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{[μ-Bis(salicylidene)-1,3-propanediaminato]copper(II)}dibromomercury(II)

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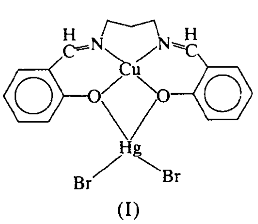
Abstract

In the title complex, dibromo-2κ^2Br-μ-{2,2′-[1,3-propanediylbis(nitrilomethylidyne)]diphenolato}-1κ^4N,N',O,O':2κ^2O,O'-copper(II)mercury(II), $[Cu(C_{17}H_{16}N_2O_2)\{HgBr_2\}]$, the Cu^{II} and Hg^{II} atoms are doubly bridged by the two O atoms of the N,N'-bis(salicylidene)-1,3-propanediaminate (SALPD^{2-}) ligand, and the two N atoms of this ligand complete the distorted square-planar coordination around the Cu^{II} atom. The coordination around the Hg^{II} atom is distorted tetrahedral, involving two bridging O atoms and two Br atoms. The bond angles in the $HgCuO_2$ bridging plane are in the range 61.4 (1)–108.0 (2)°. The Cu···Hg distance is 3.523 (3) Å.

Comment

The title complex, (I), was synthesized in the process of extending our structural studies on doubly oxygen-bridged dimeric homonuclear metal complexes to heteronuclear complexes (Ülkü *et al.*, 1998, and references therein).



(I)

As can be seen from Fig. 1, the distorted square-planar coordination around the Cu^{II} atom involves two O and two N atoms from the N,N'-bis(salicylidene)-1,3-propanediaminate (SALPD^{2-}) ligand. The Cu^{II} atom is located −0.0261 (7)° from the best plane of the coordinating O1, O2, N2 and N1 atoms. The bond angles within the coordination plane around the Cu^{II} atom range from 82.0 (2) (O1—Cu—O2) to 170.7 (2)° (O1—Cu—N2). In the bridging plane, the two Cu—O distances [1.919 (4) and 1.921 (3) Å] are equal within experimental error and compare well with values reported previously (Baker *et al.*, 1970; Ülkü *et al.*, 1997; Tahir *et al.*, 1996), but the Hg—O bond lengths [2.418 (4) (Hg—O1) and 2.519 (3) Å (Hg—O2)] are slightly different. The Cu···Hg distance is 3.523 (3) Å.

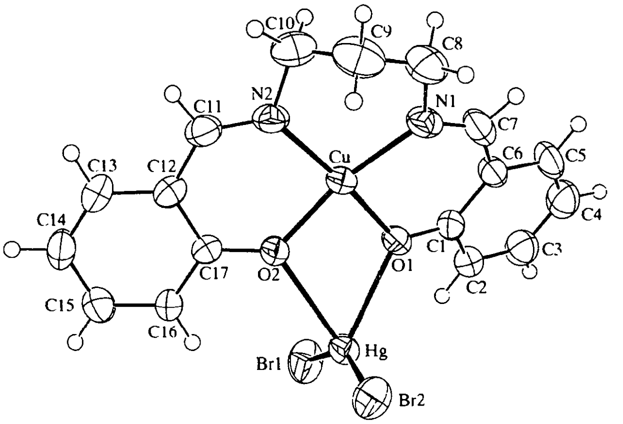


Fig. 1. *ORTEP* (Johnson, 1965) drawing of the title compound with the atomic numbering scheme. The displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as small circles of arbitrary radii.

The coordination around the Hg^{II} atom is distorted tetrahedral consisting of two bridging O atoms and two Br atoms. The smallest bond angle of the distorted tetrahedron around the Hg^{II} atom is 61.4 (1)° (O1—Hg—O2) and the largest bond angle is 152.25 (3)° (Br1—Hg—Br2). The dihedral angle between the O1/Hg/O2 and Br1/Hg/Br2 moieties is 87.2 (1)°. The distances of the *para*-positioned Cu and C9 atoms of the Cu/N1/C8/C9/C10/N2 chelate ring from the plane of the other four atoms are 0.185 (1) and 0.648 (7) Å, respectively. In the Cu/N2/C11/C12/C17/O2 chelate ring, the Cu^{II} atom has the greatest deviation of 0.0695 (7) Å from the best plane, while the greatest deviation from the best plane of the Cu/N1/C7/C6/C1/O1 chelate ring is for the O1 atom [0.178 (4) Å].

