Crystal and molecular structure of organic semiconductor $(ET)_8[Hg_4Cl_{12}] \cdot 2C_6H_6$

O. A. D'yachenko, V. V. Gritsenko, and R. N. Lyubovskaya*

Institute of Chemical Physics in Chernogolovka, Russian Academy of Sciences, 142432 Chernogolovka, Moscow Region, Russian Federation. Fax: +7 (096) 576 4009. E-mail: doa@icp.ac.ru

A new organic semiconductor, $(ET)_8[Hg_4Cl_{12}] \cdot 2C_6H_6$, obtained in the $ET^+-HgCl_3^--PhF$ system has been studied by X-ray structural analysis. Radical cations of bis(ethylenedithio)tetrathiafulvalene (ET) in the organic layer of the structure are packed in stacks of α-type. The average angle between the planes of ET cations from adjacent stacks is 50.1°. The anionic layer is formed by four-charge centrosymmetric $[Hg_4Cl_{12}]^{4-}$ complexes and benzene solvate molecules. A comparative crystal-chemical study of the salts obtained by the reaction in the $ET^+-HgX_3^--PhY$ system (where X = Cl, Br, and I; Y = F, Cl, and Br) made it possible to reveal a substantial effect of the sizes of the X and Y atoms on the composition of the salts and on the structural characteristics of the layers, which are responsible for the various conductivities of these salts.

Key words: organic conductors, bis(ethylenedithio)tetrathiafulvalene, mercury halides, crystal structure.

A study of the temperature dependence of the conductivity of radical cation salts of the type $(ET)_8[Hg_4X_{12}(PhY)_2]$ (**1**: X = Y = Cl, **2**: X = Cl and Y = Br; **3**: X = Br and Y = Cl; **4**: X = Y = Br), based on bis(ethylenedithio)tetrathiafulvalene (ET), demonstrated[1] that the temperature of the metal—dielectric transition in these compounds depends substantially on the nature of the X and Y atoms. The effect of the same atom (X = Y) is more pronounced in the composition of the HgX_3^- anion than in the composition of the PhY solvate. According to the results of X-ray structural studies,[2—4] salts **1—4** are isostructural and are characterized by the same types of structures of the anions and anionic and cationic layers. We found that a decrease in the sizes of the anionic $[Hg_4X_{12}(PhY)_2]^{4-}$ complexes in going from Br derivatives to Cl derivatives results in a closer arrangement of ET radical cation salts in their conducting layers as evidenced by the shortened intermolecular S...S contacts (Table 1). This shortening of the S...S contacts suggests that the inverse dependence between the temperature of the metal—dielectric transition and the densities of conducting layers in salts **1—4** occurs.

Further crystal-chemical studies of new salts obtained in the same $ET^+-HgX_3^--PhY$ system, in which salts **1—4** were obtained, demonstrated that in some cases, the solvate molecules captured from the PhY solvent can affect not only the structure of the conducting layer but also the crystal structure of the compound as a whole. Thus, the $(ET)_8[Hg_4Br_{12}(MeC_6H_4Cl)_2]$ salt (**5**) obtained along with salt **3** in the $ET^+-HgBr_3^--PhCl$ system, differs from the latter in the composition of solvate molecules, which results in different structures of anions, anionic and cationic layers, and accounts for the difference in their physical properties: compound **5** is a semiconductor; compound **3** is a metal.[4]

In this work, as part of a continuing study of the effect of the nature of the solvate on the structure and properties of conducting salts, we performed an X-ray structural study of the semiconductor $(ET)_8[Hg_4Cl_{12}] \cdot 2C_6H_6$ (**6**) obtained in the $ET^+-HgCl_3^--PhF$ system. According to the results of the study performed, fluorobenzene is not involved in the crystal structure of **6**; benzene acts as a solvate. Apparently, as in the synthesis of salt **5**, one reagent is involved in the reaction of electrochemical oxidation of ET, whereas the salt obtained contains another reagent, which is contained in the initial reagent as an impurity.

