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Bis[(*N*,*N*-3-oxapentamethylenethiocarbamoylthioacetato)triphenyltin] hydrate and bis(dicyclohexylammonium) bis(3-oxapentamethylenethiocarbamoylthioacetate)†

SEIK WENG NG[a] AND JAMES M. HOOK[b]

[a]*Institute of Postgraduate Studies and Research, University of Malaya, 50603 Kuala Lumpur, Malaysia, and* [b]*NMR Facility, School of Chemistry, University of New South Wales, Sydney 2052, Australia. E-mail: hlnswen@umcsd.um.edu.my*

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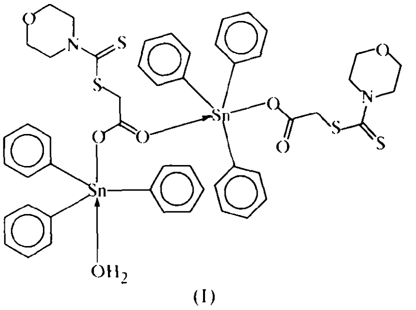
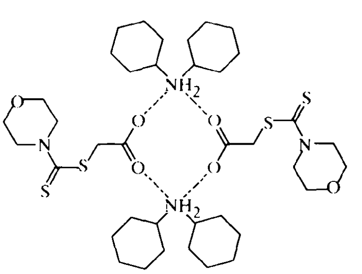
Abstract

In the two independent dinuclear molecules of bis[(*N*,*N*-3-oxapentamethylenethiocarbamoylthioacetato)triphenyltin] hydrate, $[Sn_2(C_6H_5)_6(C_7H_{10}NO_3S_2)_2(H_2O)]$, the aqua(*N*,*N*-3-oxapentamethylenethiocarbamoylthioacetato)triphenyltin moiety uses its carbonyl O atom to connect to the Sn atom of the (*N*,*N*-3-oxapentamethylenethiocarbamoylthioacetato)triphenyltin moiety, to furnish *trans*-C_3SnO_2 trigonal bipyramidal geometries for the Sn atoms in the dinuclear compound. Hydrogen bonds link the coordinated water of one symmetry-independent molecule to the carbonyl and heterocyclic O atoms of the other independent molecule [$O\cdots O$ = 2.66 (4) and 2.75 (2), and 2.769 (8) and 2.667 (7) Å, respectively] in a layer structure. The orthorhombic unit cell of bis(dicyclohexylammonium) bis(3-oxapentamethylenethiocarbamoylthioacetate), $2C_{12}H_{24}N^+\cdot 2C_7H_{10}NO_3S_2^-$, contains four centrosymmetric clusters of two cations and two anions. The C—O bonds in the carboxyl $-CO_2$ fragment of the anion are delocalized. The ammonium N atom is hydrogen bonded to two carboxyl O atoms belonging to different anions [$N\cdots O$ = 2.732 (4) and 2.738 (4) Å] to form an eight-membered $O—C—O\cdots N\cdots O—C—O\cdots N\cdots$ ring.

Comment

Triphenyltin thiocarbamoylthioacetates, $[(C_6H_5)_3SnO_2CCH_2SC(S)NR_2]$, have been assigned carboxylate-bridged structures on the basis of spectroscopic measurements (Ng & Kumar Das, 1991). The assignments have been corroborated by crystal structure analysis for the NR_2 = $N(CH_3)_2$ (Ng & Kumar Das, 1995*a*), $N(C_2H_5)_2$ and $N(CH_3)(C_6H_5)$ (Lo *et al.*, 1999) derivatives. The NR_2 = $NO(CH_2CH_2)_2$ derivative, (I), crystallizes with half a water molecule, and there are two independent dinuclear molecules in the unit cell. Each consists of a water-coordinated aqua(*N*,*N*-3-oxapentamethylenethiocarbamoylthioacetato)triphenyltin moiety


(I)


