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AN X-RAY STRUCTURAL STUDY OF NONVALENCE INTERACTIONS AND COORDINATION IN ORGANOMETALLIC COMPOUNDS.

XX.* THE CRYSTAL STRUCTURE OF PHENYLMERCURY 2-DIMETHYLAMINOTHIOPHENOLATE

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UDC 548.737

The structure of phenylmercury 2-dimethylaminothiophenolate has been determined by x-ray diffraction (R = 0.0277 from 3227 reflections). The mercury atom has the typical linear configuration of the valence bonds, which have their usual lengths, Hg–C 2.076(6) and Hg–S 2.365(2) Å. At the same time, for the observed conformation of the molecule, the Hg atom approaches the N atom of the dimethylamino-group to a distance of 2.657(6) Å, corresponding to a secondary bond. The nitrogen atom has a pyramidal bond configuration, and its unshared electron pair is oriented towards the mercury atom. The presence of the secondary bond Hg...N does not lead to significant bending of the S–Hg–N unit (the angle at the Hg atom is 178.0(2)°).

INTRODUCTION

The ^{19}F NMR spectra of the PhHg-, Ph_3Sn-, and Ph_3Pb-derivatives of 2,6-dibromo-4-fluorothiophenol [2] and the ^{35}Cl NQR spectra of the corresponding derivatives of 2,6-dichlorothiophenol [3] indicate the existence of intramolecular coordination interactions in the PhHg-derivatives of o-halogeno-substituted thiophenols, both in solution and in the crystal.

On the other hand, according to the results of a study of exchange equilibria involving the PhHg-, Ph_2Sb-, Ph_3Sn-, and Ph_3Pb-derivatives of substituted thiophenols [4, 5], the five- and six-membered chelate rings in o-substituted phenylmercury thiophenolates in all cases are more stable than the chelate rings in the other organometallic derivatives of o-substituted thiophenols. At the same time, the study of organometallic derivatives of o-substituted thiophenols by x-ray structural analysis has shown, in particular, that there is no intra-

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* For Part XIX, see [1].

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TABLE 1. Coordination of the Atoms (× 10^4, or × 10^5 for the Hg atom) and the Parameters B_{ij} of the Anisotropic Temperature Factor $T = \exp[-{}^1/_4 (B_{11}a^{*2}h^2 + B_{22}b^{*2}k^2 + \ldots + 2B_{23}b^* c^* kl)]$

Atom	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Hg	2227(3)	8551(2)	11775(2)	1,56(1)	1,43(1)	1,15(1)	—0,13(1)	0,54(1)	0,17(1)
S	977(2)	1510(2)	—136(1)	2,03(7)	1,58(7)	0,90(6)	—0,60(6)	0,37(5)	0,06(5)
N	3706(8)	776(5)	1478(4)	2,0(3)	1,7(2)	1,5(2)	0,5(2)	0,7(2)	0,5(2)
C(1)	3123(9)	2035(6)	197(4)	1,7(3)	1,2(2)	1,3(2)	0,1(2)	0,9(2)	—0,2(2)
C(2)	4268(9)	1660(6)	949(4)	1,6(3)	1,7(3)	1,7(3)	0,3(2)	1,0(2)	—0,1(2)
C(3)	5891(10)	2218(7)	1172(5)	1,6(3)	3,1(4)	2,4(3)	0,5(3)	0,5(3)	0,3(3)
C(4)	6453(10)	3091(8)	658(6)	1,4(3)	2,8(4)	3,7(4)	—0,5(3)	0,7(3)	0,3(3)
C(5)	5362(10)	3399(7)	—101(6)	2,3(3)	2,1(3)	3,4(4)	—0,1(3)	1,6(3)	0,5(3)
C(6)	3708(10)	2893(6)	—332(5)	2,4(3)	1,4(3)	1,8(3)	0,1(2)	1,1(3)	0,2(2)
C(7)	4458(10)	842(8)	2416(5)	2,2(3)	3,8(4)	1,3(3)	0,7(3)	0,1(3)	0,7(3)
C(8)	3995(11)	—384(7)	1134(5)	3,0(4)	1,5(3)	3,0(4)	0,8(3)	1,0(3)	0,4(3)
C(1′)	—519(9)	313(6)	2321(4)	1,5(3)	1,4(3)	1,0(2)	0,0(2)	0,6(2)	0,3(2)
C(2′)	—1155(9)	—801(6)	2397(4)	2,0(3)	1,4(3)	1,4(2)	0,0(2)	0,6(2)	0,1(2)
C(3′)	—1628(9)	—1173(6)	3175(5)	1,8(3)	1,5(3)	1,8(3)	—0,1(2)	0,6(2)	0,5(2)
C(4′)	—1451(9)	—436(7)	3881(4)	1,7(3)	2,2(3)	1,5(3)	—0,1(2)	0,8(2)	0,3(2)
C(5′)	—856(10)	682(7)	3810(4)	2,3(3)	2,2(3)	1,3(2)	—0,4(3)	0,7(2)	—0,2(2)
C(6′)	—356(10)	1063(6)	3039(4)	2,4(3)	1,8(3)	1,2(2)	—0,9(2)	0,4(2)	0,0(2)

