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Bis(μ-salicylato-$\kappa^3O,O':O'$)bis[(imidazole-κN^3)-(salicylato-κ^2O,O')cadmium(II)]

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Key indicators

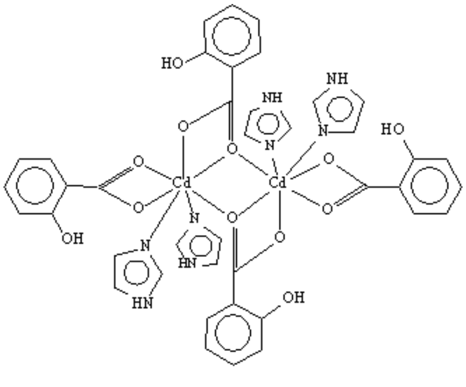
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.026
wR factor = 0.062
Data-to-parameter ratio = 16.6

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

Each Cd atom in the title centrosymmetric dinuclear complex, $[Cd_2(Hsal)_4(Him)_4]$ (where $Hsal^-$ is the salicylate monoanion, $C_7H_5O_3^-$, and Him is imidazole, $C_3H_4N_2$), shows a pentagonal–bipyramidal geometry. Five carboxylate O atoms of different salicylates and two imidazole N atoms are coordinated to each Cd atom. The two Cd atoms are bridged by two tridentate chelating salicylate ligands, and the Cd$\cdots$Cd distance is 3.86 (3) Å. A two-dimensional layer structure is formed *via* intermolecular hydrogen bonds.

Comment

Many structures of transition metal complexes containing the salicylate ligand have been reported. In the structural investigation of these complexes, it has been found that salicylic acid functions either as a monodentate ligand (Hanic & Michalov, 1960) or as a multidentate ligand (Vincent *et al.*, 1986), with versatile binding modes and coordination. The structures of the dinuclear complexes $[M(\mu_2\text{-Hsal})(\text{Hsal})(L)_2]_2$ (where L is a water molecule or an N-heterocycle ligand) have only been reported for the complexes $[Mn(Hsal)_2(H_2O)_2]_2$ (Devereux *et al.*, 1995) and $[Cd(Hsal)_2(H_2O)_2]_2$ (Charles *et al.*, 1983). Recently, we obtained the title novel dinuclear cadmium complex $[Cd(\mu_2\text{–}Hsal)(Hsal)(Him)_2]_2$, (I), by the reaction of $Cd(NO_3)\cdot 4H_2O$, salicylic acid and imidazole in an aqueous solution, and its crystal structure is reported here.



(I)

As shown in Fig. 1, the crystal structure of (I) may be described as containing essentially isolated asymmetrically bridged dimers of $Cd(Hsal)_2(Him)_2$. The centre of the dimer corresponds to a crystallographic centre of symmetry. The dimer is formed by two asymmetric bridging carboxylate O atoms, with $Cd1—O5^i$ 2.381 (2) Å and Cd—O5 2.530 (2) Å [symmetry code: (i) $-x$, $2 - y$, $-z$]. The Cd$\cdots$Cd distance is 3.86 (3) Å.

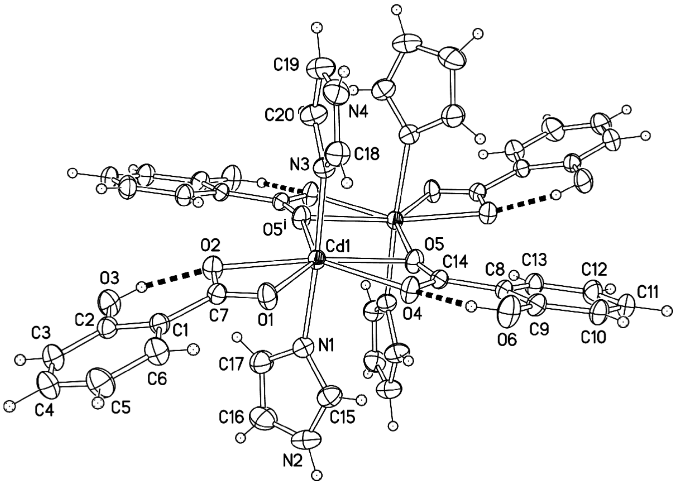


Figure 1
A view of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. Dashed lines indicate intramolecular hydrogen bonds.

The coordination polyhedron of each Cd atom is an approximate pentagonal bipyramid, with the five carboxyl O atoms in the equatorial plane, two of which are bridging atoms (O5 and O5^i). The two imidazole N atoms are in axial positions, with a mean Cd—N distance of 2.246 (2) Å.

