

Two Cd and Hg peroxodisulfate complexes with aromatic amines

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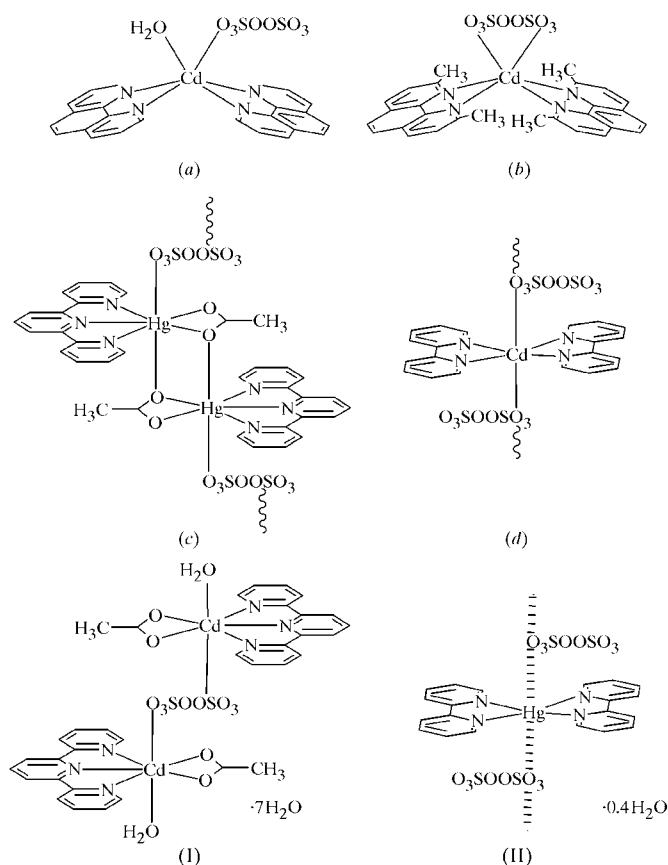
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The crystal structures of two complexes containing the peroxodisulfate anion are reported, namely μ -peroxodisulfato-1 κ O:2 κ O'-bis[(acetato- κ^2 O,O')aqua(2,2':6':2''-terpyridine- κ^3 N,N',N'')cadmium(II)] heptahydrate, [Cd₂(C₂H₃O₂)₂(S₂O₈)(C₁₅H₁₁N₂)₂(H₂O)₂].7H₂O, (I), and *catena*-poly[[[bis(2,2'-bipyridine- κ^2 N,N'')mercury(II)]- μ -peroxodisulfato- κ^2 O:O'] 0.4-hydrate], {[Hg(C₁₀H₈N₂)₂(S₂O₈)]·0.4H₂O}_n, (II). In both structures, the anion behaves as a bridge, linking neighbouring coordination polyhedra in two different ways, either tightly bound to the heptacoordinated Cd²⁺ cation forming neatly separated dimeric entities in (I) or across a shorter O—S—O path producing weakly connected chains by way of 'semi-coordination' to the Hg²⁺ cations in (II).

Comment

In two preceding articles by the present authors (Harvey, Baggio, Garland, Burton & Baggio, 2001; Harvey, Baggio, Garland & Baggio, 2001) dealing with the binding behaviour of peroxodisulfate towards some group XII cations (Cd and Hg), the anion was shown to adopt a variety of different coordination modes (see scheme), namely monodentate (case *a*), chelate (case *b*), bridging polymeric (along the anion, case *c*) or bridging polymeric (across the anion, case *d*). Some experiments aimed at coordinating this anion to Zn have so far been unsuccessful, although they provide the first examples of peroxodisulfate complexes with the anion fulfilling the role of a stabilizing counter-ion (Harvey *et al.*, 2004). Continuation of our synthetic trials with group XII metals in combination with nitrogenated ligands has now afforded two new structures, presented here, which display some slightly different binding modes for peroxodisulfate (S₂O₈), *viz.* bridging dimeric (along the anion), as in [Cd(ac)₂(S₂O₈)(tpy)(H₂O)].7H₂O, (I), and

semicoordinated polymeric (across the anion), as in [Hg-(S₂O₈)(bpy)₂].0.4H₂O, (II), where tpy is 2,2':6':2''-terpyridine, bpy is 2,2'-bipyridine and ac is acetate.



