

Synthesis and crystal structures of tetrakis(nitratomercurio)methane monohydrate and bis(sulphatomercurio)bis(aquamercurio)methane

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Abstract

The products of the reaction of Hofmann's base, $[\text{CHg}_4\text{O}(\text{OH})_2\text{OH}_2]_n$, with conc. nitric and sulphuric acid are tetrakis(nitratomercurio)methane monohydrate, $\text{C}(\text{HgNO}_3)_4 \cdot \text{H}_2\text{O}$ and bis(sulphatomercurio)bis(aquamercurio)methane, $\text{C}(\text{HgSO}_4)_2(\text{HgOH}_2)_2$. The structures were characterized by IR spectra and single crystal X-ray diffraction. In both structures the carbon atom is tetrahedrally surrounded by four mercury atoms.

Keywords: Mercury; Tetramercurated methane; X-ray structure

1. Introduction

Hofmann's base, $[\text{CHg}_4\text{O}(\text{OH})_2\text{OH}_2]_n$, originally formulated as $\text{C}_2\text{Hg}_6\text{O}_2(\text{OH})_2$ [1], gives with acids either its salts, $[\text{CHg}_4\text{O}(\text{OH}_2)]_n\text{X}_{2n}$ (X, e.g. halide, nitrate) or derivatives of tetramercurated methane, $\text{C}(\text{HgX})_4$, (X, e.g. halide, acetate, trifluoroacetate, cyanide).

The first known structures containing the CHg_4 -tetrahedral unit were those of tetrakis(trifluoroacetoxymmercurio)methane [2,3] and tetrakis(acetoxymmercurio)methane dihydrate [2,4]. Up to now only two other structures of tetramercurated methane derivatives have been published, tetrakis(cyanomercurio)methane monohydrate [5] and tetrakis(chloromercurio)methane dimethylsulphoxide solvate [6].

In this paper we report two new derivatives of tetramercurated methane: tetrakis(nitratomercurio)methane monohydrate, $\text{C}(\text{HgNO}_3)_4 \cdot \text{H}_2\text{O}$ (1), and bis(sulphatomercurio)bis(aquamercurio)methane, $\text{C}(\text{HgSO}_4)_2(\text{HgOH}_2)_2$ (2), obtained by the action of conc. nitric and sulphuric acids on Hofmann's base.

2. Experimental

The IR spectra in the region $4000\text{--}450\text{ cm}^{-1}$ were recorded on Perkin-Elmer FTIR spectrophotometer model 1600, using KBr discs.

2.1. Purification of Hofmann's base

Very pure Hofmann's base [1] was obtained by alternating acid and alkali digestion of the crude product. The suspension of the crude base (5 g) in dilute nitric acid (2 M, 100 ml) was heated with stirring (70–80°C, 15 min) and after cooling to room temperature, the white solid was filtered off, washed with nitric acid (0.01 M) and water. Afterwards, a sodium hydroxide digestion (2 M, 100 ml) was carried out analogously to that described above. The acid-alkali treatment was repeated until a sample of the base gave a clear solution with warm dilute acetic acid. The base is a lemon-yellow powder. Found: Hg, 90.85. $\text{CH}_4\text{Hg}_4\text{O}_4$. Calc.: Hg, 90.93%.

2.2. Syntheses of $\text{C}(\text{HgNO}_3)_4 \cdot \text{H}_2\text{O}$ (1) and $\text{C}(\text{HgSO}_4)_2(\text{HgOH}_2)_2$ (2)

Hofmann's base (2 g) was added in small portions to conc. nitric acid (50 ml) for 1 or conc. sulphuric acid

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(80 ml) for **2**. The suspensions were gently heated (ca. 50°C) and stirred for several minutes. After standing at room temperature for at least 2 weeks, colourless crystalline solids were filtered off, washed with dilute (0.01 M) nitric or sulphuric acid and ethanol and then dried in vacuo. Yield: for **1** 2.08 g (83%) and for **2** nearly quantitative. Loss of weight of **1** on drying (80°C, 20 mm Hg): found 1.65; calc. for the monohydrate: 1.67%. Anal. for **1**. Found: Hg, 74.51; N, 4.89. $\text{CH}_2\text{Hg}_4\text{N}_4\text{O}_{13}$. Calc.: Hg, 74.26; N, 5.19%. Anal. for **2**. Found: Hg, 76.90; S, 6.18. $\text{CH}_4\text{Hg}_4\text{O}_{10}\text{S}_2$. Calc.: Hg, 76.90; S, 6.15%.