TABLE 2. Coordinates of the Hydrogen Atoms (× 10^3)

Atom	x	y	z	Atom	x	y	z
H(3)	677	199	177	H(28)	325	– 79	137
H(4)	771	352	84	H(38)	506	—59	111
H(5)	579	406	—53	H(2′)	—135	—140	184
H(6)	285	318	—93	H(3′)	—219	—205	323
H(17)	376	12	268	H(4′)	—183	—73	449
H(27)	437	166	267	H(5′)	—82	127	435
H(37)	569	63	255	H(6′)	179	193	301
H(18)	233	—42	63				

TABLE 3. Valence Angles ω, deg

Angle	ω	Angle	ω	Angle	ω
SHgC(1′)	178,0(2)	C(2)NC(7)	115,7(6)	C(6)C(1)C(2)	119,0(6)
HgSC(1)	102,6(2)	C(2)NC(8)	110,6(6)	C(1′)C(2′)C(3′)	120,6(6)
HgC(1′)C(2′)	120,6(5)	C(7)NC(8)	110,6(6)	C(2′)C(3′)C(4′)	120,5(7)
HgC(1′)C(6′)	120,6(5)	C(1)C(2)C(3)	118,0(6)	C(3′)C(4′)C(5′)	119,6(7)
SC(1)C(2)	124,2(5)	C(2)C(3)C(4)	122,8(7)	C(4′)C(5′)C(6′)	120,6(7)
SC(1)C(6)	116,8(5)	C(3)C(4)C(5)	118,4(8)	C(5′)C(6′)C(1′)	120,0(6)
NC(2)C(1)	119,5(6)	C(4)C(5)C(6)	120,7(8)	C(6′)C(1′)C(2′)	118,7(6)
NC(2)C(3)	122,4(6)	C(5)C(6)C(1)	120,9(7)		

molecular coordination in the crystals of the Ph_3Sn-derivative of 2,6-dibromo-4-fluorothiophenol [6] and triphenyl-lead o-bromothiophenolate [7]. It was therefore of interest to study the possible existence of chelate rings in the crystals of the PhHg-derivatives of o-substituted thiophenols. On crystallization, phenylmercury 2-dimethylaminothiophenolate gives well-formed single crystals, and this made it possible to use it as a model compound for the solution of this problem by an x-ray structural study.

EXPERIMENTAL

Preparation of Phenylmercury 2-Dimethylaminothiophenolate (I). 0.51 g (0.0035 mole) of 2-methylaminothiophenol [9] was added to a hot solution of 1.03 g (0.0035 mole) of phenylmercury hydroxide [8] in 50 ml of ethanol. The reaction mixture was cooled, and the resulting precipitate was filtered off and recrystallized from alcohol. This gave 1.2 g (80%) of colorless crystals of compound I with mp 93-95°. Found: C 39.00, H 3.65, Hg 46.14%; calculated for $C_4H_{15}HgNS$: C 39.07, H 3.49, Hg 46.7%.

