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Structure of methyl(*o*-nitrobenzenethiolato)mercury

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Abstract—The compound, $MeHgSC_6H_4NO_2$-*o*, crystallizes in the monoclinic space group, $P2_1/n$ with $Z = 8$. In each of the two independent molecules of $MeHgSC_6H_4NO_2$-*o*, there is a S---O intramolecular interaction [2.73(2) (molecule A) and 2.81(2) Å (molecule B)] with the three nearest intermolecular neighbours to each Hg atom being 2 S and 1 O atoms: in molecule A, Hg---O = 3.48(2), Hg---S = 3.322(4) and 3.539(4) Å; in molecule B, Hg---O = 3.61(3), Hg---S = 3.257(4) and 3.647(5) Å. The geometry at Hg becomes trigonal bipyramidal if all three of these secondary contacts are included with the primary linear bonds [Hg—C = 2.08(2) and Hg—S = 2.379(4) Å, C—Hg—S = 176.4(5)° in molecule A; Hg—C = 2.04(2) and Hg—S = 2.366(4) Å, C—Hg—S = 177.0(5)° in molecule B]. The intramolecular $(H_3)C$—Hg—S---O(NO) fragment is near linear in each of the two independent molecules. © 1997 Elsevier Science Ltd

Keywords: organomercury; thiolates; non-bonded interactions.

Intramolecular non-bonded sulfur–oxygen contacts less than the sum of the van der Waals radii (3.25 Å) have been variously determined by diffraction methods in organic compounds [1] (the individual van der Waals radii for S and O are taken to be 1.85 and 1.40 Å, respectively [2]). Examples of compounds exhibiting these interactions are the family of *o*-nitroarenethiolato species, **1** (X = alkyl [3], aryl [4], alkoxy [5], halo [5a,6], pseudohalo [7], sulfido [8], etc).

Other structural features of these compounds are near linear X—S···O angles and near coplanarity of the *ortho* NO_2 group and the aryl ring. In addition, the S---O separation is dependent on the nature of the X group (Table 1).

The primary bonds to Hg in mercury compounds, Z—Hg—Y, generally provide a near linear geometry at mercury, with a Z—Hg—Y valency angle in the range from 170 to 180°. In addition to the primary bonds, secondary and weaker interactions (of an intra- and/or intermolecular nature) are frequently found between the Hg atom and suitable donor atoms (S, N, O, etc.) resident in the molecule [9]. Including these secondary interactions, various geometries at mercury can be realised, e.g., "T"-shaped, as in 2-Cl-4-BrC_6H_3OHgPh [10], planar four-coordinate, as in 4-Me-2-$HOC_6H_4HgOC_6H_3$Me-4-NO-6 [11] and octahedral in PhHgOAc [12].

With both the S---O interactions and the secondary bonding at Hg in mind, we have studied the X-ray structure of $MeHgSC_6H_4NO_2$-*o* and we now report our findings.

EXPERIMENTAL

NMR spectra were obtained on a Bruker 250 MHz instrument; IR spectra were recorded on a Philips Analytical PU9800 and Nicolet 205 Fourier-transform spectrometers. The X-ray data were collected by the EPSRC data collection service based at the University of Wales, Cardiff.

o-Nitrobenzenethiol was prepared by a published procedure from *o*-chloronitrobenzene and sodium sulfide [13].