2.3. Crystal structure determinations

Crystals of **1** became opaque when left in air, which was probably caused by the loss of the solvate water molecules. A crystal for X-ray analysis was therefore sealed inside a Lindemann glass capillary with some mother liquor.

For both compounds the X-ray diffraction data were collected at room temperature on a Philips PW1100 diffractometer using graphite-monochromatized Mo K α radiation ($\lambda = 0.71069 \text{ \AA}$). Unit cell dimensions were obtained by least squares refinement of the setting

angles of 20 reflections ($18 < 2\theta < 28^\circ$) and 18 reflections ($22 < 2\theta < 26^\circ$) for **1** and **2** respectively. The unit cell parameters and details of data collection and refinement are given in Table 1. Standard reflections monitored every 2 h indicated a continuous decay amounting to 13% for **1**, and no significant change in intensities for **2**. The intensities were corrected for decay, Lorentz, polarization and absorption effects. An empirical absorption correction with DIFABS [7] was made for **1**, and a Gaussian absorption correction for **2** was made with ACXR [8].

Positions of Hg atoms were determined by the Patterson method. Subsequent full-matrix least squares refinement on F , and difference Fourier maps revealed all non-hydrogen atoms. Hydrogen atoms could not be located and were not included in the structure factor calculations. Only Hg atoms in **1**, and all non-H atoms except the C atom in **2** were refined anisotropically. In spite of the sufficient number of reflections for anisotropic refinement of all atoms in **1**, some light atoms did not have well-defined temperature ellipsoids. This is due to the poor quality of the data because of crystal decomposition and the use of an empirical absorption correction for a crystal with a very high linear absorption coefficient. Nevertheless, the absorption cor-

Table 1
Crystal data, data collection parameters and refinement details for $\text{C}(\text{HgNO}_3)_4 \cdot \text{H}_2\text{O}$ (**1**) and $\text{C}(\text{HgSO}_4)_2(\text{HgOH})_2$ (**2**)

	1	2
Empirical formula	$\text{CH}_2\text{Hg}_4\text{O}_{13}\text{N}_4$	$\text{CH}_4\text{Hg}_4\text{O}_{10}\text{S}_2$
Formula weight	1080.4	1042.5
Crystal system	Monoclinic	Monoclinic
Space group	$P2_1/n$	$C2/c$
a (Å)	14.637(4)	12.942(4)
b (Å)	11.604(4)	7.917(1)
c (Å)	7.953(3)	11.944(2)
β (deg)	104.54(2)	116.31(1)
V (Å ³)	1307.5(8)	1097.0(4)
Z	4	4
D_{calc} (g cm ⁻³)	5.488	6.312
$F(000)$	1840	1768
Face indices, distance from centroid (mm)		(110), (1 - 10), (-110), (-1 - 10) 0.021; (001), (00 - 1) 0.117;
μ (cm ⁻¹)	452.2	542.3
Min., max. transmission coefficients	0.689, 1.918	0.077, 0.208
Range of collected data (deg)	$2 < \theta < 30$	$2 < \theta < 30$
Scan type	θ - 2θ	ω
Scan speed (deg s ⁻¹)	0.04	0.04
Scan width (deg)	1.2	1.0
Number of collected reflections	3146	1332
Number of unique reflections with $I > 4\sigma(I)$	2693	1145
Number of parameters refined	109	75
$R = \sum F_o - F_c / \sum F_o $	0.078	0.031
$R_w = [\sum w(F_o - F_c)^2 / \sum w(F_o)^2]^{1/2}$	0.098	0.041
Goodness of fit	0.93	1.02
Weighting scheme	$w = 1/(\sigma^2(F) + 0.00950F^2)$	$w = 1/(\sigma^2(F) + 0.00055F^2)$
Largest final shift/estimated standard deviation	0.001	0.001
Largest final $\Delta\rho$ (e Å ⁻³)	5.6 (1.4 Å from Hg)	1.71 (1.5 Å from Hg)

Table 2

Positional parameters and equivalent isotropic thermal parameters (Hg atoms) and isotropic thermal parameters (from C to O1) (\AA^2), with estimated standard deviations in parentheses for $\text{C}(\text{HgNO}_3)_4 \cdot \text{H}_2\text{O}$ (1)