Experimental

Compound **6** crystallizes as black thin plates belonging to the triclinic system. The principal crystallographic data are as follows: $(C_{10}H_8S_8)_8Hg_4Cl_{12}(C_6H_6)_2$, M = 4461.6, a = 17.10(1) Å, b = 20.75(1) Å, c = 12.020(9) Å, α = 94.51(4), β = 111.39(5), γ = 110.60(5)°, V = 3610.5 Å^3, space group $P\bar{1}$, Z = 1, d_{calc} = 2.05 g cm^{-3}, $F(000)$ = 2194.

X-ray structural study was carried out with a rectangular-parallelepiped-shaped single crystal with dimensions of 0.42×0.11×0.07 mm. Intensities of 9620 independent reflec-

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Table 1. Shortened intermolecular S...S contacts (<3.68 Å) in structures **1—4**

Contact	*d*/Å			
	1 (metal up to 1.3 K)	**2** (T_{M-D} = 10 K)	**3** (T_{M-D} = 90 K)	**4** (T_{M-D} = 125 K)
S(1)—S(18)	3.515(6)	3.525(5)	3.550(9)	3.601(9)
S(1)—S(20)	3.430(6)	3.440(5)	3.443(8)	3.471(9)
S(2)—S(6)	3.590(5)	3.623(5)	—	—
S(2)—S(8)	3.410(5)	3.429(5)	3.508(6)	3.555(8)
S(2)—S(13)	—	—	—	3.667(9)
S(5)—S(24)	3.402(5)	3.410(5)	3.441(7)	3.468(8)
S(5)—S(29)	—	—	—	3.673(9)
S(5)—S(31)	3.580(5)	3.594(5)	3.601(3)	3.611(9)
S(7)—S(24)	3.396(4)	3.396(5)	3.407(8)	3.430(8)
S(7)—S(31)	3.647(5)	3.637(5)	3.632(4)	3.627(9)
S(8)—S(9)	3.599(5)	3.590(5)	—	—
S(9)—S(17)	3.471(6)	3.481(5)	3.502(9)	3.547(9)
S(10)—S(25)	3.401(6)	3.409(5)	3.425(8)	3.457(9)
S(10)—S(27)	3.479(5)	3.489(5)	3.454(7)	3.469(8)
S(11)—S(17)	3.479(5)	3.493(5)	3.480(8)	3.519(9)
S(14)—S(31)	3.574(5)	3.590(5)	3.557(6)	3.595(8)
S(15)—S(21)	3.462(5)	3.464(5)	3.483(7)	3.511(8)
S(15)—S(23)	3.535(6)	3.543(5)	3.551(8)	3.581(8)
S(16)—S(31)	3.577(4)	3.606(5)	3.617(7)	3.663(7)
S(26)—S(30)	3.615(5)	3.634(5)	3.629(7)	3.652(8)
S(26)—S(32)	3.409(6)	3.421(5)	3.503(8)	3.546(9)

tions (maximum 2θ was 46.5°), of which 4212 were with $I > 3\sigma(I)$, were measured on a four-circle automated KM-4 diffractometer (KUMA DIFFRACTION, Poland) using the ω/2θ scanning technique (λ = 0.7107 Å, graphite monochromator). The positions of atoms were refined anisotropically by the least-squares method to $R = 0.058$ and $R_w = 0.061$, $\omega = 1.0/(\sigma^2(F) + 0.005561F^2)$. The intensities of Bragg reflections were corrected for X-ray absorption (μ(Mo-Kα) = 5.40 mm^{-1}) by the crystal (the Diffabs program[5]). Hydrogen atoms were not located. Atomic coordinates and equivalent isotropic fac-