Preparation of $MeHgSC_6H_4NO_2$-o

To a hot solution of MeHgCl (2.51 g, 0.01 mol) in CCl_4 (100 cm^3) were successively added a solution of

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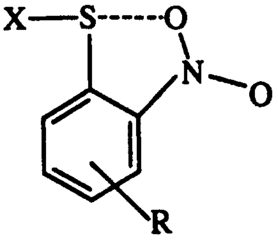
Table 1. Values of S---O (Å) and X—S---O (°) in 1 (R = H) as determined by X-ray crystallography



(1)

X	S---O (Å)	X—S---O (°)	Electronegativity of the binding atom of X[a]	Reference
Cl	2.379(5)	176.9(3)	3.54	[5b]
	2.408(5)	178.5(3)		
MeO	2.458(2)	176.4(2)	3.22	[5b]
NCS	2.504(7)	176.8(2)	2.65	[7]
o-$O_2NC_6H_4SS$	2.592(3)	175.04(8)	2.65	[8a]
	2.598(3)	178.70(8)		
$Ph_3SnCH(NCS)CH_2$	2.655(5)	172.7(3)	2.67	[3b]
$Ph_3Sn(CH_2)_2CH(CH_2Cl)$	2.610(5)	177.5(3)	2.67	[3a]
o-$O_2NC_6H_4$	2.656(1)	171.7(1)	2.67	[4b]
	[2.900(2)	134.3(1)]		
p-$MeOC_6H_4$	2.641(2)	177.69(8)	2.67	[4a]
MeHg	2.73(2)	172.4(5)		This study
	2.81(2)	172.7(5)		

[a] Values from [22]. L. C. Allen, *J. Am. Chem. Soc.* 1989, **111**, 9003.

o-$O_2NC_6H_4SH$ (1.55 g, 0.01 mol) in CCl_4 (50 cm^3) and NEt_3 (1.4 cm^3). The mixture was cooled to room temperature, filtered and the filtrate rotary evaporated. The residue from the filtrate was recrystallized several times from EtOH to give yellow crystals of $MeHgSC_6H_4NO_2$-*o*, m.p. 104–105°C. Found: C, 23.0; H, 2.0; N, 3.6. $C_7H_7HgNO_2S$ requires: C, 22.7; H, 1.9; N, 3.8%. ^1H NMR ($CDCl_3$, 250 MHz), δ: 0.99 [s, 3H, $J(^1H—^{199}Hg)$ = 168 Hz, MeHg], 7.27 [m, 1H], 7.36 [m, 1H] 7.66 [m, 2H] (aryl-H). ^{13}C NMR ($CDCl_3$, 62.9 MHz), δ: 11.6, 124.0, 126.3, 127.1, 131.0, 137.3. ν_{max} (KBr) (cm^{-1}): 1586, 1514, 1446, 1337, 1247, 1104, 1055, 1039, 854, 775, 727

X-ray analysis of $MeHgSC_6H_4NO_2$-o

A yellow crystal measuring 0.07 × 0.11 × 0.11 mm was used for X-ray reflection data collection. Data were measured on a Delft Instruments FAST diffractometer with monochromatized Mo-K_α radiation. Corrections were made for Lorentz and polarization effects. Crystal data and structure refinement details are given in Table 2. The mercury atom positions were located a by direct methods procedure using [14] SIR88 and the structure was completed by Fourier refinement using the program [15] CRYSTALS. Hydrogen-atom positions were calculated from geometric considerations using a C—H bond length of 1.00 Å and an isotropic thermal vibration parameter (U_{iso}) of 0.05 $Å^2$. Hydrogen-atom positions and thermal parameters were not refined. Full-matrix least squares calculations with anisotropic thermal vibration parameters for all non-hydrogen atoms were performed using the program CRYSTALS. During refinement the phenyl group was refined as a rigid group. Molecular diagrams were obtained using the program [16] CAMERON.