Experimental

Ammonia solution (20 ml) was added to a solution of N,N'-bis(salicylidene)-1,3-propanediamine (2.82 g, 10 mmol) in hot ethanol (30 ml) and the mixture was heated. A solution of $CuCl_2\cdot 2H_2O$ (1.70 g, 10 mmol) in hot water (30 ml) was added and the resulting mixture was set aside. After 2 h, the resulting copper complex, *i.e.* $[Cu(C_{17}H_{16}N_2O_2)]$, was filtered off and dried. This complex (0.343 g, 1 mmol) was dissolved in hot dioxane (50 ml) and a solution of $HgBr_2$ (0.360 g, 1 mmol) in

MeOH (20 ml) was added. Brown crystals suitable for X-ray single-crystal diffraction analysis were obtained after 24 h.

Crystal data

$[CuHgBr_2(C_{17}H_{16}N_2O_2)]$
$M_r = 704.277$
Triclinic
$P\overline{1}$
$a = 9.0081\ (10)$ Å
$b = 9.5235\ (10)$ Å
$c = 11.9340\ (13)$ Å
$\alpha = 97.054\ (9)^\circ$
$\beta = 105.340\ (9)^\circ$
$\gamma = 106.471\ (9)^\circ$
$V = 924.90\ (19)$ Å^3
$Z = 2$
$D_x = 2.529$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9–18^\circ$
$\mu = 13.7$ mm^{-1}
$T = 295$ K
Prismatic
$0.125 \times 0.100 \times 0.075$ mm
Brown

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical *via* ψ scans (Fair, 1990)
$T_{min} = 0.222$, $T_{max} = 0.358$
3547 measured reflections
3370 independent reflections

2713 reflections with $F > 3\sigma(F)$
$R_{int} = 0.012$
$\theta_{max} = 25.9^\circ$
$h = -11 \rightarrow 10$
$k = -11 \rightarrow 11$
$l = 0 \rightarrow 14$
3 standard reflections
frequency: 120 min
intensity decay: 1%

Refinement

Refinement on F
$R = 0.024$
$wR = 0.032$
$S = 1.14$
2713 reflections
226 parameters
H-atom parameters constrained

$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.817$ e Å^{-3}
$\Delta\rho_{min} = -0.247$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

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

Cu···Hg	3.523 (3)	Cu—O1	1.921 (3)
Hg—Br1	2.4489 (8)	Cu—O2	1.919 (4)
Hg—Br2	2.4579 (8)	Cu—N1	1.991 (5)
Hg—O1	2.418 (4)	Cu—N2	1.957 (4)
Hg—O2	2.519 (3)		
Br1—Hg—Br2	152.25 (3)	O1—Cu—N1	90.7 (2)
Br1—Hg—O1	107.9 (1)	O1—Cu—N2	170.7 (2)
Br1—Hg—O2	103.8 (1)	O2—Cu—N1	167.8 (2)
Br2—Hg—O1	99.4 (1)	O2—Cu—N2	91.7 (2)
Br2—Hg—O2	93.5 (1)	N1—Cu—N2	96.5 (2)
O1—Hg—O2	61.4 (1)	Hg—O1—Cu	108.0 (2)
O1—Cu—O2	82.0 (2)	Hg—O2—Cu	104.3 (1)

All H atoms were placed geometrically 0.95 Å from their parent atoms. H-atom displacement parameters were fixed as $U_{iso}(H) = 1.3U_{eq}$(parent) and a riding model was used for all H atoms.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1993). Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *MolEN*. Program(s) used to refine structure: *MolEN*. Molecular graphics: *MolEN* version of *ORTEP* (Johnson, 1965). Software used to prepare material for publication: *MolEN*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: OS1060). Services for accessing these data are described at the back of the journal.

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$[ReO_2(dppp)_2]I_x[ReO_4]_{1-x}\cdot xH_2O\cdot CH_3OH$ for $x = 0.17\ (1), 0.36\ (1)$ and 1 [dppp is 1,3-bis(diphenylphosphino)propane]

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Abstract

The crystal structures of $[ReO_2(dppp)_2]I_x[ReO_4]_{1-x}\cdot xH_2O\cdot CH_3OH$, where $x = 0.17\ (1), 0.36\ (1)$ and 1, *i.e.* dioxo[propane-1,2-diylbis(diphenylphosphine)-*P,P′*]-rhenium iodide perrhenate hydrate methanol solvate, with iodide partially substituted by the perrhenate ion,