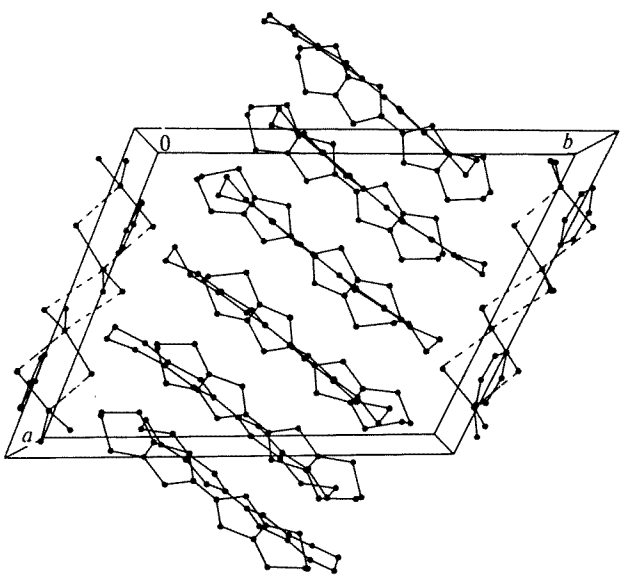


Fig. 1. The *ab* projection of the crystal structure of $(ET)_8[Hg_4Cl_{12}] \cdot 2C_6H_6$ salt.

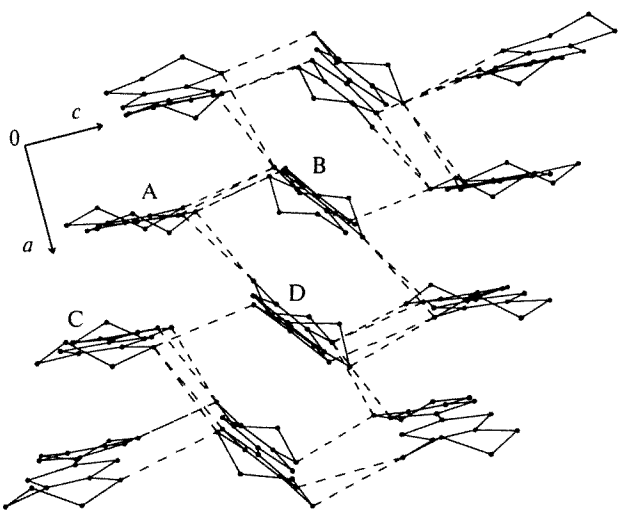


Fig. 2. Packing of ET radical cations in the conducting layer of the $(ET)_8[Hg_4Cl_{12}] \cdot 2C_6H_6$ compound. Dashed lines indicate cation—cation contacts.

Table 2. Atomic coordinates and equivalent isotropic thermal factors (B_{eq}) in the $(ET)_8[Hg_4Cl_{12}] \cdot 2C_6H_6$ structure