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}^a or U_{iso}
Hg1	0.50425(6)	0.26874(9)	0.56109(13)	0.0161(3) ^a
Hg2	0.37578(6)	0.08652(8)	0.73970(12)	0.0150(3) ^a
Hg3	0.27161(6)	0.30216(8)	0.47327(13)	0.0165(2) ^a
Hg4	0.40379(7)	0.36527(8)	0.86742(13)	0.0164(3) ^a
C	0.384(2)	0.253(2)	0.656(3)	0.009(4)
N1	0.625(2)	0.385(2)	0.385(4)	0.031(5)
O11	0.621(2)	0.279(2)	0.454(3)	0.027(4)
O12	0.690(2)	0.400(3)	0.317(4)	0.045(6)
O13	0.571(2)	0.456(3)	0.410(5)	0.055(8)
N2	0.434(2)	-0.137(2)	0.881(3)	0.021(4)
O21	0.353(1)	-0.078(2)	0.825(3)	0.020(4)
O22	0.425(2)	-0.232(2)	0.939(3)	0.034(5)
O23	0.509(2)	-0.092(3)	0.883(4)	0.049(7)
N3	0.150(2)	0.460(2)	0.240(3)	0.018(4)
O31	0.155(2)	0.348(2)	0.266(3)	0.026(4)
O32	0.090(2)	0.488(2)	0.103(3)	0.031(5)
O33	0.203(2)	0.525(3)	0.340(4)	0.050(7)
N4	0.345(2)	0.534(2)	1.083(3)	0.018(4)
O41	0.425(2)	0.498(2)	1.060(3)	0.025(4)
O42	0.353(2)	0.616(2)	1.187(4)	0.036(5)
O43	0.271(2)	0.490(2)	1.009(3)	0.031(5)
O1	0.832(2)	0.232(2)	0.649(4)	0.039(6)

$$^a U_{\text{eq}} = (1/3) \sum_j \sum_i U_{ij} a_i^* a_j^* a_i \cdot a_j$$

reaction with DIFABS gave better results than did a Gaussian correction. The crystal of 1 was irregularly shaped, with 13 ill-defined faces and of approximate size $0.4 \times 0.4 \times 0.3 \text{ mm}^3$. The empirical absorption correction significantly lowered the values of R_{int} (from 0.150 to 0.049) and R at that stage of refinement (from 0.151 to 0.092). Atomic scattering factors and anomalous dispersion coefficients were taken from *International Tables for X-ray Crystallography* [9]. No extinction correction was made. Computing was carried out on an IBM PC/AT-compatible computer using SHELX76 [10] and SHELXS86 [11] programs.

Table 3

Positional parameters and equivalent isotropic thermal parameters (\AA^2), with estimated standard deviations in parentheses for $\text{C}(\text{HgSO}_4)_2(\text{HgOH}_2)_2$ (2)

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}^a or U_{iso}
Hg1	0.13880(4)	0.10885(6)	0.31859(4)	0.0168(1)
Hg2	0.00300(4)	-0.19884(6)	0.10754(4)	0.0168(2)
S	0.2207(2)	0.4868(4)	0.3601(3)	0.0152(8)
O1	0.2613(7)	0.3047(12)	0.3807(9)	0.021(3)
O2	0.3223(8)	0.5913(14)	0.3904(8)	0.027(4)
O3	0.1387(10)	0.5068(16)	0.2296(10)	0.039(4)
O4	0.1695(9)	0.5221(15)	0.4461(10)	0.032(4)
O5	0.0084(8)	-0.3220(12)	-0.0484(9)	0.023(3)
C	0	-0.0545(25)	0.25	0.019 ^b

$$^a U_{\text{eq}} = (1/3) \sum_j \sum_i U_{ij} a_i^* a_j^* a_i \cdot a_j$$

^bAtom was refined isotropically.

Atomic coordinates for 1 and 2 are given in Tables 2 and 3 respectively. Lists of anisotropic thermal parameters and full lists of bond distances and angles have been deposited at the Cambridge Crystallographic Data Centre. Lists of structure factors are available from the authors.

