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# $\mu$-3-(Carboxylatomethoxy)phenylsulfanylacetato-$\kappa O$:$O'$-bis[aquabis(1,10-phenanthroline-$\kappa^2 N,N'$)-cadmium(II)] dinitrate

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The reaction of cadmium nitrate with sodium 3-(carboxymethoxy)phenylsulfanylacetate in the presence of 1,10-phenanthroline yields the title centrosymmetric dinuclear salt, $[Cd_2(C_{10}H_9O_5S)(C_{12}H_8N_2)_4(H_2O)_2](NO_3)_2$, with the disordered ($C_{10}H_9O_5S$) ligand lying on a special position of $\overline{1}$ site symmetry. In the centrosymmetric dication, the water-coordinated Cd atom is chelated by the two heterocyclic ligands; it is covalently bonded to the O atom of the dicarboxylate in a distorted octahedral geometry.

**Key indicators**

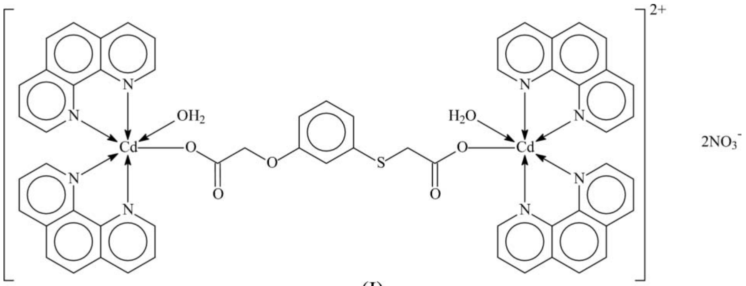
Single-crystal X-ray study
$T$ = 295 K
Mean $\sigma$(C–C) = 0.007 Å
Disorder in main residue
$R$ factor = 0.044
$wR$ factor = 0.140
Data-to-parameter ratio = 14.9

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

## Comment

In a previous study, cadmium nitrate was reacted with the sodium salt of 4-(carboxymethoxy)phenylsulfanylacetic acid in the presence of 1,10-phenanthroline to afford a five-coordinate mononuclear dihydrate in which the deprotonated dicarboxylic acid binds to the bis-chelated metal centre through only one carboxylate group. In the crystal structure, half the metal centres are coordinated by the $-OCH_2CO_2$ group and the other half by the $-SCH_2CO_2$ group, the ligand being disordered (Huo *et al.*, 2005).

With 3-(carboxymethoxy)phenylsulfanylacetic acid, the analogous synthesis yielded the title centrosymmetric dinuclear compound, (I), in which one carboxylate functions as a bridge between the two heterocycle-chelated Cd atoms; the charge is balanced by two nitrate ions (Fig. 1). The carboxylate and water O atoms are *cis* to each other in a distorted octahedral polyhedron around the metal centre (Table 1).



(I)

The dication is hydrogen bonded to the anions (Table 2); the packing appears to be compact, as there are no solvent-accessible voids.

## Experimental

Cadmium nitrate tetrahydrate (0.62 g, 2 mmol) and 1,10-phenanthroline (0.80 g, 4 mmol) were added to a hot aqueous solution of 3-(carboxymethoxy)phenylsulfanylacetic acid (0.48 g, 2 mmol). Sodium hydroxide (0.2 $M$) was added until the pH of the mixture was 6. The solution was set aside and colourless prismatic crystals of (I) deposited from the solution over a week. CHN analysis, calculated for $C_{58}H_{44}Cd_2N_{10}O_{13}S$: C 51.71, H 3.27, N 10.40%; found: C 51.74, H 3.24, N 10.36%.

### *Crystal data*

$[Cd_2(C_{10}H_9O_5S)(C_{12}H_8N_2)_4(H_2O)_2](NO_3)_2$
$M_r = 1345.89$
Triclinic, $P\bar{1}$
$a = 8.157$ (2) Å
$b = 12.355$ (3) Å
$c = 13.932$ (3) Å
$\alpha = 96.70$ (3)°
$\beta = 100.37$ (3)°
$\gamma = 101.49$ (3)°
$V = 1336.4$ (6) Å$^3$
$Z = 1$
$D_x = 1.672$ Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 12523 reflections
$\theta = 3.0$–27.5°
$\mu = 0.91$ mm$^{-1}$
$T = 295$ (2) K
Block, colourless
0.32 × 0.28 × 0.20 mm

### *Data collection*

Rigaku R-AXIS RAPID IP diffractometer
$\omega$ scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min} = 0.549$, $T_{max} = 0.839$
13173 measured reflections
6052 independent reflections
5103 reflections with $I > 2\sigma(I)$
$R_{int} = 0.020$
$\theta_{max} = 27.5°$
$h = -10 \rightarrow 10$
$k = -15 \rightarrow 16$
$l = -15 \rightarrow 18$