Atom	*x*	*y*	*z*	B_{eq}/Å^2
Hg(1)	0.5988(1)	0.9794(1)	0.4435(1)	5.2
Hg(2)	0.8684(1)	1.0252(0)	0.5668(1)	4.8
Cl(1)	0.5293(4)	0.9246(3)	0.5722(5)	5.1
Cl(2)	0.7428(4)	1.0888(3)	0.5543(5)	4.8
Cl(3)	0.5279(4)	0.9458(3)	0.2240(4)	4.5
Cl(4)	0.9590(4)	1.0511(3)	0.7868(5)	4.4
Cl(5)	0.7364(4)	0.9091(3)	0.4676(5)	4.9
Cl(6)	0.9436(4)	1.0862(3)	0.4483(5)	6.4
S(1)	1.0360(3)	0.6676(3)	0.6260(4)	3.5
S(2)	0.8981(3)	0.5212(2)	0.5073(4)	3.3
S(3)	0.7543(3)	0.3590(2)	0.4100(4)	2.8
S(4)	0.6435(3)	0.2051(2)	0.3570(4)	3.3
S(5)	0.6302(3)	0.2062(2)	0.0581(4)	3.1
S(6)	0.7416(3)	0.3603(2)	0.1602(4)	2.7
S(7)	0.8837(3)	0.5280(2)	0.2582(4)	2.9
S(8)	1.0175(3)	0.6760(2)	0.3257(4)	3.3
S(9)	1.3615(3)	0.6944(2)	0.1702(4)	2.8
S(10)	1.2265(3)	0.5467(2)	0.0959(4)	3.0
S(11)	1.0913(3)	0.3795(2)	0.0031(4)	2.7
S(12)	0.9837(3)	0.2239(2)	−0.0835(4)	3.0
S(13)	0.8488(3)	0.2562(2)	0.0597(4)	3.1
S(14)	0.9776(3)	0.4051(2)	0.1202(4)	2.7
S(15)	1.1146(3)	0.5716(2)	0.2165(4)	2.8
S(16)	1.2285(3)	0.7251(2)	0.3174(4)	3.0
S(17)	0.4229(3)	0.2419(2)	1.1161(4)	3.2
S(18)	0.5373(3)	0.3951(2)	1.1754(4)	3.1
S(19)	0.6749(3)	0.5597(2)	1.2760(4)	3.2
S(20)	0.8038(3)	0.7088(2)	1.3964(4)	3.4
S(21)	0.8035(3)	0.7166(2)	1.1043(4)	2.8
S(22)	0.6728(3)	0.5670(2)	1.0313(4)	2.9
S(23)	0.5301(3)	0.3991(2)	0.9282(4)	3.0
S(24)	0.4176(3)	0.2463(2)	0.8202(4)	3.1
S(25)	0.5453(3)	0.3155(2)	0.4320(4)	3.3
S(26)	0.6702(3)	0.4645(2)	0.5570(4)	3.2
S(27)	0.8102(3)	0.6306(2)	0.6583(4)	2.6
S(28)	0.9307(3)	0.7828(2)	0.7073(4)	3.1
S(29)	0.8004(3)	0.8110(2)	0.8586(4)	3.2
S(30)	0.6993(3)	0.6553(2)	0.7812(4)	3.0
S()31	0.5563(3)	0.4899(2)	0.6744(4)	3.4
S(32)	0.4102(3)	0.3444(3)	0.5743(5)	3.6
C(1)	1.057(1)	0.7457(8)	0.561(1)	4.0
C(2)	1.096(1)	0.7410(8)	0.465(1)	3.7