(II)

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† Alternative names: aqua-1κ*O*-μ-[(4-morpholinecarbothioylthio)acetato]-1:2κ²*O*:*O*′-[(4-morpholinecarbothioylthio)acetato]-2κ*O*-hexaphenyl-1κ³*C*,2κ³*C*-ditin monohydrate and bis(dicyclohexylammonium) bis[(4-morpholinecarbothioylthio)acetate].

that uses its carbonyl O atom to bridge to the non-water-coordinated (*N*,*N*-3-oxapentamethylenethiocarbamoylthioacetato)triphenyltin moiety [molecule *A*: Sn—O_{water} = 2.422 (3) and Sn—O_{bridge} = 2.493 (4) Å; molecule *B*: Sn—O_{water} = 2.415 (4) and Sn—O_{bridge} = 2.530 (4) Å]. The water molecule of one independent molecule is linked by hydrogen bonds to the carbonyl and heterocyclic O atoms of the other independent molecule in a layer structure. Triphenyltin *N*,*N*-3-oxapentamethylenedithiocarbamate, on the other hand, exists as a tetrahedral molecule (Ng, 1997*b*). The hydrogen-bonding scheme contrasts with that of bis[(*N*,*N*-3-oxapentamethylenethiocarbamoylacetato)triphenyltin] hydrate, which comprises an aqua(*N*,*N*-3-oxapentamethylenethiocarbamoylacetato)triphenyltin moiety that is bridged to the

