263 (8)	0.075 (2)	
C3	0.3819 (3)	0.3202 (4)	0.9252 (7)	0.064 (2)	
C4	0.4282 (3)	0.3658 (4)	0.6656 (8)	0.067 (2)	
C5	0.3825 (4)	0.3243 (5)	0.5539 (8)	0.082 (2)	
C6	0.2850 (3)	0.2308 (4)	0.5105 (8)	0.066 (2)	
C7	0.2775 (4)	0.1568 (4)	0.5977 (11)	0.079 (2)	
C8	0.2060 (8)	0.001 (2)	0.218 (2)	0.110 (6)	
C9	0.290 (1)	−0.011 (2)	−0.071 (4)	0.16 (1)	0.50
H1	0.3021	0.1890	0.2800	0.119*	
H2	0.3014	0.2509	0.8878	0.091*	
H3	0.3807	0.3185	1.0532	0.077*	
H4	0.4592	0.3967	0.6082	0.080*	
H5	0.3839	0.3287	0.4263	0.098*	
H7a	0.3226	0.1341	0.6081	0.119*	
H7b	0.2469	0.1275	0.5220	0.119*	
H7c	0.2583	0.1618	0.7180	0.119*	
H8a	0.1824	0.0302	0.3075	0.165*	0.50
H8b	0.2378	−0.0312	0.2802	0.165*	0.50
H8c	0.1721	−0.0259	0.1471	0.165*	0.50
H9a	0.3228	0.0105	−0.1542	0.243*	0.50
H9b	0.2535	−0.0343	−0.1406	0.243*	0.50
H9c	0.3140	−0.0451	0.0067	0.243*	0.50

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.0485 (3)	0.0563 (3)	0.0423 (3)	0.000	−0.00370 (19)	0.000
Cu1	0.0469 (5)	0.0587 (5)	0.0439 (5)	0.000	−0.0162 (3)	0.000
S1	0.110 (3)	0.076 (2)	0.125 (4)	0.003 (2)	−0.011 (3)	−0.032 (3)
O1	0.078 (3)	0.099 (3)	0.060 (2)	−0.027 (3)	0.002 (2)	−0.027 (2)
O2	0.11 (1)	0.12 (1)	0.17 (1)	−0.025 (8)	0.005 (9)	−0.07 (1)
N1	0.050 (2)	0.062 (2)	0.041 (2)	−0.009 (2)	−0.012 (2)	−0.003 (2)
C1	0.068 (3)	0.076 (4)	0.048 (3)	−0.021 (3)	−0.013 (2)	−0.001 (3)
C2	0.079 (4)	0.103 (5)	0.044 (3)	−0.046 (4)	−0.015 (3)	0.015 (3)
C3	0.070 (3)	0.084 (4)	0.037 (2)	−0.021 (3)	−0.012 (2)	0.006 (2)
C4	0.069 (3)	0.088 (4)	0.043 (3)	−0.033 (3)	−0.005 (2)	−0.001 (3)
C5	0.089 (4)	0.118 (6)	0.037 (2)	−0.056 (4)	−0.010 (3)	0.001 (3)
C6	0.072 (4)	0.069 (3)	0.057 (3)	−0.020 (3)	−0.007 (3)	−0.001 (3)
C7	0.085 (4)	0.061 (3)	0.090 (5)	−0.018 (3)	−0.031 (4)	0.001 (3)
C8	0.12 (1)	0.11 (1)	0.10 (1)	0.01 (5)	−0.037 (9)	−0.02 (5)
C9	0.11 (1)	0.17 (3)	0.21 (2)	−0.02 (2)	0.02 (1)	−0.13 (3)

Geometric parameters (Å, °)

Cu1—N1	2.055 (4)	S1—C8	1.79 (2)
Cu1—I1	2.6739 (6)	S1—C9	1.79 (2)
Cu1—N1^{i}	2.055 (4)	O1—H1	0.82
Cu1—I1^{ii}	2.6738 (6)	C2—H2	0.93
Cu1—Cu1^{ii}	2.793 (2)	C3—H3	0.93
O1—C6	1.438 (8)	C4—H4	0.93
N1—C4	1.328 (7)	C5—H5	0.93
N1—C3	1.343 (7)	C7—H7a	0.96
C1—C5	1.378 (8)	C7—H7b	0.96
C1—C2	1.415 (8)	C7—H7c	0.96
C1—C6	1.551 (7)	C8—H8a	0.96
C2—C3	1.382 (8)	C8—H8b	0.96
C4—C5	1.404 (7)	C8—H8c	0.96
C6—C7	1.517 (9)	C9—H9a	0.96
C6—C6^{iii}	1.52 (1)	C9—H9b	0.96
S1—O2	1.43 (1)	C9—H9c	0.96
Cu1—I1—Cu1^{ii}	62.97 (3)	C6—O1—H1	109.5
N1—Cu1—N1^{i}	113.2 (3)	C3—C2—H2	120.1
N1—Cu1—I1	104.7 (1)	C1—C2—H2	120.1
N1—Cu1—I1^{ii}	108.7 (1)	N1—C3—H3	117.8
N1^{i}—Cu1—I1^{ii}	104.7 (1)	C2—C3—H3	117.8
N1^{i}—Cu1—I1	108.7 (1)	N1—C4—H4	118.1
I1—Cu1—I1^{ii}	117.03 (3)	C5—C4—H4	118.1
N1—Cu1—Cu1^{ii}	123.4 (1)	C1—C5—H5	119.7