Structure (I) consists of two independent though similarly formulated dinuclear units, each built up of two symmetry-related [Cd(ac)(S₂O₈)_{0.5}(tpy)(H₂O)] groups. The symmetry centre halves the peroxodisulfate anion, which acts as a link between the two cationic centres (Fig. 1). The Cd^{II} ion is coordinated in the form of a pentagonal bipyramid by the tridentate tpy group through amino atoms N1_n, N2_n and N3_n (*n* = A or B in the two different moieties), and the bidentate ac through carboxylate atoms O1_m and O2_m (*m* = X or Y). The two planar groups bind in a coplanar fashion and define the (planar) pentagonal base, with mean deviations from the best plane of 0.07 (1) and 0.05 (1) Å for moieties A and B, respectively. In addition, the two independent base planes are nearly parallel to each other, subtending a dihedral angle of 4.5 (1)°. The external apical position is occupied by one aqua ligand [Cd1—O1W = 2.354 (4) Å and Cd2—O2W = 2.328 (5) Å], while the internal position corresponds to the outermost O atom in the stretched peroxodisulfate [Cd1—O1A = 2.486 (4) Å and Cd2—O1B = 2.412 (4) Å]. These apical axes are nearly perpendicular to the basal planes, to which they subtend angles of 88.2 (1)/85.7 (1) and 88.6 (1)/84.3 (1)° in A and B, respectively.

As observed in almost all the structures where they act as a ligand, the tridentate tpy groups are subject to some strain, being obliged to stretch inwards so as to be able to chelate the