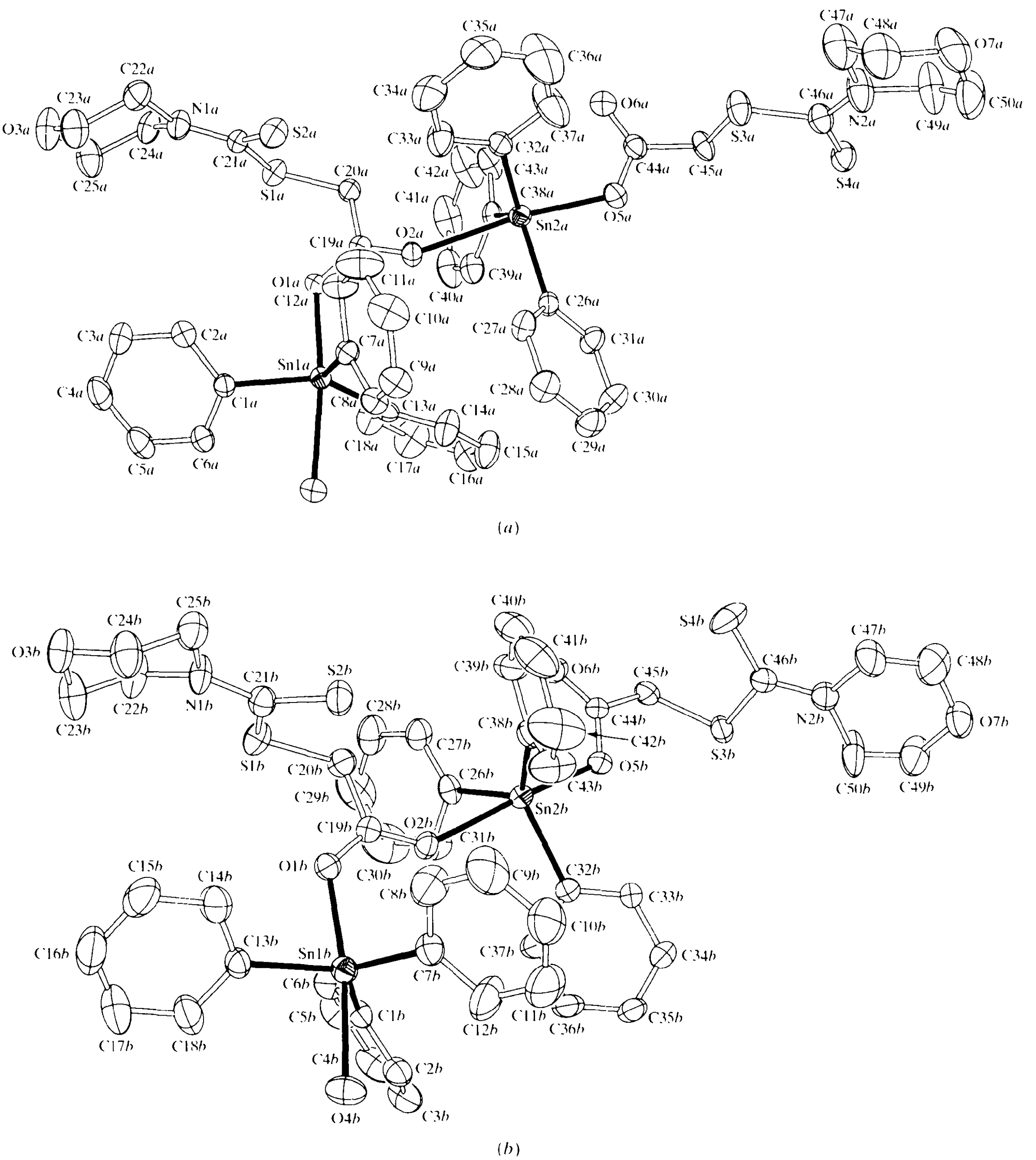

Fig. 1. *ORTEP*II (Johnson, 1976) plots of (*a*) molecule *A* of (I) and (*b*) molecule *B* of (I), showing the atom-numbering schemes. Displacement ellipsoids are drawn at the 30% probability level and H atoms are not shown.

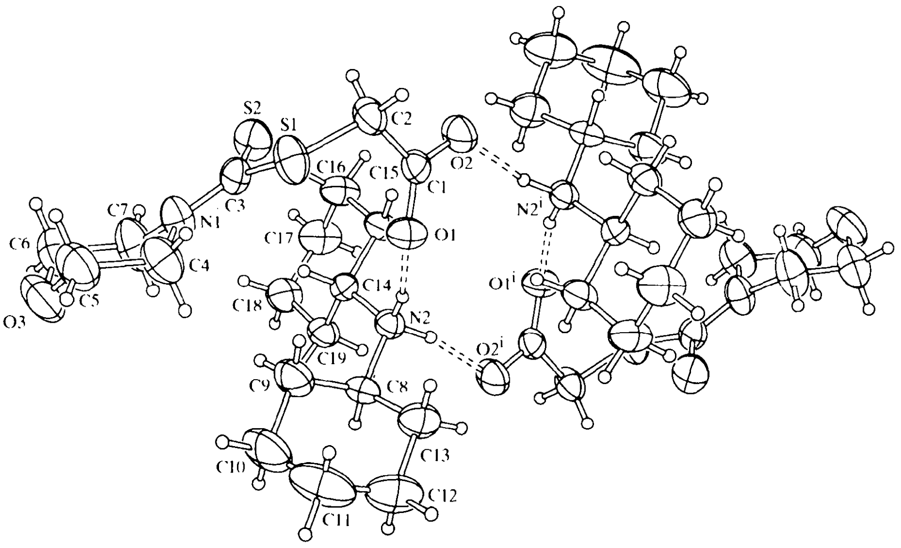

Fig. 2. *ORTEP*II (Johnson, 1976) plot of (II), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level, except for H atoms, which are shown as spheres of arbitrary radii [symmetry code: (i) $2 - x$, $-y$, $1 - z$].