I1—Cu1—Cu1^{ii}	58.52 (2)	C4—C5—H5	119.7
C4—N1—C3	115.9 (4)	C6—C7—H7a	109.5
C4—N1—Cu1	120.8 (4)	C6—C7—H7b	109.5
C3—N1—Cu1	123.3 (3)	H7a—C7—H7b	109.5
C5—C1—C2	115.3 (5)	C6—C7—H7c	109.5
C5—C1—C6	121.5 (5)	H7a—C7—H7c	109.5
C2—C1—C6	123.0 (5)	H7b—C7—H7c	109.5
C3—C2—C1	119.8 (5)	S1—C8—H8a	109.5
N1—C3—C2	124.3 (5)	S1—C8—H8b	109.5
N1—C4—C5	123.7 (5)	H8a—C8—H8b	109.5
C1—C5—C4	120.7 (5)	S1—C8—H8c	109.5
O1—C6—C7	112.0 (6)	H8a—C8—H8c	109.5
O1—C6—C6^{iii}	107.3 (6)	H8b—C8—H8c	109.5
C7—C6—C6^{iii}	111.8 (6)	S1—C9—H9a	109.5
O1—C6—C1	106.8 (5)	S1—C9—H9b	109.5
C7—C6—C1	109.2 (5)	H9a—C9—H9b	109.5
C6^{iii}—C6—C1	109.6 (7)	S1—C9—H9c	109.5
O2—S1—C8	110 (1)	H9a—C9—H9c	109.5
O2—S1—C9	106.3 (8)	H9b—C9—H9c	109.5
C8—S1—C9	97 (2)		
Cu1^{ii}—I1—Cu1—N1	−120.5 (1)	Cu1—N1—C3—C2	−179.6 (6)
Cu1^{ii}—I1—Cu1—N1^{i}	118.3 (1)	C1—C2—C3—N1	2(1)
Cu1^{ii}—I1—Cu1—I1^{ii}	0.0	C3—N1—C4—C5	−3(1)
N1^{i}—Cu1—N1—C4	−116.2 (5)	Cu1—N1—C4—C5	178.6 (6)
I1^{ii}—Cu1—N1—C4	−0.3 (5)	C2—C1—C5—C4	4(1)
I1—Cu1—N1—C4	125.5 (5)	C6—C1—C5—C4	−179.8 (7)
Cu1^{ii}—Cu1—N1—C4	63.8 (5)	N1—C4—C5—C1	−0.4 (13)
N1^{i}—Cu1—N1—C3	65.3 (5)	C5—C1—C6—O1	21.1 (9)
I1^{ii}—Cu1—N1—C3	−178.8 (5)	C2—C1—C6—O1	−163.4 (7)
I1—Cu1—N1—C3	−53.0 (5)	C5—C1—C6—C7	142.4 (8)
Cu1^{ii}—Cu1—N1—C3	−114.7 (5)	C2—C1—C6—C7	−42.1 (9)
C5—C1—C2—C3	−5(1)	C5—C1—C6—C6^{iii}	−94.8 (9)
C6—C1—C2—C3	179.0 (7)	C2—C1—C6—C6^{iii}	80.7 (9)
C4—N1—C3—C2	1.8 (10)		

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2	0.82	1.99	2.77 (1)	158

Fig. 1

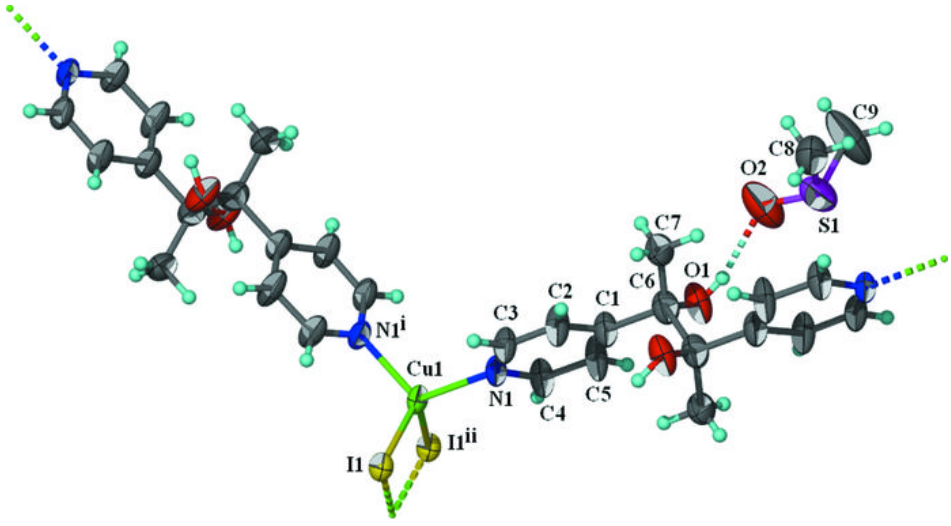

Fig. 2