TABLE 4. Equations of the Planes $Ax + By + Cz - D = 0$ of Individual Fragments of the Molecule, the Deviations of the Atoms from the Planes (Å), and the Angles between the Planes, deg

Plane	Atoms and their deviations from the plane					*A*	*B*	*C*	*D*
I	Hg S C(1)					0,3975	−0,9054	−0,1491	−1,2250
II	C(1) −0,023(7)	C(2) 0,025(7)	C(3) −0,006(8)	C(4) −0,016(9)	C(5) 0,018(8)	0,4984	−0,7012	−0,5098	−0,5958
	C(6) 0,002(7)	Hg* −1,1003(2)	S* − 0,119(2)	N* 0,031(6)					
III	C(2)	C(7)	C(8)	N* 0,415(6)	Hg* 3,0524(3)	−0,9920	0,1024	−0,0737	−2,9280
IV	C(1′) −0,001(7)	C(2′) 0,002(7)	C(3′) 0,004(7)	C(4′) −0,012(7)	C(5′) 0,013(8)	−0,8909	0,3250	−0,3173	−0,0777
	C(6′) −0,006(7)	Hg* −0,0378							

Angles between the planes I-II 24.6°, III-III 121.9°

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*Atoms not taken into account in the calculation of the equations of the corresponding planes.

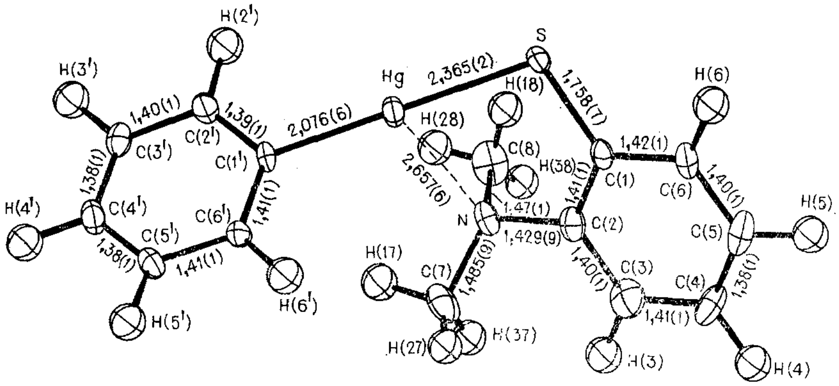


Fig. 1. Structure of the molecule of phenylmercury 2-dimethylaminophenolate.

The crystals of $C_{14}H_{15}HgNS$ are monoclinic; at $-120°$, $a = 7.749(2)$, $b = 11.504(3)$, $c = 15.697(3)$ Å, $\beta = 100.34(2)°$, $V = 1376.3(8)$ Å^3, $d_{calc} = 2.07$ g/cm^3, $Z = 4$, space group $P2_1/c$, $\mu(MoK\alpha) = 112.0$ cm^{-1}.

The unit cell parameters and the intensities of 3282 reflections with $I > 2\sigma$ were measured on a "Syntex $P2_1$" automatic four-circle diffractometer (recording at $-120°$C, $\lambda MoK\alpha$, graphite monochromator, $\theta/2\theta$ scanning, $0 < 2\theta \leq 60°$). The absorption for the actual form of the crystal was taken into account by the published method [10].

The structure was determined by the heavy-atom method and refined by the method of least squares in the full-matrix anisotropic approximation. The hydrogen atoms of the methyl groups were located in a difference synthesis; the positions of the hydrogen atoms of the benzene rings were calculated geometrically. The final refinement was carried out in the full-matrix anisotropic approximation for fixed values of the positional and temperature ($B_{iso} = 3.5$ Å^2) parameters of the hydrogen atoms from 3227 reflections with $F > 5\sigma$. The final R-factor is 0.0277 ($R_G = 0.0368$). The coordinates of the atoms other than hydrogen, and their anisotropic temperature parameters B_{ij}, are given in Table 1, and the coordinates of the hydrogen atoms are given in Table 2.

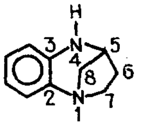
RESULTS AND DISCUSSION

The structure of the molecule and the bond lengths are shown in Fig. 1, and the valence angles are given in Table 3.

As in the great majority of organomercury compounds, the mercury atom has the typical linear valence bond configuration. The length of the bond Hg–C(1) with the phenyl ring, 2.076(6) Å, agrees with the analogous values in other organomercury compounds [11]. The length of the bond Hg–S with the thiophenol residue, 2.365(2) Å, is close to that found in phenylmercury 2,6-dimethylthiophenolate (2.33(1) Å) [12], in 1-methyl-4-thiouracilyl-p-mercurobenzoic acid (2.342(3) Å) [13], and in benzyl(triphenylmethylthio)mercury (2.363 Å) [14]. The S–C(1) distance, 1.758(7) Å, has the usual value for the length of S–C(Ph) bonds.