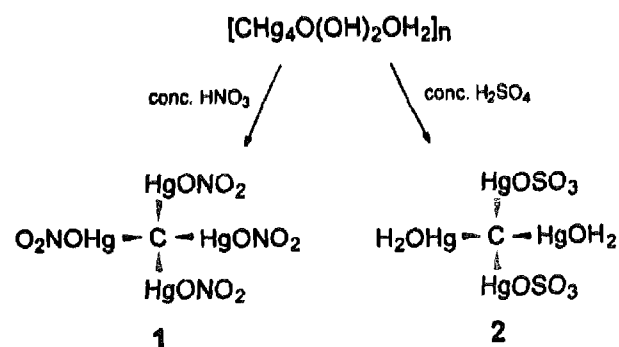
3. Results and discussion

The products of the reaction of Hofmann's base with conc. nitric and sulphuric acids are tetrakis(nitratomercurio)methane monohydrate, $\text{C}(\text{HgNO}_3)_4 \cdot \text{H}_2\text{O}$ (1) and bis(sulphatomercurio)bis(aquamercurio)methane, $\text{C}(\text{HgSO}_4)_2(\text{HgOH}_2)_2$ (2), Scheme 1.

Compounds 1 and 2 are colourless crystalline solids characterized by analytical data, and IR spectra and single crystal X-ray diffraction. 1 and 2 are insoluble and cannot be recrystallized. Good quality crystals suitable for the crystallographic investigation were obtained by prolonged standing of the suspensions in concentrated acids (approx. 2 months for 1 and even more for 2).

The IR spectral data are consistent with the covalent type of nitrate and sulphate groups bound to mercury in compounds 1 and 2 respectively. The strong absorption bands observed at 1501 cm^{-1} and at 1278 cm^{-1} as well as the medium band at 1010 cm^{-1} indicate the presence of the unidentate nitrate group in 1 [12,13]. In the IR spectrum of 2, very strong bands at 1120 and 1019 cm^{-1} , and a strong band at 931 cm^{-1} show the presence of a unidentate sulphate group [13].

When dilute nitric and sulphuric acids were used in the reaction with Hofmann's base, quite different compounds were obtained. The isolated microcrystalline products were insoluble and of a variable mercury content depending upon the conditions. The X-ray powder diffraction patterns of the compounds isolated after 3 weeks from 20% nitric and sulphuric acids were not identical with those of compounds 1 and 2 respectively. They are probably polymeric, similar to trimercured acetaldehyde which forms three different polymeric



Scheme 1.

Table 4
Selected interatomic distances (Å) and bond angles (deg) with estimated standard deviations in parentheses for $C(HgNO_3)_4 \cdot H_2O$ (1)

Hg1–C	2.09(3)	Hg1–O11	2.10(3)
Hg2–C	2.06(2)	Hg2–O21	2.08(2)
Hg3–C	1.99(2)	Hg3–O31	2.12(2)
Hg4–C	2.09(2)	Hg4–O41	2.14(2)
O11–Hg1–C	176.8(9)	Hg2–C–Hg3	113.7(12)
O21–Hg2–C	174.0(9)	Hg2–C–Hg4	109.4(11)
O31–Hg3–C	176.2(10)	Hg3–C–Hg4	109.4(12)
O41–Hg4–C	171.7(9)	Hg1–O11–N1	109.8(20)
Hg1–C–Hg2	108.8(13)	Hg2–O21–N2	112.0(17)
Hg1–C–Hg3	109.2(12)	Hg3–O31–N3	111.9(19)
Hg1–C–Hg4	106.1(13)	Hg4–O41–N4	111.0(18)
Hg1...O13	2.78(4)	Hg3...O42 ^d	2.91(2)
Hg1...O31 ^a	2.75(2)	Hg3...O ^f	2.96(3)
Hg1...O42 ^b	2.84(3)	Hg4...O22 ^c	3.02(2)
Hg2...O23	2.88(3)	Hg4...O41 ^b	2.89(3)
Hg2...O23 ^c	3.05(3)	Hg4...O43	2.87(3)
Hg2...O12 ^a	2.94(3)	Hg4...O ^e	2.93(3)
Hg2...O43 ^d	2.77(2)	O...O33 ^h	2.87(4)
Hg3...O33	2.88(3)	O...O43 ⁱ	2.86(3)
Hg3...O21 ^e	3.05(2)		

Symmetry codes: ^a $x+0.5, 0.5-y, z+0.5$; ^b $1-x, 1-y, 2-z$; ^c $1-x, -y, 2-z$; ^d $0.5-x, y-0.5, 1.5-z$; ^e $0.5-x, 0.5+y, 1.5-z$; ^f $x-0.5, 0.5-y, z-0.5$; ^g $x-0.5, 0.5-y, z+0.5$; ^h $1-x, 1-y, 1-z$; ⁱ $x+0.5, 0.5-y, z-0.5$.

structures depending on the concentration of the dilute nitric acid [14,15].