The stability of complex (I) in the solid state is further enhanced by intramolecular hydrogen bonding between the hydroxyl groups of the Hsal$^-$ function and one carboxylate O atom of the same ligand. The hydrogen-bond distances (O···O) and angles (O—H···O) are in the ranges 2.486 (2)–2.547 (2) Å and 147–149°, respectively. The dihedral angle between the two benzene rings is 25.7 (3)°, whereas the dihedral angle between the two imidazole rings is 36.9 (3)°. A two-dimensional layer structure is formed *via* intermolecular hydrogen bonds between the H atoms of the imidazole N atoms and the O atoms of the carboxyl groups (Table 2 and Fig. 2).

Experimental

The title complex was prepared by the addition of cadmium nitrate tetrahydrate (20 mmol) to an aqueous solution of imidazole (40 mmol) and salicylic acid (40 mmol). The pH was adjusted to 6 with 0.1 *M* NaOH and the mixed solution was stirred for 30 min at room temperature. The resulting solution was filtered and colourless prismatic crystals of (I) were isolated from the filtrate after about 7 d. Analysis calculated for $C_{40}H_{36}Cd_2N_8O_{12}$: C 45.93, H 3.47, N 10.71%; found: C 45.76, H 3.42, N 10.90%.

Crystal data

$[Cd_2(C_7H_5O_3)_4(C_3H_4N_2)_4]$
M_r = 1045.57
Triclinic, $P\bar{1}$
a = 9.339 (2) Å
b = 9.793 (2) Å
c = 12.251 (3) Å
α = 79.21 (3)°
β = 86.28 (3)°
γ = 69.76 (3)°
V = 1032.7 (5) Å^3
Z = 1
D_x = 1.681 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 9315 reflections
θ = 3.0–27.5°
μ = 1.10 mm^{-1}
T = 293 (2) K
Prism, colourless
0.35 × 0.24 × 0.18 mm

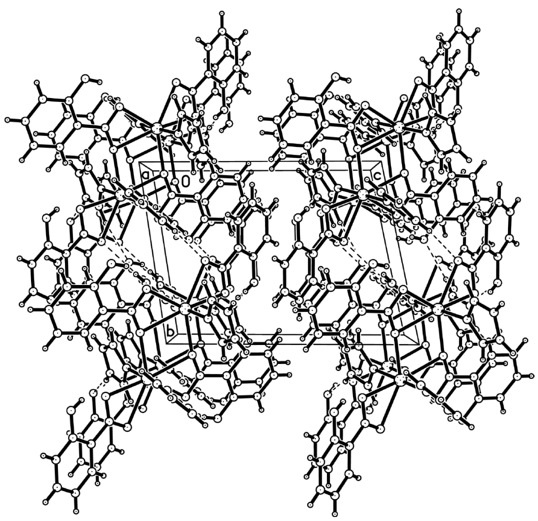


Figure 2
The packing of (I). Dashed lines indicate hydrogen bonds.

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.699, T_{max} = 0.826
9420 measured reflections
4661 independent reflections
4211 reflections with $I > 2\sigma(I)$
R_{int} = 0.016
θ_{max} = 27.5°
$h = -12 \rightarrow 12$
$k = -11 \rightarrow 12$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.062
S = 1.08
4661 reflections
280 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0349P)^2 + 0.2931P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.80 e Å^{-3}
$\Delta\rho_{min}$ = −0.50 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cd1—N1	2.251 (2)	Cd1—O4	2.404 (2)
Cd1—N3	2.232 (2)	Cd1—O5^i	2.381 (2)
Cd1—O1	2.538 (2)	Cd1—O5	2.530 (2)
Cd1—O2	2.390 (2)		
N1—Cd1—O1	89.93 (7)	N3—Cd1—O2	93.93 (7)
N1—Cd1—O2	82.76 (7)	N3—Cd1—O4	91.57 (7)
N1—Cd1—O4	91.76 (7)	N3—Cd1—O5	96.49 (7)
N1—Cd1—O5	86.54 (7)	N3—Cd1—O5^i	88.45 (7)
N1—Cd1—O5^i	90.44 (7)	N3—Cd1—N1	176.44 (7)
N3—Cd1—O1	89.10 (7)		

Symmetry code: (i) $-x, 2-y, -z$.

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O3—H21···O2	0.82	1.75	2.486 (2)	149
O6—H22···O4	0.82	1.82	2.547 (2)	147
N2—H23···O1^{ii}	0.86	2.13	2.847 (3)	140
N4—H24···O3^{iii}	0.86	2.03	2.884 (3)	172

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

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H atoms were placed in calculated positions, with C—H = 0.93 Å (aromatic), N—H = 0.86 Å (imidazole) and O—H = 0.82 Å (hydroxyl). All H atoms were refined using the riding model approximation, with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C, N) and $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(O).

Data collection: *RAPID-AUTO* (Rigaku Corporation, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

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