C(3)	0.9679(9)	0.6023(8)	0.490(1)	2.7
C(4)	0.9600(9)	0.6032(8)	0.374(1)	2.9
C(5)	0.8498(9)	0.4772(7)	0.355(1)	2.6
C(6)	0.7896(9)	0.4071(7)	0.313(1)	2.5
C(7)	0.6917(9)	0.2766(8)	0.300(1)	2.6
C(8)	0.6862(9)	0.2768(8)	0.186(1)	3.2
C(9)	0.613(1)	0.1323(9)	0.239(1)	6.0
C(10)	0.580(1)	0.1326(9)	0.118(1)	5.8
C(11)	1.3602(9)	0.7793(8)	0.224(1)	3.6
C(12)	1.3452(9)	0.7798(8)	0.345(1)	3.1
C(13)	1.2634(9)	0.6336(8)	0.174(1)	2.7
C(14)	1.2104(9)	0.6450(7)	0.231(1)	2.5
C(15)	1.1321(9)	0.5108(7)	0.129(1)	2.4
C(16)	1.0732(9)	0.4407(7)	0.087(1)	2.6
C(17)	0.9983(9)	0.3056(7)	−0.004(1)	2.4
C(18)	0.9469(9)	0.3160(8)	0.051(1)	3.2
C(19)	0.868(1)	0.1677(8)	−0.108(1)	4.0
C(20)	0.851(1)	0.1724(8)	0.004(1)	3.8
C(21)	0.3384(9)	0.1793(7)	0.970(1)	2.8
C(22)	0.3782(9)	0.1677(8)	0.880(1)	3.0
C(23)	0.4757(9)	0.3149(8)	1.064(1)	3.6
C(24)	0.4726(9)	0.3167(8)	0.953(1)	3.0
C(25)	0.5763(9)	0.4447(8)	1.080(1)	3.0
C(26)	0.6359(9)	0.5161(8)	1.124(1)	3.6
C(27)	0.7444(9)	0.6423(8)	1.263(1)	2.9
C(28)	0.7432(9)	0.6444(8)	1.149(1)	3.0
C(29)	0.8277(9)	0.7872(8)	1.331(1)	3.2
C(30)	0.8789(9)	0.7830(8)	1.251(1)	3.5
C(31)	0.4344(9)	0.2521(8)	0.418(1)	3.8
C(32)	0.422(1)	0.2608(8)	0.534(1)	4.1
C(33)	0.5665(9)	0.3941(8)	0.527(1)	3.3
C(34)	0.515(1)	0.4066(8)	0.581(1)	4.0
C(35)	0.6538(9)	0.5250(8)	0.643(1)	2.8
C(36)	0.7143(9)	0.5962(8)	0.692(1)	2.7
C(37)	0.8391(9)	0.7189(7)	0.725(1)	2.5
C(38)	0.7895(9)	0.7297(7)	0.783(1)	2.8
C(39)	0.907(1)	0.8611(8)	0.728(1)	4.0
C(40)	0.905(1)	0.8755(9)	0.849(1)	4.6
C(41)	0.666(1)	1.0000(9)	1.015(1)	6.1
C(42)	0.681(1)	0.9868(9)	0.908(1)	4.9
C(43)	0.758(1)	0.9831(9)	0.902(1)	5.2
C(44)	0.835(1)	0.9925(9)	1.013(1)	5.1
C(45)	0.824(1)	1.0031(9)	1.124(1)	5.0
C(46)	0.745(1)	1.0102(9)	1.120(1)	4.5