RESULTS AND DISCUSSION

The compound $MeHgSC_6H_4NO_2$-*o* was obtained from MeHgCl and *o*-$O_2NC_6H_4SH$ in CCl_4 solution in the presence of the base, NEt_3. Formed along with the desired product were significant amounts of the disulfide, *o*-$O_2NC_6H_4SSC_6H_4NO_2$-*o*. Complete sep-

Table 2. Data collection and refinement parameters for $MeHgSC_6H_4NO_2$-*o*

Molecular formula	$C_7H_7HgNO_2S$
Formula weight (a.m.u.)	369.79
Temperature (K)	293(2)
Wavelength (Å)	0.71069
Crystal system	Monoclinic
Space group	$P\,2_1/n$
a (Å)	20.717(6)
b (Å)	4.041(1)
c (Å)	21.383
β (°)	91.45(2)
V (Å^3)	1790
Z	8
D_x (Mg m^{-3})	1.82
μ (mm^{-1})	17.38
Max $\lvert h\rvert(\lvert k\rvert, \lvert l\rvert)$	22(4,24)
Reflections measured	7037
No. of unique reflections	2729
R_{int}	0.14
No. of classed observed	1306
Observation criterion [$F > n\sigma(F)$]	3
Refinement method	Full-matrix least-squares on F
No. parameters refined	193
Weighting scheme	Chebychev polynomial [1]
Weighting scheme coefficients[a]	26.1, −21.0, 22.8
R	0.0578 (5.78%)
R_w	0.0419 (4.19%)
Min (max) $\Delta\rho$ (e Å^{-3})	−3.68(2.91)

[a] $W = w[1\text{-}(\Delta F/6\sigma(F))^2]^2$, where w is calculated using a three term Chebychev series with coefficients given.

aration of the two products took several recrystallizations to achieve. The compound $Hg(SC_6H_4NO_2\text{-}o)_2$ was obtained with similar difficulty from $HgCl_2$ and $o\text{-}O_2NC_6H_4SH$ in EtOH. Unfortunately, $Hg(SC_6H_4NO_2\text{-}o)_2$ did not provide suitable crystals for X-ray crystallography.

Crystal structure of $MeHgSC_6H_4NO_2$-*o*

The atom numbering scheme and atom arrangements in the two independent molecules are shown in Fig. 1. Selected bond lengths and angles are listed in Table 3.

The primary bonds to mercury in each of the two independent molecules of $MeHgSC_6H_4NO_2$-*o* are arranged in a linear geometry about mercury, the S—Hg—C valency angles being 176.4(5)° [C(1)—Hg(1)—S(1)] in molecule A and 177.0(5)° [C(2)—Hg(2)—S(2)] in molecule B. The primary bond lengths are Hg(1)—S(1) = 2.379(4) and Hg(1)—C(1) = 2.08(2) Å in molecule A and Hg(2)—S(2) = 2.366(4) and Hg(2)—C(2) = 2.04(2) Å in molecule B. The lengths are in the normal ranges found for such bonds [9], e.g. the values found in $MeHgSC_6H_2\text{-}2,4,6\text{-}Pr^i_3$ are 2.344(4) and 2.07(3) Å [17], in MeHgS-pyridyl-*o* 2.374(2) and 2.089(7) Å [18] and in $PhHgSC_6H_4NMe_2$-*o* 2.365(2) and 2.076(6) Å [19].

Secondary intermolecular Hg---S contacts have been reported in various organomercury thiolates, e.g. values determined in [17] $MeHgSC_6H_2\text{-}2,4,6\text{-}Pr^i_3$, $PhHgSC_6H_3Me_2\text{-}2,6$ [20] and $PhHgSC_6H_4NMe_2$-*o* [19] were 3.269(3), 3.18 and 3.224(2) Å, respectively. In $PhHgSC_6H_4NMe_2$-*o*, there is an additional contact [19] to the intramolecular nitrogen [Hg—N = 2.657(6) Å]. In $MeHgSC_5H_4N$-*o*, there are two intermolecular Hg---S contacts [3.322(2) and 3.520(2) Å] and an intramolecular Hg—N interaction [Hg---N = 2.980(5) Å.