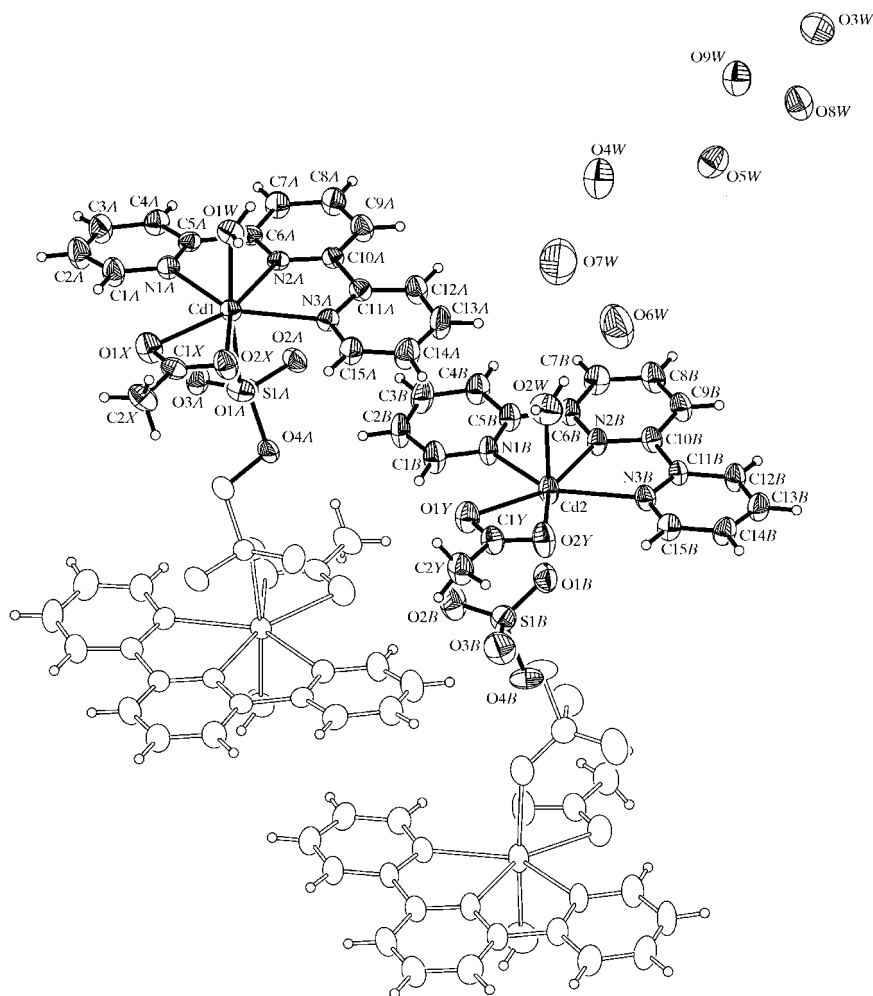


Figure 1

A molecular drawing of the two non-equivalent centrosymmetric dinuclear moieties in (I). Displacement ellipsoids are drawn at the 40% probability level. Full ellipsoids denote the independent atoms and empty ellipsoids the symmetry-related atoms.

cation. Thus, all the sp^2 angles centred at C5, C6, C10 and C11 (A/B) show a clear asymmetry, those facing the cation being some 5° smaller than the other two. As a possible consequence of this, the tpy molecules deviate somewhat from planarity, mainly through rotations of the aromatic rings along the axis connecting them [maximum angles to the mean plane normal are $8.8(1)$ and $5.4(1)^\circ$ for A and B, respectively]. Interactions to the metal (Cd–O distances) and the C–O bonds are also self-consistently similar.

Finally, the peroxodisulfate anions bridge the cationic centres, connecting them into the two similar dinuclear moieties building up the structure. There is, however, a qualitative conformational difference between them: each terminal SO_3 group in the anion leans towards its neighbouring aromatic amine in moiety A, but towards the opposite ac group in moiety B. This difference is shown in Fig. 2 *via* a least-squares superposition of both molecules. In structure B, internal atom O4B of the peroxodisulfate anion is slightly disordered over three clear positions, of which only the most populated one has been chosen for this representation (see below). The long molecular units [with separations of $14.79(1)$ and $14.75(1)$ Å between the outermost aqua O atoms in units

A and B, respectively] are linear and almost parallel to each other, the longest axes subtending an angle of $11.1(2)^\circ$ to each other. They pack with the bulky aromatic amines fitting into the voids of their neighbours, but without significant π – π interaction among pyridine groups. The crystal packing is stabilized through seven independent crystallization water molecules which, together with the two aqua ligands, provide a rich collection of H atoms to which the ac and peroxodisulfate O atoms act as acceptors. Unfortunately, it was not possible to locate the hydration water H atoms with any degree of confidence, although it was possible to locate those corresponding to the bound water. In spite of this limitation, the three-dimensional linkages are clear, as shown in Fig. 3, where the water molecules can be seen in the zone $z \sim 0.50$. All the water O atoms take part in this hydrogen-bonded network, with O...O separations in the expected range of $2.75(1)$ – $3.11(1)$ Å.

The structure bears striking similarities to an anhydrous Hg homologue, $Hg(ac)_2(S_2O_8)(tpy)$, (III) (case *c*; Harvey, Baggio, Garland & Baggio, 2001). Among the similarities, we note the behaviour of the central O atom in one of the peroxodisulfate units [O4B in (I) and O4F in (III)]: atom O4B is split over

three different sites, while in its Hg counterpart, atom O4F presents an abnormally prolate displacement ellipsoid, some 2.5 times larger than those corresponding to the terminal O atoms. This suggests that the site might be located in a very shallow energy minimum. Both structures present two independent dinuclear entities formed by two heptacoordinated cations with a pentagonal-bipyramidal environment, both having one tpy and one ac moiety in the planar base and a centrosymmetric peroxodisulfate anion stretching all along and joining two cationic centres. However, in (III), these elemental units link to each other through one of the ac O atoms to determine infinite one-dimensional structures, while in (I), this polymeric link is prevented by the site being occupied by a ‘terminal’ aqua ligand.