tors are given in Table 2. All calculations were performed using the SHELX-76 and SHELX-86 program packages.[6]

Results and Discussion

The crystal structure of **6** consists of layers (Fig. 1). Organic layers formed by four crystallographically independent ET radical cations (A, B, C, and D) alternate along the *b* axis of the crystal with inorganic layers formed by $[Hg_4Cl_{12}]^{4-}$ anions and benzene solvate molecules.

Like the organic layer of compound **5**, ET radical cations of salt **6** are packed in stacks of α type (Fig. 2). Cationic stacks are parallel to the [10$\bar{1}$] diagonal of the crystal. The dihedral angle between the planes of ET cations from adjacent stacks averages 50.1°. No shortened S...S contacts (<3.68 Å) are observed in stacks. The average interplanar distance in the stack is 3.8 Å. The ET cations of adjacent stacks are linked to each other in a side-to-side fashion *via* shortened S...S contacts (Table 3). Cations A, B, C, and D have 13, 14, 12, and 11 such contacts, respectively. Bond lengths and bond angles in

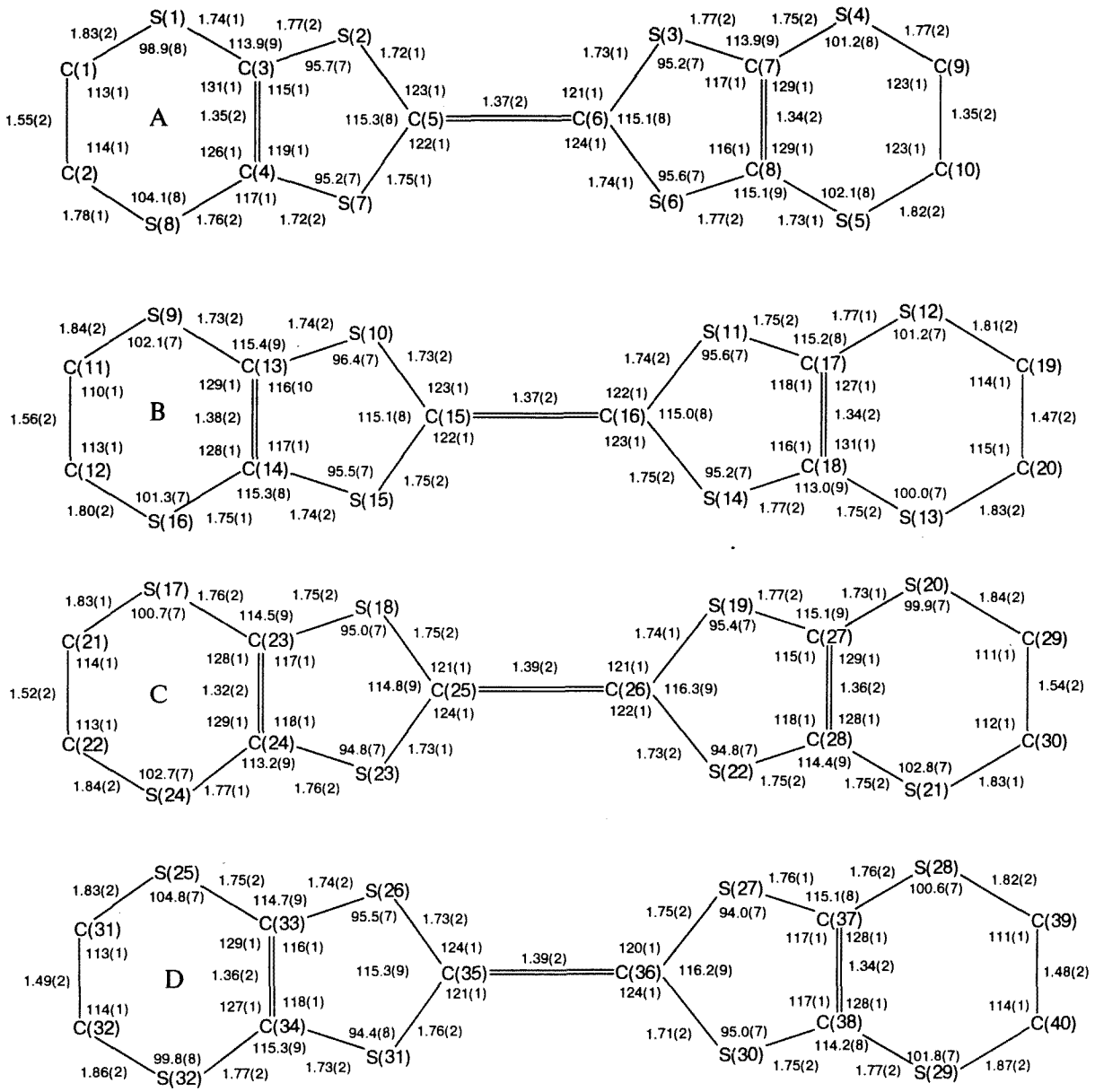


Fig. 3. Bond lengths (Å) and bond angles (deg) in ET radical cations of the $(ET)_8[Hg_4Cl_{12}] \cdot 2C_6H_6$ compound.

ET radical cations averaged over four cations (A, B, C, and D) are given in Fig. 3.

The $[Hg_4Cl_{12}]^{4-}$ anion (Fig. 4) is a four-charge centrosymmetric tetramer that consists of four $HgCl_3$ groups. The Hg(1) and Hg(2) atoms have a slightly distorted trigonal configuration of the Hg—Cl bonds (Table 4). The bond lengths and bond angles for the Hg(1) atom are in the ranges 2.387(4)—2.488(6) Å and 114.6(2)—124.4(2)°, respectively; the corresponding parameters for the Hg(2) atom are in the ranges 2.400(6)—2.465(6) Å and 117.3(2)—119.6(2)°, respectively. The Hg(1) and Hg(2) atoms deviate from the Cl(1)—Cl(2)—Cl(3) and Cl(4)—Cl(5)—Cl(6) planes by 0.11 and 0.32 Å, respectively. The Hg(1) atom forms secondary bonds with the Cl(1′) (3.410(7) Å) and Cl(5) (3.120(7) Å) atoms, which complete its configuration to a trigonal bipyramid. The Hg(2) atom form one secondary (though rather strong) bond with the Cl(2) atom (2.858(7) Å). Therefore, a coordination polyhedron about the Hg(2) atom is a trigonal pyramid. The distances between the