3.1. Crystal structures of 1 and 2

Distances and angles in the structures of 1 and 2 are given in Tables 4 and 5 respectively, and their molecular structures with the atom labelling scheme are shown on Figs. 1 and 2 respectively.

Table 5
Selected interatomic distances (Å) and bond angles (deg) with estimated standard deviations in parentheses for $C(HgSO_4)_2(HgOH_2)_2$ (2)

Hg1–C	2.065(12)	Hg2–C	2.064(11)
Hg1–O1	2.104(9)	Hg2–O5	2.131(11)
Hg1–C–Hg2	110.05(3)	C–Hg1–O1	171.3(3)
Hg1–C–Hg1 ^a	102.47(4)	C–Hg2–O5	173.6(3)
Hg1–C–Hg2 ^a	110.52(2)		
Hg2–C–Hg2 ^a	112.77(3)		
Hg1...O2 ^b	2.760(11)	O5...O1 ^b	2.934(11)
Hg1...O4 ^c	2.991(9)	O5...O2 ^b	2.848(14)
Hg2...O1 ^b	2.991(10)	O5...O3 ^e	2.607(13)
Hg2...O2 ^d	2.747(8)	O5...O4 ^h	2.642(17)
Hg2...O3 ^c	2.893(12)		
Hg2...O4 ^f	3.001(12)		

Symmetry codes: ^a $-x, y, 0.5-z$; ^b $0.5-x, y-0.5, 0.5-z$; ^c $0.5-x, 0.5-y, 1-z$; ^d $x-0.5, 0.5-y, z-0.5$; ^e $x, y-1, z$; ^f $-x, y-1, 0.5-z$; ^g $-x, -y, -z$; ^h $x, -y, z-0.5$.

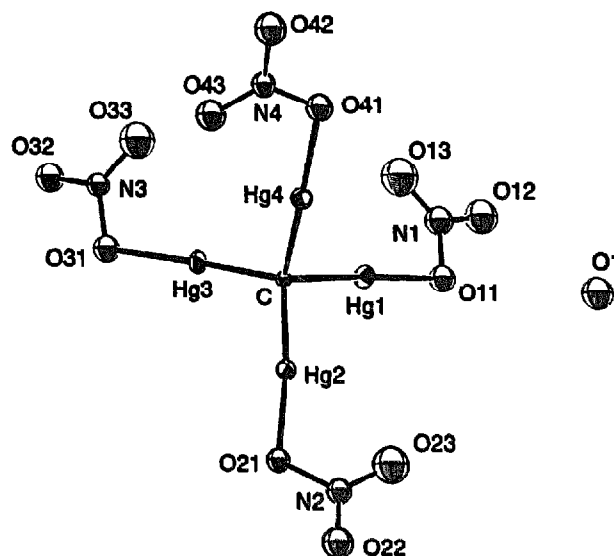


Fig. 1. ORTEP drawing of $C(HgNO_3)_4 \cdot H_2O$ (1) with the atom numbering scheme. The thermal ellipsoids are at the 50% probability level.

Both structures contain the tetramercurated methane unit with four mercury atoms tetrahedrally surrounding the carbon atom. The CHg_4 tetrahedron is in the general position in 1 and lies on the two-fold axis in 2. Both tetrahedra are slightly deformed, as can be seen from the values of the Hg–C–Hg angles which are within the range 106(1) to 114(1)° in 1 and 102.47(4) to 112.77(3)° in 2. The Hg–C distances range from 1.99(2) to 2.09(2) Å (mean value 2.06(2) Å) in 1, and are 2.064(11) and 2.065(12) Å in 2. These values are similar to the structures of other tetramercurated methane derivatives [3–6].