Structure (II) (Fig. 4) consists of a Hg^{II} centre coordinated to two bpy groups, which lie about halfway between a parallel and a perpendicular disposition [the dihedral angle between the best planes is 41.8 (3)°]. A fractional hydration water molecule [site-occupancy factor = 0.40 (1)] completes the structure. As in the tpy ligands in (I), the bpy groups show chelation strain, both in the small value of the N–C–Csp² angle facing the cation and in the rotation of the pyridine groups around the C5–C6 bonds [4.1 (1) and 5.2 (1)° for units

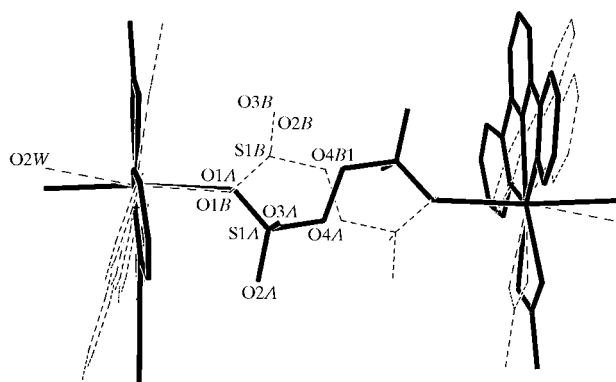


Figure 2
A schematic superposition diagram, showing the different orientation of the peroxodisulfate anions in the independent moieties in (I).

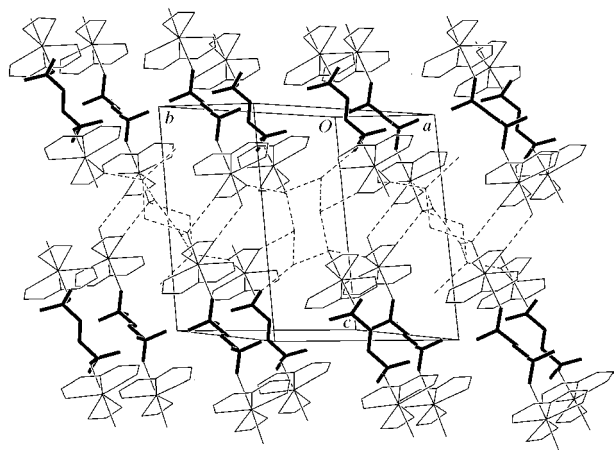


Figure 3
A packing view of (I), showing the hydrogen-bonding scheme suggested by OW...OW contacts (see *Comment* for details).

A and *B*, respectively]. These four Hg–N interactions, in the range 2.229 (3)–2.340 (3) Å, provide most of the coordination involvement of Hg, as a bond-valence calculation (Brown & Altermatt, 1985) shows: the four amino N atoms provide *ca* 1.86 of the expected total of 2. The rest is provided by two peroxodisulfate O atoms lying at very long ‘semicoordination’ distances [2.703 (3)–2.953 (3) Å] from the cation. A search of the Cambridge Structural Database (CSD, November 2004 version; Allen, 2002) revealed that tetracoordinated Hg^{II} cations (according to CSD standard defaults) bound to four N atoms tend to display unusual coordination geometries (sometimes with semicoordinated O or N ligands at distances in the range 2.70–3.00 Å), as well as bond-valence balances far less than ideal. In Table 5, we present a summary of the results found, which therefore define the HgN₄(O₂) configuration in (II) as normal.

These latter interactions link [Hg(bpy)₂] centres together to form chains along the *a* axis. The anion does not coordinate alongside, as in structure (I), but sideways, mainly along the shortest bridge, *viz.* Hg–O1–S1–O3–Hg. A complementary interaction for chain stabilization is the hydrogen bonding, in which the water molecule takes part (Fig. 5 and Table 4), linking neighbouring peroxodisulfate groups. A close relative to structure (II) is its Cd homologue [Cd(bpy)₂(S₂O₈)·H₂O, (IV) (case *d*; Harvey, Baggio, Garland & Baggio, 2001). They share the same distorted basal coordination of the two bpy groups, while the main structural differences consist in the apical coordination of the anion, which in (IV) is a strong Cd–O bond with a distance only slightly longer than the basal ones. There is, in addition, a different character to