non-water-coordinated (*N*,*N*-3-oxapentamethylenethiocarbamoylacetato)triphenyltin moiety [Sn—O$_{water}$ = 2.298 (4) and Sn—O$_{bridge}$ = 2.381 (4) Å]; however, the water molecule is instead linked to adjacent carbonyl and carbamoyl O atoms [O$_{water}$···O = 2.615 (7) and 2.685 (8) Å, respectively] into a linear chain structure (Ng, 1996*b*). Compound (I) differs in having a doubly bonded O atom in place of an S atom, so that (I) is not expected to use this atom to form hydrogen bonds with water, which interacts instead with the heterocyclic O atom. The electron-withdrawing ability of the substituents on the acetato group in the triphenyltin thiocarbamoylthioacetates directly affects the Lewis acceptor ability of the triphenyltin cation, as well as indirectly affecting the coordinated water. The R_2NCS_2– group is more strongly electron-withdrawing than the $R_2NC(=O)S$– group; further increasing the Lewis acidity of the triphenyltin cation by introducing a second dithiocarbamoyl R_2NCS_2– group in the anion leads to the formation of a triorganotin carboxylate having both coordinated and lattice water, as exemplified by aquabis(*N*,*N*-dimethylthiocarbamoylthio)acetatotriphenyltin hydrate. In this compound, the lattice water interacts indirectly with the Sn atom through the coordinated water in an 'outer-sphere coordination' arrangement (Ng & Kumar Das, 1995*b*).

The Sn—O$_{water}$, Sn—O$_{bridge}$ and O$_{water}$···O interactions in the bis[(*N*,*N*-3-oxapentamethylenethiocarbamoylthioacetato)triphenyltin] hydrate are longer than those in the bis[(*N*,*N*-3-oxapentamethylenethiocarbamoylacetato)triphenyltin] hydrate. The Sn—O$_{water}$ distances are somewhat longer than distances found in mononuclear hydrates (Kumar Das *et al.*, 1977), as are the carboxylate bridging distances (Ng *et al.*, 1988; Tiekink, 1991, 1994).

The *N*,*N*-3-oxapentamethylenethiocarbamoylthioacetate anion, characterized as its dicyclohexylammonium salt, (II), exists as an anhydrous dimeric ion pair. The hydrogen-bonding interactions between the carboxyl O and ammonium N atoms are already maximized, and the compound does not require lattice water to consolidate the structure. Its architecture is similar to those found in monohydrated dicyclohexylammonium dimethylthiocarbamoylthioacetate, which displays a characteristic centrosymmetric eight-membered O—C—O···N···O—C—O···N··· hydrogen-bonded ring [N—O = 2.706 (4) and 2.750 (3) Å; Ng, 1993], bis(dicyclohexylammonium) bis[bis(dimethylthiocarbamoylthio)acetate] [2.701 (3) and 2.796 (3), and 2.698 (3) and 2.837 (3) Å; Ng, 1996*a*] and bis(dicyclohexylammonium) bis[dicyclohexylthiocarbamoylthioacetate] [2.706 (2) and 2.757 (2) Å; Ng, 1997*a*].

Experimental

The organotin carboxylate was obtained by condensing *N*,*N*-3-oxapentamethylenethiocarbamoylthioacetic acid (Nachmias, 1952; Pluijgers & van der Kerk, 1961) and triphenyltin hydroxide in a 1:1 molar ratio in ethanol (Ng & Kumar Das, 1991). The reagents were heated in a small volume of ethanol until they dissolved completely; the compound separated as plates from the filtered solution on slow cooling. The ammonium carboxylate was similarly synthesized from the carboxylic acid and dicyclohexylamine (1:1 molar ratio); it separated as large crystals. Solid-state ^{119}Sn NMR (relative to Me_4Sn): cluster of four sites centered at −300, −307, −337 and −347 p.p.m.