### *Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.140$
$S = 1.07$
6052 reflections
407 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.096P)^2 + 0.2549P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.92$ e Å$^{-3}$
$\Delta\rho_{min} = -0.81$ e Å$^{-3}$

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

| | | | |
|---|---|---|---|
| Cd1—O1 | 2.267 (3) | Cd1—N4 | 2.332 (3) |
| Cd1—O1*w* | 2.312 (3) | S1—C26 | 1.75 (1) |
| Cd1—N1 | 2.359 (3) | S1—C27 | 1.716 (6) |
| Cd1—N2 | 2.364 (3) | O1′—C26′ | 1.46 (1) |
| Cd1—N3 | 2.374 (3) | O1′—C31 | 1.462 (6) |
| | | | |
| O1—Cd1—O1*w* | 86.8 (1) | O1*w*—Cd1—N4 | 112.1 (1) |
| O1—Cd1—N1 | 111.7 (1) | N1—Cd1—N2 | 70.7 (1) |
| O1—Cd1—N2 | 86.5 (1) | N1—Cd1—N3 | 92.4 (1) |
| O1—Cd1—N3 | 155.0 (1) | N1—Cd1—N4 | 153.0 (1) |
| O1—Cd1—N4 | 88.6 (1) | N2—Cd1—N3 | 108.6 (1) |
| O1*w*—Cd1—N1 | 87.6 (1) | N2—Cd1—N4 | 94.0 (1) |
| O1*w*—Cd1—N2 | 152.8 (1) | N3—Cd1—N4 | 70.9 (1) |
| O1*w*—Cd1—N3 | 87.8 (1) | | |

**Table 2**
Hydrogen-bond geometry (Å, °).

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1*W*—H1*w*1···O2 | 0.82 | 1.97 | 2.692 (6) | 146 |
| O1*W*—H1*w*2···O3 | 0.82 | 2.12 | 2.904 (8) | 161 |

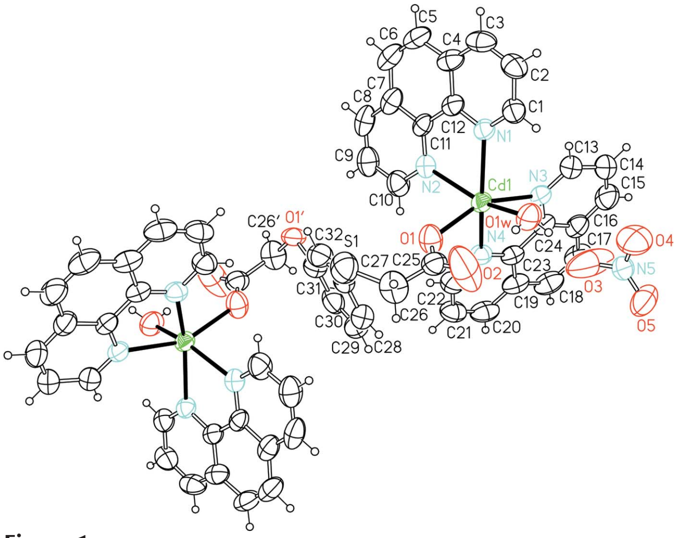


**Figure 1**
A plot of (I), with displacement ellipsoids drawn at the 50% probability level and H atoms shown as spheres of arbitrary radii. One component of the disorder has been omitted for clarity. Only one nitrate anion of the formula unit is shown. [Symmetry code for unlabelled atoms: $1 - x$, $1 - y$, $1 - z$]

The $C_{10}H_9O_5S$ ligand, which is a 1,3-disubstituted benzene, is disordered about a centre of inversion; the ring and the –$OCH_2$ and –$SCH_2$ units are disordered but not the –$CO_2$ units, which are related by exact inversion symmetry. The C27–C32 ring was refined as a rigid hexagon with sides of 1.39 Å, and the displacement parameters were restrained to be approximately isotropic. For the –$OCH_2CO_2$ arm, the two C—O distances were restrained to 1.45 (1) Å and for the –$SCH_2CO_2$ arm, the two S—O distances were restrained to 1.75 (1) Å. The displacement parameters of the methylene C atoms were constrained to be equal to each other. Carbon-bound H atoms were placed in calculated positions, with C—H$_{aromatic}$ = 0.93 and C—H$_{aliphatic}$ = 0.97 Å, and were included in the refinement in the riding-model approximation. Water H atoms were also similarly treated, with O—H 0.82 Å. For all H atoms, $U_{iso}(H) = 1.2U_{eq}$(parent).

Data collection: *RAPID AUTO* (Rigaku, 1998); cell refinement: *RAPID AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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