The plane of the fragment HgSC(1) (Table 4) forms an angle of 24.6° with the plane of the benzene ring C(1)... C(6), and the fragment PhHg is directed towards the dimethylamino-group of the thiophenol ring as a result of rotation about the S–C(1) bond. The mercury atom is brought closer to the nitrogen atom, to a distance of 2.657(6) Å, which is ~ 0.4-0.5 Å shorter than the sum of the van der Waals radii of the atoms of mercury (1.5-1.6 Å) [12, 15, 16] and nitrogen (1.55 Å) [17], and corresponds to a secondary coordinate bond. The nitrogen atom has a pyramidal bond configuration (the sum of the valence angles is 336.9°), and its unshared electron pair is oriented towards the mercury atom: The angles Hg ... N–C(2), Hg... N–C(7), and Hg... N–C(8) are 106.2, 112.3, and 100.5° respectively.

The bonds between the nitrogen atom and the substituents are not equivalent: the lengths of two of them, N–C(7) and N–C(8), equal to 1.485(9) and 1.47(1) Å, have the usual values for N–C(sp^3) bonds, whereas the third bond, N–C(2), is much shorter, 1.429(9) Å. At the same time, this last bond is still shorter than the N(1)–C(2) bond (1.452 Å) and longer than the N(4)–C(3) bond (1.387 Å) in the molecule of benzo[b][1,4]diazabicyclo[3,2,1]octane (II) [18]:



As a result of the rigid geometry of this tricyclic molecule, the unshared electron pair of the N(1) atom is oriented approximately parallel to the plane of the benzene ring and is completely removed from conjugation with it. The N(1)−C(2) bond is therefore a "pure" N−C(sp^2) bond, whereas the N(4)−C(3) bond, on the contrary, is a typical "aniline" C−N bond with its typical increased multiplicity. In molecule II the sum of the valence angles at the N(1) atom is 319°, and that at the N(4) atom is 346°, that is the sum of the valence angles at the N atom (336.9°) found in the structure of compound I studied in the present work is also intermediate between these limiting values. Thus in compound I, in spite of the participation of the unshared electron pair of the nitrogen atom in the secondary bond Hg... N, some conjugation between it and the benzene ring C(1)... C(3) is preserved (the angle between the plane C(1)... C(6) and the normal to the plane C(2)C(7)C(8) is 58°).

The secondary bond Hg... N closes a five-membered chelate metal-containing ring, in which the nitrogen atom is not positioned completely symmetrically relative to the triad S−Hg−C(1): The angles N ... Hg−S and N... Hg−C(1) are 76.3(1) and 105.5(2)°. The sum of these angles (181.8°) differs little from 180°, that is the coordination Hg... N does not lead to a significant decrease in the angle between the covalent bonds of the Hg atom situated opposite the N atom.

In the crystal, the molecules are joined to form centrosymmetric dimers by a pair of intermolecular secondary interactions Hg... S' and S... Hg' at a distance of 3.224(2) Å, comparable with that found in the crystals of phenylmercury 2,6-dimethylthiophenolate, 3.18 Å [12], and slightly smaller than the sum of the van der Waals radii (3.35-3.45 Å) of these atoms [15-17]. The angles S'... Hg−S, S'... Hg−C(1'), and S'... Hg... N are 88.44(5), 94.9(2), and 104.6(1)° respectively. Thus with allowance for all the secondary interactions, the mercury atom in the crystal of compound I has a coordination polyhedron of the "swing" type.

The scatter in the bond lengths in the benzene rings of the molecule is 1.38(1)-1.42(1) Å. The benzene rings are planar to within 0.01-0.02 Å, and the nearest atoms of the substituents are only slightly displaced from these planes (see Table 4). All this indicates that the present x-ray structural study has a high accuracy.