The crystal structure of 1 consists of $C(HgONO_2)_4$ and water molecules, as shown in Fig. 3. The two short

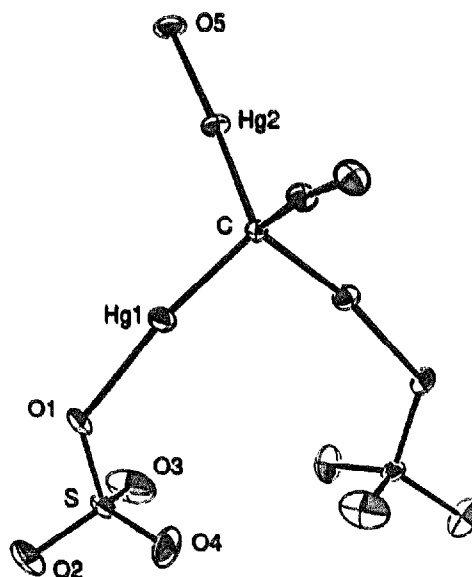


Fig. 2. ORTEP drawing of $C(HgSO_4)_2(HgOH_2)_2$ (2) with the atom numbering scheme. The thermal ellipsoids are at the 50% probability level.

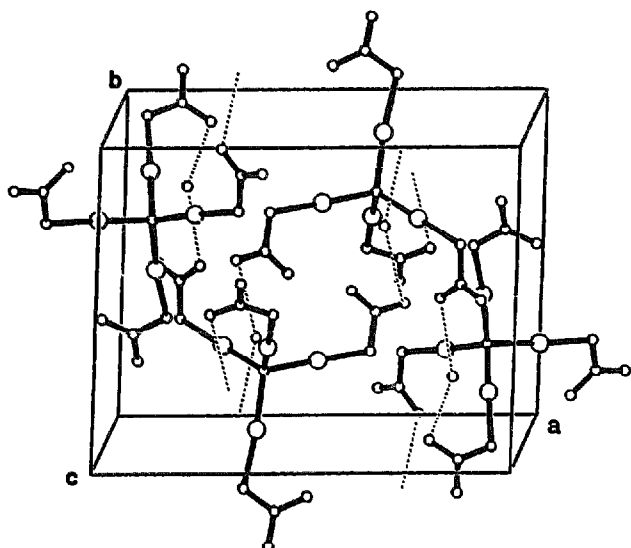


Fig. 3. Packing diagram of $C(HgNO_3)_4 \cdot H_2O$ (1) with possible hydrogen bonds drawn by dashed lines.

contacts of 2.86(3) and 2.87(4) Å between the water oxygen atoms and oxygen atoms of the nitrate ligands indicate hydrogen bonds. The Hg–O bond lengths are in the range 2.08(2) to 2.14(2) Å, mean value 2.11(1) Å, which is similar to that in other mercury compounds with covalently bonded nitrate, e.g. NO_3HgCH_2COOH [16], $[Hg(H_2OHg)(NO_3Hg)CCOO]NO_3$ and $2(NO_3Hg)_3CCOOH \cdot HNO_3$ [17] where these values are 2.10(2) Å, 2.12(2) Å and 2.12(2) Å (mean value) respectively. There is a lengthening of the N–O bond at the oxygen bonded to the mercury atom (mean value 1.33(1) Å) in comparison with the other N–O bonds (mean value 1.22(1) Å). All four mercury atoms have three (Hg1) or four (Hg2–Hg4) neighbouring oxygen atoms at a distance shorter than the sum of the van der Waals radii (ca. 3.1 Å [18,19]). The C–Hg–O angles

deviate slightly from linearity and range from 171.7(9) to 176.8(9)°.

The crystal structure of compound 2 consists of $C(HgOH_2)_2(HgOSO_3)_2$ molecules connected together by hydrogen bonds. The packing diagram of 2 with possible hydrogen bonds is shown on Fig. 4. Four short O···O contacts 2.61(1), 2.64(2), 2.85(1) and 2.93(1) Å indicate the possibility of formation of four hydrogen bonds. To enable formation of these bonds the two hydrogen atoms have to be disordered. Since the hydrogen atoms were not found it was supposed that they are located on the ligand water oxygen atom, thus forming a monomercured oxonium cation. Such oxonium cations are often found in mercured compounds containing oxygen, and can be mono-, di- and trimercured. Hydrogen atoms were found in the neutron diffraction studies of e.g. $Hg_3(OH)_2(SO_4)_2 \cdot H_2O$ [20] and $Hg(OH)F$ [21] which contain dimercurated oxonium cations, or in $HgSO_4(H_2O)$ [22] which contains a disordered monomercured oxonium cation and a sulphate ligand similar to that in 1. Some aliphatic organomercury compounds also contain mercured oxonium cations, e.g. in the structures of $[OHg_3CCHO]NO_3 \cdot H_2O$ [14] and $2[OHg_3CCHO]NO_3 \cdot HNO_3$ [15] the oxonium cation is trimercured, in $[HOHg_2(NO_3Hg)CCHO]NO_3$ [15] it is dimercurated, and in $[Hg(H_2OHg)(NO_3Hg)CCOO]NO_3$ [17] a monomercured oxonium cation is formed.