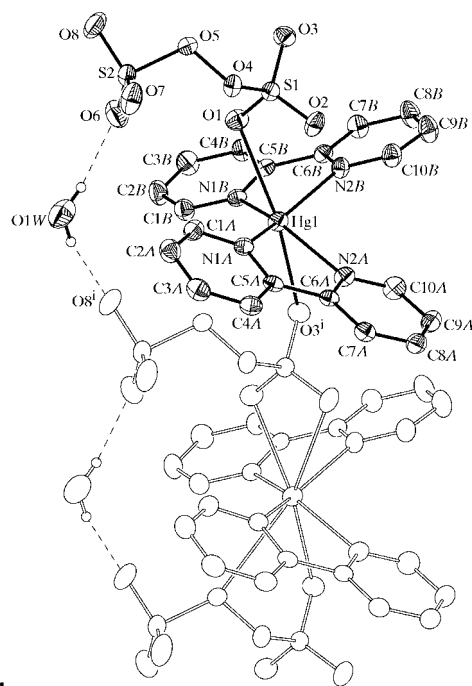


Figure 4
A molecular drawing of the polymeric structure of (II). Displacement ellipsoids are drawn at the 40% probability level. Full ellipsoids denote the independent atoms and empty ellipsoids the symmetry-related atoms. Note the intrachain hydrogen bond in which atom O1W takes part. [Symmetry code: (i) 1 + *x*, *y*, *z*.]

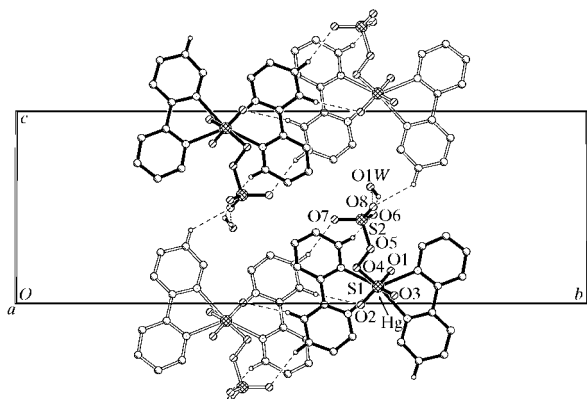


Figure 5
A packing view of (II) down [100], showing the hydrogen-bonding contacts. Note that the O1W intrachain bonds are almost vertical in this view and are therefore rather difficult to see.

the water-based (O1W) hydrogen bonds: while in (II) these are intrachain and provide chain cohesion, in (IV) they are interchain linking neighbouring chains together.

Being strictly centrosymmetric, the peroxodisulfate anions in (I) share the property of presenting a perfectly planar S—O—O—S nucleus, which also contains one of the three terminal O atoms (O_{term}) at each side (in a *trans* position to the core), configuring a six-membered O—S—O—O—S—O planar entity (see previous discussion in the two preceding articles of this series; Harvey, Baggio, Garland, Burton & Baggio, 2001). These latter O atoms are special in that they subtend much smaller $O_{\text{term}}\text{—S1—O}_{\text{core}}$ angles than the remaining two O_{term} atoms, as can be confirmed from the values reported in Table 1. That this must be a highly preferred conformation is also concluded from the fact that, even when the conformation is not well determined, as in the disordered *B* moiety in structure (I), the most populated orientation is precisely that which fulfils the referred conditions. The peroxodisulfate anion in (II) instead departs noticeably from planarity [$S1\text{—}O4\text{—}O5\text{—}S2 = -54.4(2)^\circ$]. What both compounds share, however, is the special characteristic of the ‘quasi-*trans*’ O atoms, *i.e.* those subtending the smallest $O_{\text{core}}\text{—}O_{\text{core}}\text{—}S\text{—}O_{\text{term}}$ torsion angle, which also subtend the smallest $O_{\text{term}}\text{—}S1\text{—}O_{\text{core}}$ angle (Table 3).