The three nearest donor atoms to each of the mercury atoms in the two independent molecules of $MeHgSC_6H_4NO_2$-*o* are one oxygen and two sulfur atoms. The relevant distances to Hg(1) in molecule A are: Hg(1)---S^{i}(1) = 3.322(4), Hg(1)---S^{iii}(1) = 3.539(4) and Hg(1)---O^{ii}(22) = 3.48(2) Å; the distances to Hg(2) in molecule B are Hg(2)---S^{iv}(2) = 3.257(4), Hg(2)---S^{vi}(2) = 3.647(5) and Hg(2)---O^{v}(12) = 3.61 Å) [symmetry operations: (i) $1-x$, $1-y$, $2-z$: (ii) $1.5-x$, $-0.5+y$, $1.5-z$; (iii) x, $-1+y$, z; (iv) $2-x$, $2-y$, $2-z$; (v) $1-x$, $2-y$, $2-z$; (vi) x, $1+y$, z]. With the value of the van der Waals radius [21] of Hg taken to be 1.73 Å, the sum of the van der Waals radii for Hg and O and Hg and S are 3.13 and 3.58 Å, respectively. Thus, in molecule A, the Hg(1)---S^{i}(1) interaction is well-within, Hg(1)---S^{iii}(1) is just within and Hg(1)---

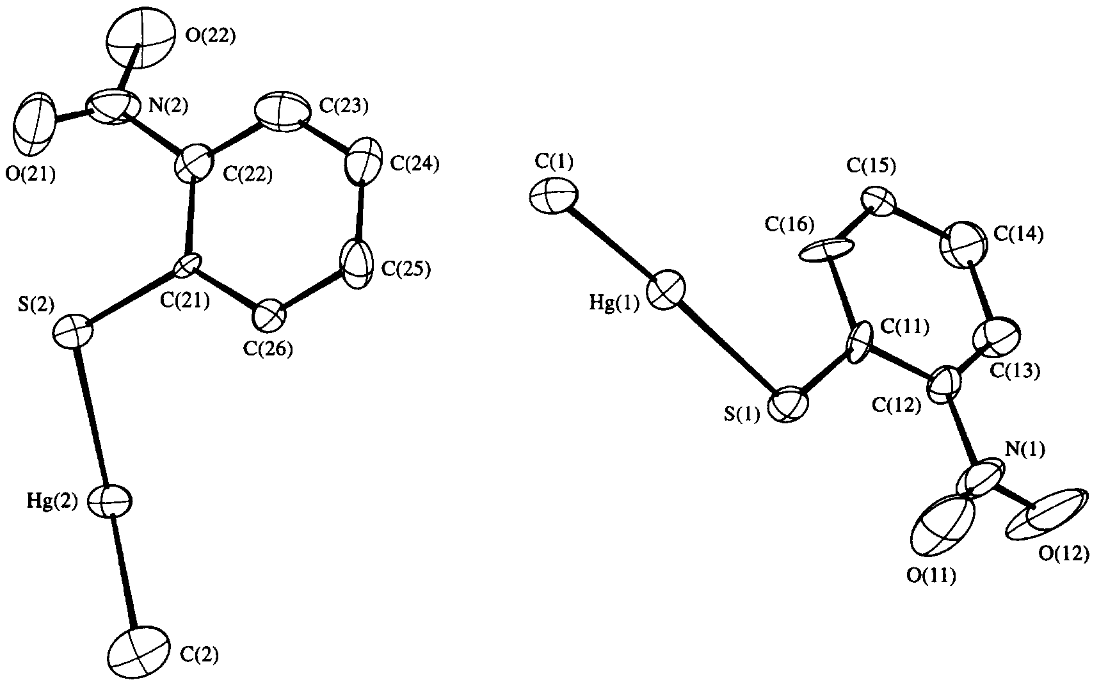
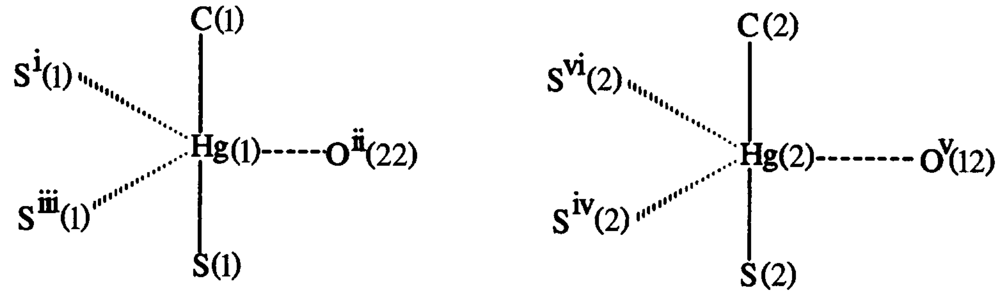