Compound (I)

Crystal data

$[Sn_2(C_6H_5)_6(C_7H_{10}NO_3S_2)_2(H_2O)]$
$M_r = 1158.56$
Triclinic
$P\bar{1}$
$a = 9.6433\ (6)$ Å
$b = 21.545\ (2)$ Å
$c = 25.366\ (2)$ Å
$\alpha = 101.730\ (6)°$
$\beta = 93.312\ (5)°$
$\gamma = 96.534\ (6)°$
$V = 5108.8\ (6)$ Å^3
$Z = 4$
$D_x = 1.506$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 12.25$–$12.75°$
$\mu = 1.192$ mm^{-1}
$T = 298\ (2)$ K
Plate
$0.55 \times 0.55 \times 0.15$ mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.524$, $T_{\max} = 0.662$
19 136 measured reflections
17 961 independent reflections

12 531 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.019$
$\theta_{\max} = 24.97°$
$h = 0 \rightarrow 11$
$k = -25 \rightarrow 25$
$l = -30 \rightarrow 30$
3 standard reflections frequency: 60 min intensity decay: 5.0%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.116$
$S = 0.994$
17 961 reflections
1027 parameters
H atoms riding, with $U({\rm H}) = 1.5U_{\rm eq}({\rm C,O})$
$w = 1/[\sigma^2(F_o^2) + (0.0531P)^2 + 4.1636P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 0.966$ e Å^{-3}
$\Delta\rho_{\min} = -0.554$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °) for (I)*

Sn1*A*—C1*A*	2.132 (3)	Sn1*B*—C1*B*	2.126 (3)
Sn1*A*—C7*A*	2.140 (3)	Sn1*B*—C7*B*	2.125 (4)
Sn1*A*—C13*A*	2.121 (3)	Sn1*B*—C13*B*	2.129 (3)
Sn1*A*—O1*A*	2.149 (3)	Sn1*B*—O1*B*	2.133 (4)
Sn1*A*—O4*A*	2.422 (3)	Sn1*B*—O4*B*	2.415 (4)
Sn2*A*—C26*A*	2.132 (3)	Sn2*B*—C26*B*	2.128 (4)
Sn2*A*—C32*A*	2.135 (3)	Sn2*B*—C32*B*	2.148 (3)
Sn2*A*—C38*A*	2.124 (3)	Sn2*B*—C38*B*	2.119 (3)
Sn2*A*—O2*A*	2.493 (4)	Sn2*B*—O2*B*	2.530 (4)
Sn2*A*—O5*A*	2.131 (4)	Sn2*B*—O5*B*	2.150 (4)
C1*A*—Sn1*A*—C13*A*	118.9 (2)	C1*B*—Sn1*B*—C7*B*	122.4 (2)
C1*A*—Sn1*A*—C7*A*	118.7 (2)	C1*B*—Sn1*B*—C13*B*	118.2 (2)
C1*A*—Sn1*A*—O1*A*	86.1 (1)	C1*B*—Sn1*B*—O1*B*	96.8 (2)
C1*A*—Sn1*A*—O4*A*	86.5 (1)	C1*B*—Sn1*B*—O4*B*	88.1 (2)
C7*A*—Sn1*A*—C13*A*	121.1 (2)	C7*B*—Sn1*B*—C13*B*	118.2 (2)
C7*A*—Sn1*A*—O1*A*	98.8 (1)	C7*B*—Sn1*B*—O1*B*	98.4 (2)
C7*A*—Sn1*A*—O4*A*	86.2 (1)	C7*B*—Sn1*B*—O4*B*	85.1 (2)
C13*A*—Sn1*A*—O1*A*	96.0 (2)	C13*B*—Sn1*B*—O1*B*	85.0 (2)
C13*A*—Sn1*A*—O4*A*	86.1 (1)	C13*B*—Sn1*B*—O4*B*	86.2 (2)
O1*A*—Sn1*A*—O4*A*	172.4 (1)	O1*B*—Sn1*B*—O4*B*	171.1 (2)
C19*A*—O1*A*—Sn1*A*	124.4 (3)	C19*B*—O1*B*—Sn1*B*	128.5 (4)
C19*A*—O2*A*—Sn2*A*	141.1 (3)	C19*B*—O2*B*—Sn2*B*	138.2 (4)
C26*A*—Sn2*A*—C32*A*	111.7 (2)	C26*B*—Sn2*B*—C32*B*	115.8 (2)
C26*A*—Sn2*A*—C38*A*	118.4 (2)	C26*B*—Sn2*B*—C38*B*	125.9 (2)
C26*A*—Sn2*A*—O2*A*	85.5 (1)	C26*B*—Sn2*B*—O2*B*	82.7 (2)
C26*A*—Sn2*A*—O5*A*	88.0 (2)	C26*B*—Sn2*B*—O5*B*	98.9 (2)
C32*A*—Sn2*A*—C38*A*	127.3 (2)	C32*B*—Sn2*B*—C38*B*	114.5 (2)
C32*A*—Sn2*A*—O2*A*	87.8 (1)	C32*B*—Sn2*B*—O2*B*	86.4 (1)
C32*A*—Sn2*A*—O5*A*	97.5 (2)	C32*B*—Sn2*B*—O5*B*	90.0 (2)
C38*A*—Sn2*A*—O2*A*	80.9 (1)	C38*B*—Sn2*B*—O2*B*	81.7 (2)
C38*A*—Sn2*A*—O5*A*	99.5 (2)	C38*B*—Sn2*B*—O5*B*	99.9 (2)
O2*A*—Sn2*A*—O5*A*	172.8 (1)	O2*B*—Sn2*B*—O5*B*	176.4 (1)
C44*A*—O5*A*—Sn2*A*	125.6 (4)	C44*B*—O5*B*—Sn2*B*	119.5 (4)