Experimental

Compound (I) was obtained by direct mixing of a methanolic solution of 2,2':6',2''-terpyridine with an aqueous solution of cadmium acetate dihydrate and potassium peroxodisulfate in a 1:1:2 molar ratio. Irrespective of mixing order, the preparation readily gave well developed plates of the same compound. Compound (II) was obtained through the slow diffusion of a methanolic solution of 2,2'-bipyridine and mercury(II) acetate into an aqueous solution of potassium peroxodisulfate, all at concentrations of 0.025 *M*. The preparation was kept in a dark environment in order to avoid unwanted reactions of the Hg^{II} ion. Although very small colourless needles began to appear immediately at the interface of the two solutions, adequate crystals suitable for X-ray diffraction required almost two months to grow. All starting materials were of reagent quality and were used without further purification.

Compound (I)

Crystal data

$[\text{Cd}_2(\text{C}_2\text{H}_3\text{O}_2)_2(\text{S}_2\text{O}_8)(\text{C}_{15}\text{H}_{11}\text{N}_3)_2 \cdot (\text{H}_2\text{O})_2] \cdot 7\text{H}_2\text{O}$
 $M_r = 1163.69$
 Triclinic, $P\bar{1}$
 $a = 11.059(6) \text{ \AA}$
 $b = 14.254(7) \text{ \AA}$
 $c = 14.615(8) \text{ \AA}$
 $\alpha = 87.526(8)^\circ$
 $\beta = 74.913(8)^\circ$
 $\gamma = 87.249(8)^\circ$
 $V = 2221(2) \text{ \AA}^3$

$Z = 2$
 $D_x = 1.740 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 Cell parameters from 2134 reflections
 $\theta = 3.0\text{--}24.5^\circ$
 $\mu = 1.14 \text{ mm}^{-1}$
 $T = 297(2) \text{ K}$
 Plate, colourless
 $0.40 \times 0.30 \times 0.15 \text{ mm}$

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\text{min}} = 0.68$, $T_{\text{max}} = 0.84$
 18 276 measured reflections

9478 independent reflections
 6121 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.037$
 $\theta_{\text{max}} = 28.0^\circ$
 $h = -14 \rightarrow 14$
 $k = -18 \rightarrow 18$
 $l = -18 \rightarrow 18$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.046$
 $wR(F^2) = 0.121$
 $S = 0.87$
 9474 reflections
 619 parameters

H atoms treated by a mixture of independent and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.067P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.005$
 $\Delta\rho_{\text{max}} = 0.67 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.47 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (\AA , $^\circ$) for (I).

Cd1—N2A	2.326 (3)	Cd2—O1B	2.408 (4)
Cd1—O1W	2.353 (4)	Cd2—O2Y	2.439 (4)
Cd1—N1A	2.385 (4)	S1A—O2A	1.428 (3)
Cd1—O1X	2.390 (3)	S1A—O3A	1.426 (3)
Cd1—N3A	2.393 (4)	S1A—O1A	1.457 (3)
Cd1—O2X	2.407 (3)	S1A—O4A	1.645 (3)
Cd1—O1A	2.489 (3)	O4A—O4A ⁱ	1.477 (5)
Cd2—O2W	2.332 (4)	S1B—O2B	1.416 (4)
Cd2—N2B	2.352 (4)	S1B—O3B	1.421 (4)
Cd2—N3B	2.393 (4)	S1B—O1B	1.426 (4)
Cd2—O1Y	2.390 (3)	S1B—O4B3	1.71 (2)
Cd2—N1B	2.402 (4)	O4B3—O4B3 ⁱⁱ	1.46 (3)
O2A—S1A—O4A	97.8 (2)	O2B—S1B—O4B3	85.3 (5)
O3A—S1A—O4A	106.4 (2)	O3B—S1B—O4B3	112.4 (6)
O1A—S1A—O4A	105.1 (2)	O1B—S1B—O4B3	110.4 (6)
O1A—S1A—O4A—O4A ⁱⁱⁱ	-15.3 (1)	O1B—S1B—O4B3—O4B3 ^{iv}	-42.5 (4)
O2A—S1A—O4A—O4A ⁱⁱⁱ	103.7 (1)	O2B—S1B—O4B3—O4B3 ^{iv}	72.9 (5)
O3A—S1A—O4A—O4A ⁱⁱⁱ	-135.6 (2)	O3B—S1B—O4B3—O4B3 ^{iv}	-171.7 (7)

Symmetry codes: (i) $-x + 1, -y + 2, -z + 2$; (ii) $-x + 2, -y + 1, -z + 2$; (iii) $-x + 1, -y + 2, -z + 1$; (iv) $-x, -y + 1, -z + 1$.