Fig. 1. Atom numbering scheme and atom arrangement for $MeHgSC_6H_4NO_2$-*o*.



Molecule A

C(1)-Hg(1)---S^{i}(1)	103.6(5)
C(1)-Hg(1)---S^{iii}(1)	97.2(5)
C(1)-Hg(1)---O^{ii}(22)	67.8(6)
average	89.5
S^{i}(1)---Hg(1)---S^{iii}(1)	108.21(9)
S^{i}(1)---Hg(1)---O^{ii}(22)	128.6(4)
O^{i}(22)---Hg(1)---S^{iii}(1)	123.0(4)
Σ	359.8
S(1)-Hg(1)---S^{i}(1)$^{)}$	72.8(1)
S(1)-Hg(1)---O^{ii}(22)	114.7(4)
S(1)-Hg(1)---S^{iii}(1)	83.7(1)
average	90.4

Molecule B

C(2)-Hg(2)---S^{iv}(2)	103.2(6)
C(2)-Hg(2)---S^{vi}(2)	101.1(6)
C(2)-Hg(2)---O^{v}(12)	69.1(8)
average	91.1
S^{iv}(2)---Hg(2)---S^{vi}(2)	106.73(9)
S^{iv}(2)---Hg(2)---O^{v}(12)	128.5(4)
O^{v}(12)---Hg---S^{vi}(2)	124.7(4)
Σ	359.9
S(1)-Hg(1)---S^{iii}(2)	74.4(1)
S(2)-Hg(2)---O^{v}(12)	110.8(4)
S(2)-Hg(2)---S^{vi}(2)	81.4(1)
average	88.9

Fig. 2. The coordination sphere about mercury in $MeHgSC_6H_4NO_2$-*o*.

Table 3. Bond lengths (Å) and angles (°) for $MeHgSC_6H_4NO_2$-*o*

Hg(1)—S(1)	2.379(4)	Hg(2)—S(2)	2.366(4)
Hg(1)—C(1)	2.08(2)	Hg(2)—C(2)	2.04(2)
S(1)—C(11)	1.810(7)	S(2)—C(21)	1.791(7)
O(11)—N(1)	1.19(2)	O(21)—N(2)	1.15(2)
O(12)—N(1)	1.17(2)	O(22)—N(2)	1.26(2)
N(1)—C(12)	1.43(2)	N(2)—C(22)	1.44(2)
S(1)---O(11)	2.73(2)	S(2)---O(21)	2.81(2)

S(1)—Hg(1)—C(1)	176.4(5)	S(2)—Hg(2)—C(2)	177.0(5)
Hg(1)—S(1)—C(11)	106.5(3)	Hg(2)—S(2)—C(21)	106.9(3)
O(11)—N(1)—O(12)	117.8(17)	O(21)—N(2)—O(22)	127.3(19)
O(11)—N(1)—C(12)	122.2(14)	O(21)—N(2)—C(22)	119.8(16)
O(12)—N(1)—C(12)	119.9(17)	O(22)—N(2)—C(22)	112.8(18)
S(1)—C(11)—C(12)	121.1(5)	S(2)—C(21)—C(22)	120.3(5)
S(1)—C(11)—C(16)	118.8(5)	S(2)—C(21)—C(26)	119.6(5)
N(1)—C(12)—C(11)	123.4(9)	N(2)—C(22)—C(21)	123.1(9)
N(1)—C(12)—C(13)	116.5(9)	N(2)—C(22)—C(23)	116.9(9)
O(11)---S(1)—Hg(1)	172.4(5)	O(21)---S(2)—Hg(2)	172.7(5)