Compound (II)

Crystal data

$2C_{12}H_{24}N^+ \cdot 2C_7H_{10}NO_3S_2^-$
$M_r = 805.20$
Orthorhombic
$Pbca$
$a = 20.771\ (1)$ Å
$b = 9.1734\ (6)$ Å
$c = 22.756\ (2)$ Å
$V = 4335.9\ (5)$ Å^3
$Z = 4$
$D_x = 1.233$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9$–$11°$
$\mu = 0.266$ mm^{-1}
$T = 298\ (2)$ K
Irregular block
$0.55 \times 0.50 \times 0.45$ mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.779$, $T_{\max} = 0.802$
3805 measured reflections
3803 independent reflections

2255 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.058$
$\theta_{\max} = 24.97°$
$h = 0 \rightarrow 24$
$k = -10 \rightarrow 0$
$l = -27 \rightarrow 0$
3 standard reflections frequency: 60 min intensity decay: 3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.131$
$S = 1.012$
3803 reflections
244 parameters
H atoms riding, with $U({\rm H}) = 1.5U_{\rm eq}({\rm C})$; ammonium H atoms were located and refined
$w = 1/[\sigma^2(F_o^2) + (0.0577P)^2 + 0.6161P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.244$ e Å^{-3}
$\Delta\rho_{\min} = -0.188$ e Å^{-3}
Extinction correction: *SHELXL*97 (Sheldrick, 1997*a*)
Extinction coefficient: 0.0051 (5)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 2. *Selected geometric parameters (Å, °) for (II)*

S1—C2	1.792 (3)	N1—C3	1.333 (4)
S1—C3	1.770 (3)	N1—C4	1.472 (4)
S2—C3	1.658 (3)	N1—C7	1.475 (4)
O1—C1	1.242 (3)	N2—C14	1.498 (4)
O2—C1	1.246 (4)	N2—C8	1.501 (4)
C2—S1—C3	103.1 (2)	O1—C1—C2	119.0 (3)
C5—O3—C6	109.1 (3)	O2—C1—C2	114.3 (3)
C3—N1—C4	124.5 (3)	C1—C2—S1	115.7 (2)