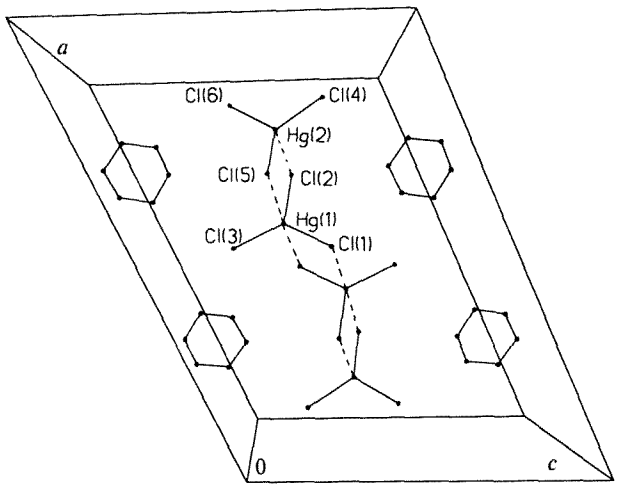


Fig. 4. The *ac* projection of the anionic layer of the $(ET)_8[Hg_4Cl_{12}] \cdot 2C_6H_6$ salt.

metal atoms in the tetramer are as follows: Hg(1)...Hg(1′) 4.381(3) Å and Hg(1)...Hg(2) 4.008(2) Å.

The anionic layer in the structure of **6** consists of $[Hg_4Cl_{12}]^{4-}$ anions and benzene solvate molecules that are parallel to the *a* axis and alternate along the *c* axis (see Fig. 4). Shortened intermolecular contacts between benzene molecules and $[Hg_4Cl_{12}]^{4-}$ anions are not observed. Therefore, unlike structures **1**—**5**, in the structure of **6** solvate molecules are not involved in the anionic complex. Previously,[8] we noted that the $(ET)_4Hg_2I_6$ salt (**7**) obtained in the ET^+—HgI_3^-—PhCl system is the first compound in this system, whose crystal structure contains no solvent as a solvate.

Therefore, crystalline products **1**—**7** obtained in the ET^+—HgX_3^-—PhY system (X = Cl, Br, and I; Y = F, Cl, and Br) can be divided into three groups depending on the position of the PhY solvate in the structure: 1) the solvent is involved in the crystal and forms secondary bonds with the anion: metals **1**—**4** of composition $(ET)_8[Hg_4X_{12}(PhY)_2]$ (X and Y = Cl and Br) and the semiconductor $(ET)_8[Hg_4Br_{12}(MeC_6H_4Cl)_2]$ (**5**); 2) the solvent is involved in the crystal but does not form secondary bonds with the anion: the semiconductor $(ET)_8[Hg_4Cl_{12}] \cdot 2C_6H_6$ (**6**); 3) the solvent is not involved in the crystal: the semiconductor $(ET)_4Hg_2I_6$ (**7**).

Analysis of this classification allows the conclusion that the sizes of the X and Y atoms substantially affect not only the composition of compounds obtained but on their conducting properties as well. Taking into account that metallic properties were found only in Cl and Br derivatives **1**—**4**, the sizes of the X and Y atoms, which are optimum for conductivity, fall within rather narrow ranges: 0.99—1.14 Å and 1.85—1.95 Å for covalent and the van der Waals radii, respectively.

Note that anionic layers in structures **1**—**6** are similar in character of the mutual arrangement of anions and solvent molecules and differ only by the presence or absence of secondary X...Y bonds between them. If this bond occurs, the direction of this bond is of importance. Thus, in the structures of types **1**—**4**, the anionic layer consists of discrete PhY...$[Hg_4X_{12}]^{4-}$...PhY complexes, whereas in salt **5**, the anionic layer consists of polymeric networks.