Intermolecular interatomic distances (Å) and angles (°)

Hg(1)—S^{i}(1)	3.322(4)	Hg(2)—S^{iv}(2)	3.257(4)
Hg(1)—O^{ii}(22)	3.48(2)	Hg(2)—O^{v}(12)	3.61(3)
Hg(1)—S^{iii}(1)	3.539(4)	Hg(2)—S^{vi}(2)	3.647(5)

C(1)—Hg(1)—S^{i}(1)	103.6(5)	C(2)—Hg(2)—S^{iv}(2)	103.2(6)
C(1)—Hg(1)—O^{ii}(22)	67.8(6)	C(2)—Hg(2)—O^{v}(12)	69.1(8)
C(1)—Hg(1)—S^{iii}(1)	97.2(5)	C(2)—Hg(2)—S^{vi}(2)	101.1(6)
S(1)—Hg(1)—S^{i}(1)	72.8(1)	S(2)—Hg(2)—S^{iv}(2)	74.4(1)
S(1)—Hg(1)—O^{ii}(22)	114.7(4)	S(2)—Hg(2)—O^{v}(12)	110.8(4)
S(1)—Hg(1)—S^{iii}(1)	83.7(1)	S(2)—Hg(2)—S^{vi}(2)	81.4(1)
S^{I}(1)—Hg(1)—O^{o}(22)	128.6(4)	S^{IV}(2)—Hg(2)—O^{v}(12)	128.5(4)
S^{I}(1)—Hg(1)—S^{iii}(1)	108.21(9)	S^{IV}(2)—Hg(2)—S^{vi}(2)	106.73(9)
O^{II}(22)—Hg(1)—S^{iii}(1)	123.0(4)	O^{V}(12)—Hg(2)—S^{vi}(2)	124.7(4)

(i) $1 - x, 1 - y, 2 - z$; (ii) $1.5 - x, -0.5 + y, 1.5 - z$; (iii) $x, -1 + y, z$; (iv) $2 - x, 2 - y, 2 - z$; (v) $1 - x, 2 - y, 2 - z$; (vi) $x, 1 + y, z$.

O^{ii}(22) outside the appropriate sums. It would thus appear that Hg(1) makes just two secondary intermolecular contacts. However, the three atoms S^{i}(1), S^{iii}(1) and O^{ii}(22) make angles with Hg(1), which sum to 359.8°, very close to the value of 360° expected for equatorial ligands in a trigonal bipyramid structure and which in addition provide average equatorial-Hg-axial angles of 90° with the C(1) and S(1) atoms (see Fig. 2). If all three atoms are considered to have a structural influence on Hg(1), a trigonal bipyramidal geometry at Hg(1) is realised.

For Hg(2), only the Hg(2)---S^{iv}(2) separation is within the appropriate sum of the van der Waals radii; both Hg(2)---S^{vi}(2) and Hg(2)---O^{v}(12) are outside their's. However, similar to the situation for Hg(1), S^{iv}(2), S^{vi}(2) and O^{v}(12) can all be considered to be in the equatorial plane to the C(2)—Hg(2)—S(2) axis and so again a trigonal bipyramidal structure can be considered also for Hg(2) (see Fig. 2).