The Hg–O5 bond length of 2.131(11) Å is similar to that in the structure of $[Hg(H_2OHg)(NO_3Hg)CCOO]NO_3$ (Hg–OH₂ 2.17(3) Å) but significantly shorter than in $HgSO_4(H_2O)$ where the distance is 2.228(2) Å. The sulphate ligand is bonded to mercury at the Hg–O1 distance of 2.104(9) Å, which is similar to that in $Hg_3(OH)_2(SO_4)_2 \cdot H_2O$ (Hg–OSO₃ 2.087(2) Å), but significantly shorter than in $HgSO_4(H_2O)$ (Hg–OSO₃ 2.179(2) Å).

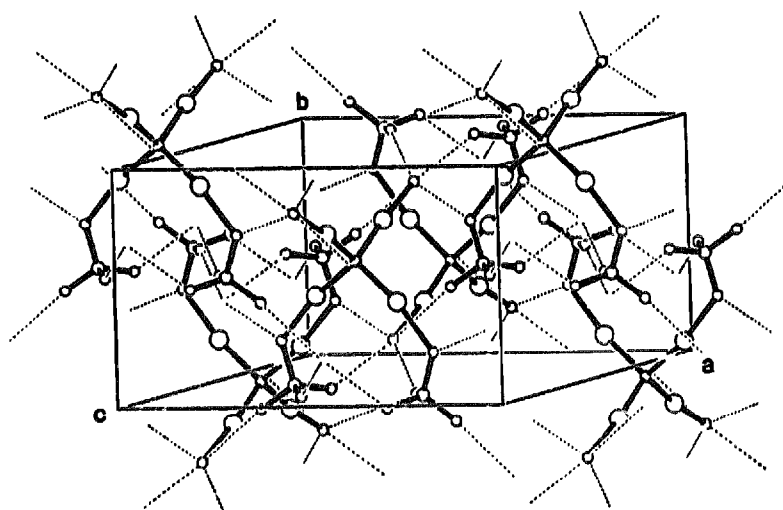


Fig. 4. Packing diagram of $C(HgSO_4)_2(HgOH_2)_2$ (2) with possible hydrogen bonds drawn by dashed lines.

A Cambridge Structural Database search [23] of the aliphatic organomercury compounds with the C–Hg–O structural unit gave 27 structures containing 59 such fragments. The mean value of the Hg–C bond is 2.082(5) Å. There are two distinct groups of data for the Hg–O interatomic distance. Of the total of 59 fragments there are 33 with the angle C–Hg–O greater than 150° (164.4–178.8°). They have bond distances in the range 2.023 to 2.181 Å (mean value 2.095(7) Å). When the angle C–Hg–O is less than 150° there always exists an Hg–X bond with C–Hg–X greater than 150°, and in such cases the Hg···O interatomic distances (all are greater than 2.45 Å) are considerably larger than the sum of covalent radii and represent contact distances. The analysis of the Hg–O bond distances showed that additional short contacts Hg···X (e.g. X = O, N) cause lengthening of the Hg–O bond. No influence of short contacts was found on the Hg–C bond length. Therefore, the long bond lengths in HgSO₄(H₂O) are caused by four short additional Hg···OSO₃ contacts in the range 2.501(1) to 2.514(1) Å. In **2** these contact distances are longer; Hg1 is approached by two O atoms at distances of 2.760(11) and 2.991(9) Å, which are shorter than the sum of the van der Waals radii. Hg2 has four contacts in the range 2.747(8) to 3.001(2) Å. All of these oxygen atoms involved in additional contacts are from the neighbouring sulphate ligands. These contacts cause bending of the C–Hg–O angles (171.3(3) and 173.6(3)°) from linearity. The S–O bond lengths (in the range 1.45(1) to 1.52(1) Å) are similar to those in the mentioned sulphates. The S–O1 bond is the longest since it involves the oxygen bonded to mercury.

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