C3—N1—C7	120.6 (3)	N1—C3—S1	113.5 (2)
C4—N1—C7	113.1 (3)	N1—C3—S2	124.5 (3)
C14—N2—C8	117.8 (2)	S1—C3—S2	122.0 (2)
O1—C1—O2	126.7 (3)		

In (I), phenyl groups were refined as rigid hexagons. The morpholinyl rings are somewhat disordered and restraints had to be applied. The N—C and O—C distances were *DFIX*ed (*DFIX* in *SHELXL*97; Sheldrick, 1997*a*) at 1.45±0.01 Å, whereas the C—C distances were *DFIX*ed at 1.54±0.02 Å; additionally, C22···C24 = C47···C49 = 2.51±0.02 Å and C23···C25 = C48···C50 = 2.37±0.02 Å.

For both compounds, data collection: *CAD*-4/*PC* (Kretschmar, 1994). Cell refinement: *CAD*-4 *Manual* (Enraf–Nonius, 1988) for (I); *CELDIM* in *CAD*-4 *Manual* for (II). For both compounds, data reduction: *XCAD*4 (Harms, 1997); program(s) used to solve structures: *SHELXS*97 (Sheldrick, 1997*b*); program(s) used to refine structures: *SHELXL*97; molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

We thank the National Science Council for R&D, Malaysia (IRPA 09-02-03-0004 and IRPA 09-02-03-0371), for supporting this work.

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A dimeric copper(II) bis(4-chlorophenoxy)acetate adduct with dimethylformamide

Yoshiyuki Kani,[a] Masanobu Tsuchimoto,[a] Shigeru Ohba,[a] Hideaki Matsushima,[b] Masakazu Noguchi[b] and Tadashi Tokii[b]

[a]*Department of Chemistry, Faculty of Science and Technology, Keio University, Hiyoshi 3-14-1, Kohoku-ku, Yokohama 223-8522, Japan, and* [b]*Department of Chemistry, Faculty of Science and Engineering, Saga University, Saga 840-8502, Japan. E-mail: ohba@chem.keio.ac.jp*

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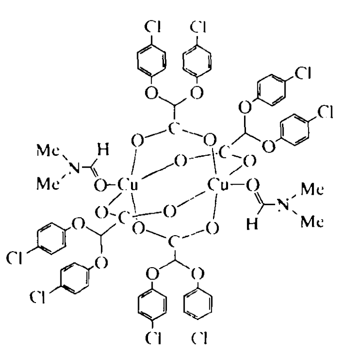
Abstract

The crystal structure of tetrakis[μ-bis(4-chlorophenoxy)acetato-O:O']bis[(dimethylformamide-O)copper(II)], $[Cu(bpca)_2(dmf)]_2$ [bpca is bis(4-chlorophenoxy)acetate, $C_{14}H_9Cl_2O_4$; dmf is dimethylformamide, C_3H_7NO], consists of a dinuclear Cu^{II} complex which has a typical cage structure, with a Cu···Cu distance of 2.685 (1) Å. The magnetic $-2J$ value is 361 cm^{-1} ($H = -2JS_1.S_2$).

Comment

The dinuclear title complex, (I), has a center of symmetry, and the conformations of the two independent bpca ligands [bpcaH is bis(4-chlorophenoxy)acetic acid] are different. The PhO—C—C(O)—O torsion angle is 55.1 (5)° for one bpca ligand (O4—C2—C1—O2) and −8.2 (5)° for the other (O8—C16—C15—O5). The planes of the two phenoxy groups in each of the bpca moieties are almost perpendicular to each other, the dihedral angles being 85.8 (3) and 89.8 (3)°. However, the ring-to-ring dihedral angle of the phenoxy groups is 59.7° in the crystals of bpcaH (Smith & Kennard, 1981).



(I)

In this study, the magnetic susceptibility of the title complex was measured using the Faraday method over

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