metal-organic papers

Acta Crystallographica Section E Structure Reports Online

ISSN 1600-5368

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Key indicators

Single-crystal X-ray study T = 298 KMean $\sigma(C-C) = 0.006 \text{ Å}$ Disorder in solvent or counterion R factor = 0.037 wR factor = 0.095 Data-to-parameter ratio = 12.5

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

Tetrakis(μ -naphthalene-1-acetato- $\kappa^2 O:O'$)bis[(N,N-dimethylformamide- κO)copper(II)] N,N-dimethylformamide disolvate

The centrosymmetric title compound, $[Cu_2(C_{12}H_9O_2)_4(C_3H_7NO)_2]\cdot 2C_3H_7NO$, consists of a dinuclear copper paddle-wheel cage structure bridged by four bidentate naphthaleneacetate carboxylate groups. The *N*,*N*-dimethyl-formamide molecules at the axial positions complete the octahedral coordination.

Received 31 May 2006 Accepted 14 September 2006

Comment

Naphthalene-1-acetic acid is a plant-growth regulator and our attention is focused on its coordination with the Cu^{II} ion. A green crystal of the cupric naphthalene-1-acetate salt, (I), was obtained from an *N*,*N*-dimethylformamide (DMF) solution. The crystal structure of (I) consists of a centrosymmetric dinuclear cage unit, in which each Cu^{II} atom is coordinated by four O atoms [O1ⁱ, O2, O3ⁱ and O4; symmetry code: (i) 1 - x, 1 - y, 1 - z] from four different carboxylate groups in equatorial positions and atom O5 from DMF at the axial position (Fig. 1). The dihedral angles of the naphthyl ring planes formed by the four O atoms (O1ⁱ, O2, O3ⁱ and O4) are 63.43 (6) and 60.39 (6)°, respectively. Selected bond parameters are listed in Table 1.



A dimethyl sulfoxide (DMSO) analogue of (I), viz. (II), has been reported previously (Chen *et al.*, 2004). The coordination environment of the Cu^{II} atoms in (II) is similar to that in (I), except that the axial O atoms are from the coordinated DMSO.

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Experimental

A mixture of naphthalene-1-acetic acid (2 mmol) and CuCO₃ (1 mmol) was stirred in water (20 ml); the colour of the resulting precipitate changed from blue to green after about 30 min. The precipitate was washed with water and then dissolved in DMF. Green blocks of (I) suitable for X-ray analysis were obtained by evaporation of this DMF solution at ambient temperature for two months.

7495 measured reflections

 $R_{\rm int}=0.016$

 $\theta_{\rm max} = 25.0^\circ$

4948 independent reflections 3999 reflections with $I > 2\sigma(I)$

Crystal data

$[Cu_2(C_{12}H_0O_2)_4(C_2H_7NO)_2]$	$\nu = 65.205 \ (2)^{\circ}$
2C ₃ H ₇ NO	V = 1423.7 (4) Å ³
$M_r = 1160.23$	Z = 1
Triclinic, $P\overline{1}$	$D_x = 1.353 \text{ Mg m}^{-3}$
a = 10.056 (2) Å	Mo $K\alpha$ radiation
b = 11.936 (2) Å	$\mu = 0.81 \text{ mm}^{-1}$
c = 13.636 (2) Å	T = 298 (2) K
$\alpha = 73.506 \ (2)^{\circ}$	Block, green
$\beta = 84.991 \ (2)^{\circ}$	$0.46 \times 0.33 \times 0.21 \text{ mm}$

Data collection

Bruker SMART CCD area-detector diffractometer φ and ω scans Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996) $T_{\min} = 0.707, T_{\max} = 0.848$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_0^2) + (0.0403P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.037$	+ 0.6167P]
$wR(F^2) = 0.095$	where $P = (F_0^2 + 2F_c^2)/3$
S = 1.04	$(\Delta/\sigma)_{\rm max} = 0.001$
4948 reflections	$\Delta \rho_{\rm max} = 0.35 \text{ e} \text{ \AA}^{-3}$
395 parameters	$\Delta \rho_{\rm min} = -0.27 \text{ e} \text{ \AA}^{-3}$
H-atom parameters constrained	

Table 1

Selected	geometric	parameters	(A,	°).
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Cu1-O3	1.964 (2)	Cu1-O5	2.1493 (18)
Cu1-O4 ⁱ	1.965 (2)	Cu1-Cu1 ⁱ	2.6273 (7)
Cu1-O2 ⁱ	1.9664 (18)	O2-Cu1 ⁱ	1.9664 (18)
Cu1-O1	1.9673 (18)	O4–Cu1 ⁱ	1.966 (2)
	1(0,4(,0))		04.06 (6)
03 - Cu1 - 04	168.46 (8)		84.86 (6)
$O_3 - Cu_1 - O_2^{r}$	89.95 (9)	O4 ⁱ -Cu1-Cu1 ⁱ	83.62 (6)
$O4^{i}-Cu1-O2^{i}$	89.09 (9)	O2 ¹ -Cu1-Cu1 ¹	81.65 (6)
O3-Cu1-O1	89.61 (9)	O1-Cu1-Cu1 ⁱ	86.69 (5)
O4 ⁱ -Cu1-O1	89.02 (8)	O5-Cu1-Cu1 ⁱ	172.80 (6)
$O2^{i}-Cu1-O1$	168.33 (7)	C1-O1-Cu1	119.84 (17)
O3-Cu1-O5	94.69 (8)	C1-O2-Cu1 ⁱ	126.02 (17)
O4 ⁱ -Cu1-O5	96.83 (8)	C13-O3-Cu1	122.01 (19)
O2 ⁱ -Cu1-O5	91.17 (8)	C13-O4-Cu1 ⁱ	123.40 (19)
O1-Cu1-O5	100.49 (7)	C25-O5-Cu1	120.90 (18)

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

Table 2

Hydrogen-bond geometry (Å, °).

$D - H \cdots A$	D-H	$H \cdots A$	$D \cdots A$	$D - \mathbf{H} \cdots A$
$C26-H26B\cdotsO1^{ii}$	0.96	2.49	3.430 (4)	165
Summatry and (ii)	1.2	- 1		

Symmetry code: (ii) -x + 2, -y + 1, -z + 1.



Figure 1

The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. Both disordered DMF components are shown. [Symmetry code: (A) 1 - x, 1 - y, 1 - z.]



Figure 2

A packing diagram of the title compound, showing the weak intermolecular C-H···O interactions as dashed lines. The DMF solvent molecules have been omitted for clarity. [Symmetry codes: (i) x, y, z; (ii) 2 - x, 1 - y, 1 - z].





A packing diagram of (I). The DMF solvent molecules have been omitted for clarity.

All H atoms were located in difference Fourier maps. H atoms bonded to C atoms were treated as riding atoms, with C-H = 0.93(aromatic, formyl), 0.97 (methylene) and 0.96 Å (methyl), and with $U_{iso}(H) = 1.2U_{eq}(C)$ (aromatic, formyl, methylene) or $1.5U_{eq}(C)$ (methyl). The methyl groups bonded to N of the solvent DMF were found to be disordered over two sites. The coordinates of these two sites were refined with the occupancies tied to sum to unity. The site occupancies for C28–C30 with attached H atoms and C28'–C30' with attached H atoms refined to 0.554 (7) and 0.446 (7), respectively.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; 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: *SHELXTL* and *MERCURY* (Macrae *et al.*, 2006).

We acknowledge the financial support of the Liaocheng University Science Foundation.

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