Thus the results obtained show that in the crystals of PhHg-derivatives of o-substituted thiophenols, the closure of chelate rings as a result of secondary bonds is possible. In the case of the compound studied, the formation of intermolecular coordinate bonds Hg... S does not prevent intramolecular coordination interaction Hg... N. In this respect, the compound studied is similar to phenylmercury 2-chloro-4-bromophenolate [19], and differs from the PhHg-derivatives of 2-halogeno-substituted benzenesulfonalides [20, 21], in which the intermolecular coordination interactions Hg... O prevent the formation of five-membered chelate rings in the crystal, although data on exchange equilibria [22] indicate that these rings are formed in solution.

Available published data show that in the crystals of all organomercury compounds with mercury−heteroatom (X) bonds which have been studied, intermolecular coordination Hg... X takes place. In this connection, it may be assumed that in the general case, the probability of the existence of intramolecular secondary bonds, and, in particular, chelate rings in the crystals of organomercury derivatives of substituted HN-, HO-, and HS-acids will be determined by the relative strength of the potentially possible intramolecular and intermolecular coordination interactions.

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STRUCTURE OF ORGANOPHOSPHORUS COMPOUNDS.

XIX.* AN X-RAY STRUCTURAL AND SPECTRAL STUDY OF o-DIMETHYLPHOSPHORYLBENZOIC ACID

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UDC 548.737

An x-ray structural study of o-$Me_2P(O)C_6H_4COOH$ has been carried out (diffractometer, λMo, recording at −120°C, 1351 reflections, full-matrix anisotropic-isotropic (H atoms) refinement, $R = 0.0318$). In the crystal, the molecules are joined in infinite chains along the b axis by H-bonds O−H...O (O...O 2.572 Å, O−H 0.87 Å, H...O 1.70 Å, angle OHO 175°), involving the O atoms of the phosphoryl group (P=O 1.498 Å) and the OH group (C−OH 1.313 Å). The bond lengths are P−C(Me) and P−C(Ph) 1.785 and 1.883 Å, C(Ph)−COOH 1.494 Å; the key atoms P and C of the o-substituents are displaced from the average plane of the ring by 0.027 and 0.117 Å respectively; the carboxyl group forms an angle of 14.6° with this plane. In the infrared spectra, the intermolecular H-bond is revealed by a three-component band (2800, 2500, and 1900 cm^{-1}), typical of H-bonds of the type C(O)OH...O=P, and also by a low-frequency displacement of the band due to the vibration of the phosphoryl group $\Delta\nu_{PO} = 50$ cm^{-1}.

The ability of compounds containing phosphoryl groups to form H-bonds of different strength with proton donors is well known [2]. If the molecule of the phosphoryl compound itself contains active (acidic) H atoms capable of forming H-bonds, the formation of intramolecular H-bonds with the closure of rings of different sizes is possible in a number of cases in these compounds. As shown for the case of ω-diphenylphosphoryl substituted aliphatic alcohols, in dilute solutions in CCl_4, an equilibrium is established between molecules with an intramolecular H-bond P=O...H−O and molecules with a free OH group, the proportion of the latter increasing with increase in the length of the polymethylene chain [3]. It has also been established [3] that in solutions, the strength of the intramolecular H-bond (ΔH) increases with increase in the number of units in the ring which it forms in the order $5 < 6 < 7 \approx 8$. At the same time, the crystals of α-, β-, and γ-hydroxyphosphoryl compounds and their derivatives generally contain intermolecular H-bonds, with the formation of H-bonded centrosymmetric dimers [4-6] or infinite H-bonded chains [3, 7].

In this connection, it was of interest to study the influence of the rigidity of the fragment connecting the phosphoryl and hydroxyl groups of the molecule on the nature of the H-bonds. For this purpose we carried out an x-ray structural and spectral study of o-dimethylphosphorylbenzoic acid (I), whose molecular geometry allows the formation of an intramolecular H-bond with the closure of a seven-membered ring.

EXPERIMENTAL

Since compound I was studied in order to establish the nature of the H-bonds in the crystal, the experiment was carried out at a temperature of −120°C to permit more reliable location of the H atoms. The

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*For Part XVIII, see [1].

Institute of Heteroorganic Compounds, Academy of Sciences of the USSR. Institute of Physiologically Active Substances, Academy of Sciences of the USSR. Translated from Zhurnal Strukturnoi Khimii, Vol. 22, No. 5, pp. 100-104, September-October, 1981. Original article submitted June 18, 1980.

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