Table 2

Hydrogen-bond geometry (\AA , $^\circ$) for (I).

$D\text{—}H \cdots A$	$D\text{—}H$	$H \cdots A$	$D \cdots A$	$D\text{—}H \cdots A$
O1W—H1WA \cdots O2X ⁱⁱⁱ	0.82 (4)	1.99 (4)	2.774 (5)	159 (4)
O1W—H1WB \cdots O3W ^{iv}	0.82 (4)	2.07 (4)	2.866 (5)	165 (5)
O2W—H2WA \cdots O6W	0.83 (4)	1.93 (4)	2.754 (6)	171 (6)
O2W—H2WB \cdots O4W ^v	0.82 (4)	2.04 (4)	2.827 (6)	161 (6)

Symmetry codes: (iii) $-x + 1, -y + 2, -z + 1$; (iv) $-x, -y + 1, -z + 1$; (v) $-x + 1, -y + 1, -z + 1$.

Compound (II)

Crystal data

[Hg(C ₁₀ H ₈ N ₂) ₂ (S ₂ O ₈)]·0.4H ₂ O	<i>D</i> _x = 2.112 Mg m ⁻³
<i>M</i> _r = 712.28	Mo <i>K</i> α radiation
Monoclinic, <i>P</i> 2 ₁ / <i>n</i>	Cell parameters from 2344 reflections
<i>a</i> = 7.3340 (11) Å	<i>θ</i> = 3.1–22.7°
<i>b</i> = 30.125 (6) Å	<i>μ</i> = 7.12 mm ⁻¹
<i>c</i> = 10.4340 (17) Å	<i>T</i> = 297 (2) K
<i>β</i> = 103.689 (15)°	Needle, colourless
<i>V</i> = 2239.8 (7) Å ³	0.50 × 0.20 × 0.10 mm
<i>Z</i> = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	4938 independent reflections
<i>φ</i> and <i>ω</i> scans	4000 reflections with <i>I</i> > 2σ(<i>I</i>)
Absorption correction: multi-scan (<i>SADABS</i> ; Sheldrick, 1996)	<i>R</i> _{int} = 0.033
<i>T</i> _{min} = 0.20, <i>T</i> _{max} = 0.50	<i>θ</i> _{max} = 28.1°
13 178 measured reflections	<i>h</i> = -9 → 9
	<i>k</i> = -37 → 39
	<i>l</i> = -13 → 11

Refinement

Refinement on <i>F</i> ²	H atoms treated by a mixture of independent and constrained refinement
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)] = 0.028	<i>w</i> = 1/[σ ² (<i>F</i> _o ²) + (0.035 <i>P</i>) ²]
<i>wR</i> (<i>F</i> ²) = 0.061	where <i>P</i> = (<i>F</i> _o ² + 2 <i>F</i> _c ²)/3
<i>S</i> = 0.84	(Δ/ <i>σ</i>) _{max} = 0.012
4938 reflections	Δ <i>ρ</i> _{max} = 1.41 e Å ⁻³
332 parameters	Δ <i>ρ</i> _{min} = -0.72 e Å ⁻³

Table 3

Selected geometric parameters (Å, °) for (II).