The intramolecular S---O(NO) distances and Hg—S---O angles in the two independent molecules, S(1)---O(11) = 2.73(2) Å and Hg(1)—S(1)---O(11) = 172.4(5)° in molecule A and S(2)---O(21) = 2.81(2) Å and Hg(2)—S(2)---O(21) = 172.7(5)° in molecule B, clearly indicate S---O interactions. These separations are a little greater than the separations for alkyl and aryl *o*-nitroaryl sulfides (**1**, X = R), which generally fall in the region 2.6–2.7 Å, but they are still well within the sum of the van der Waals radii for S and O (3.25 Å). One of the more noticeable differences between the two independent molecules of $MeHgSC_6H_4NO_2$-*o* is the angles between the planes of the nitro and aryl groups, 4.98 and 32.40°, in molecule A and B, respectively. In keeping with these values, the shorter S---O separation (and hence stronger interaction) is found in molecule A.

The combination of the near linear C—Hg—S and Hg—S—O moieties results in a near linear 4 atom chain, C—Hg—S—O. The intermolecular associations provide an interesting three-dimensional-packing arrangement, as illustrated in Fig. 3.

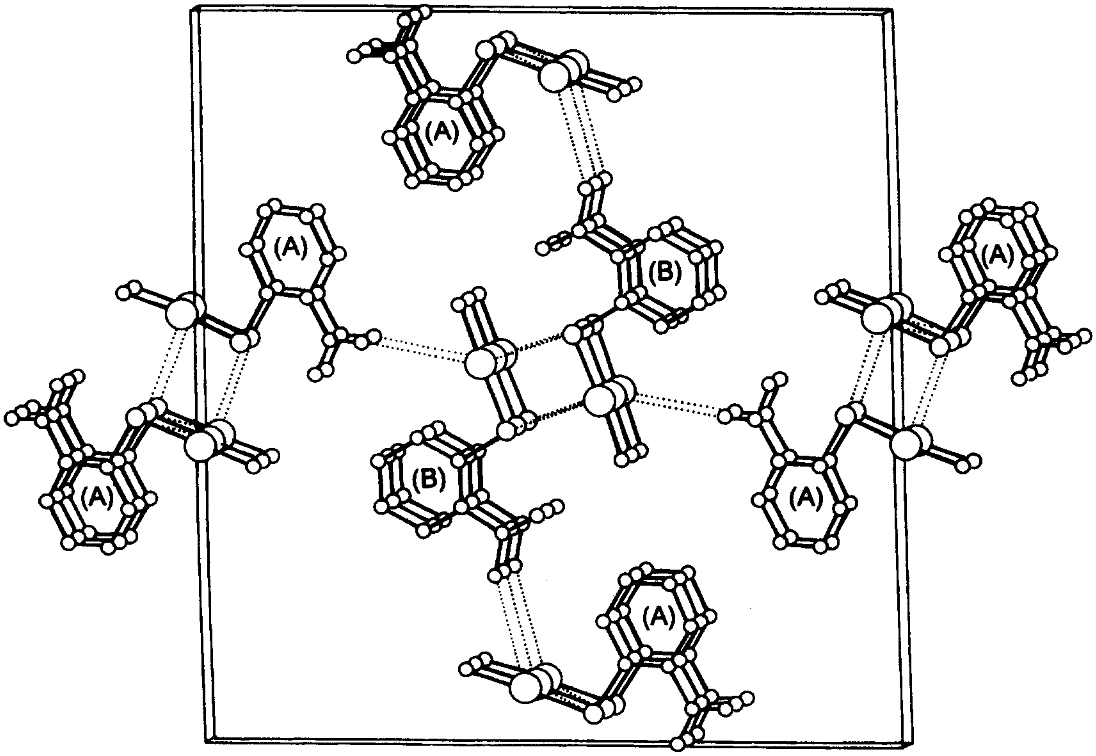


Fig. 3. The packing arrangement in $MeHgSC_6H_4NO_2$-*o*.

CONCLUSIONS

Both the S···O intramolecular interaction and the secondary bonding to Hg, as highlighted in the Introduction, were shown to occur in $MeHgSC_6H_4NO_2$-*o*. The intermolecular bonding results in a three-dimensional network structure.

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