Hg1–N1 <i>B</i>	2.229 (3)	S1–O1	1.439 (3)
Hg1–N1 <i>A</i>	2.232 (3)	S1–O4	1.653 (3)
Hg1–N2 <i>A</i>	2.332 (3)	S2–O7	1.413 (3)
Hg1–N2 <i>B</i>	2.341 (3)	S2–O6	1.431 (3)
Hg1–O1	2.703 (3)	S2–O8	1.438 (3)
Hg1–O3 ⁱ	2.953 (3)	S2–O5	1.665 (3)
S1–O3	1.430 (3)	O4–O5	1.477 (3)
S1–O2	1.436 (3)		
O3–S1–O2	115.92 (18)	O7–S2–O6	115.1 (2)
O3–S1–O1	113.78 (17)	O7–S2–O8	115.4 (2)
O2–S1–O1	114.27 (17)	O6–S2–O8	114.7 (2)
O3–S1–O4	106.13 (15)	O7–S2–O5	106.41 (17)
O2–S1–O4	98.38 (15)	O6–S2–O5	106.43 (17)
O1–S1–O4	106.24 (15)	O8–S2–O5	96.09 (17)
S1–O4–O5–S2	125.56 (17)	O6–S2–O4–O5	112.3 (3)
O1–S1–O4–O5	-69.8 (2)	O7–S2–O5–O4	49.3 (3)
O2–S1–O4–O5	171.8 (2)	O8–S2–O5–O4	168.1 (2)
O3–S1–O4–O5	51.6 (2)		

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

Table 4

Hydrogen-bond geometry (Å, °) for (II).

<i>D</i> –H... <i>A</i>	<i>D</i> –H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> –H... <i>A</i>
O1 <i>W</i> –H1 <i>WB</i> ...O8 ⁱ	0.82 (2)	1.92 (7)	2.703 (13)	160 (16)
C7 <i>A</i> –H7 <i>AA</i> ...O2 ⁱⁱ	0.93	2.41	3.336 (5)	171
C8 <i>A</i> –H8 <i>AA</i> ...O7 ⁱⁱ	0.93	2.40	3.301 (5)	163
C1 <i>A</i> –H1 <i>AA</i> ...O6	0.93	2.60	3.430 (5)	150

Symmetry codes: (i) *x* + 1, *y*, *z*; (ii) -*x* + 1, -*y*, -*z* + 1.

H atoms defined by the stereochemistry were placed in their calculated positions and allowed to ride on their host C atoms, with

Table 5

Miscellaneous information on structures with HgN₄ centres.

CSD refcode	DA (°)†	CN‡	BVS§
BAYPUN	90.8	N4	2.21
BPYRHG	40.5	N4	1.83
COKDUB	80.4	N4	1.83
DOMDIS10	90.6	N4	2.10
ENHGPC10	62.7	N4	1.70
ICIYEY	41.0	N4	2.30
MAPDIQ	79.8	N4	1.87
MUXIY	63.8	N4	1.85
VANDAP	77.8	N4	2.15
ZEMRIS	87.6	N4	1.84
BOJXAZ	83.2	N4 + O2	2.03 + 0.15
IKUGAW	83.5	N4 + O2	2.02 + 0.13
NUGTUE	34.9	N4 + O2	1.96 + 0.22
(II) in this work	41.0	N4 + O2	1.86 + 0.16

† DA is the dihedral angle in the coordination polyhedron. ‡ CN is the coordination core (O atoms in the last four entries lie in the range 2.70–3.00 Å). § BVS is the bond-valence sum, according to Brown & Altermatt (1985).

aromatic C–H = 0.93 Å, methyl C–H = 0.96 Å, and *U*_{iso}(H) = 1.2*U*_{eq}(C) and 1.5*U*_{eq}(C) for aromatic and methyl H atoms, respectively. Those corresponding to the aqua ligand in (I) and to the partial hydration water molecule in (II) were located in a Fourier map and were refined with similarity restraints and a riding displacement parameter, with O–H = 0.82 (4) Å, H...H = 1.40 (4) Å and *U*_{iso}(H) = 1.2*U*_{eq}(O). The remaining water H atoms in (I) could not be confidently located and were accordingly omitted from the model. The rather high residual peak in (II) appears at less than 1 Å from the cation. Four reflections affected by beam-stop shadowing were excluded from the refinement in structure (I).

For both compounds, data collection: *SMART-NT* (Bruker, 2001); cell refinement: *SAINT-NT* (Bruker, 2000); data reduction: *SAINT-NT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-PC* (Sheldrick, 1994).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: GA1102). Services for accessing these data are described at the back of the journal.

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