In salt **6**, secondary X...Y bonds between solvate

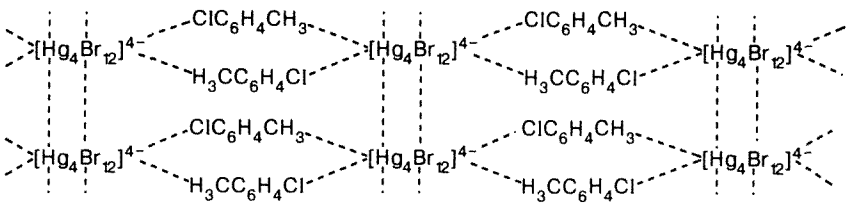
Table 3. Shortened intermolecular S...S contacts (<3.68 Å) in the conducting layer of structure **6**

Contact	*d*/Å
S(1)...S(13) (2−*x*, 1−*y*, 1−*z*)	3.477(6)
S(1)...S(14) (2−*x*, 1−*y*, 1−*z*)	3.554(7)
S(3)...S(25)	3.481(8)
S(3)...S(26)	3.666(7)
S(4)...S(16) (2−*x*, 1−*y*, 1−*z*)	3.582(6)
S(4)...S(25)	3.517(7)
S(5)...S(9) (2−*x*, 1−*y*, 1−*z*)	3.574(6)
S(3)...S(13)	3.502(8)
S(6)...S(9) (2−*x*, 1−*y*, 1−*z*)	3.598(6)
S(6)...S(13)	3.671(7)
S(8)...S(11) (2−*x*, 1−*y*, 1−*z*)	3.580(6)
S(8)...S(15)	3.584(7)
S(8)...S(16)	3.429(8)
S(9)...S(23)	3.517(7)
S(9)...S(24) (2−*x*, 1−*y*, 1−*z*)	3.491(8)
S(12)...S(21) (2−*x*, 1−*y*, 1−*z*)	3.518(8)
S(17)...S(21) (*x*, *y*, 1+*z*)	3.489(6)
S(18)...S(25) (*x*, *y*, 1+*z*)	3.593(6)
S(19)...S(32) (2−*x*, 1−*y*, 1−*z*)	3.564(8)
S(20)...S(27) (*x*, *y*, 1+*z*)	3.637(4)
S(20)...S(28) (*x*, *y*, 1+*z*)	3.445(6)
S(20)...S(32) (1−*x*, 1−*y*, 2−*z*)	3.591(8)
S(21)...S(29)	3.665(6)
S(21)...S(30)	3.529(6)

Table 4. Interatomic distances (*d*) and bond angles (ω) in the $[Hg_4Cl_{12}]^{4-}$ anion of salt **6**

Distance	*d*/Å	Angle	ω/ged
Hg(1)—Cl(1)	2.401(7)	Cl(1)—Hg(1)—Cl(1′)	83.7(2)
Hg(1)—Cl(2)	2.488(6)	Cl(1)—Hg(1)—Cl(3)	124.4(2)
Hg(1)—Cl(5)	3.120(7)	Cl(1′)—Hg(1)—Cl(2)	90.3(2)
Hg(2)—Cl(4)	2.426(6)	Cl(1′)—Hg(1)—Cl(5)	173.1(2)
Hg(2)—Cl(6)	2.400(6)	Cl(2)—Hg(1)—Cl(5)	83.0(2)
Hg(1)—Cl(1′)	3.410(7)	Cl(2)—Hg(2)—Cl(4)	102.0(2)
Hg(1)—Cl(3)	2.387(4)	Cl(2)—Hg(2)—Cl(6)	101.8(2)
Hg(2)—Cl(2)	2.858(7)	Cl(4)—Hg(2)—Cl(6)	117.3(2)
Hg(2)—Cl(5)	2.465(6)	Cl(1)—Hg(1)—Cl(2)	114.6(2)
		Cl(1)—Hg(1)—Cl(5)	100.4(2)
		Cl(1′)—Hg(1)—Cl(3)	88.0(2)
		Cl(2)—Hg(1)—Cl(3)	120.4(2)
		Cl(3)—Hg(1)—Cl(5)	94.1(2)
		Cl(2)—Hg(2)—Cl(5)	89.1(2)
		Cl(5)—Hg(2)—Cl(6)	119.6(2)

Scheme 1



molecules and anions are not observed, and the anionic layer consists of discrete benzene molecules and $[Hg_4Cl_{12}]^{4-}$ anions.

The differences mentioned in the structures of anionic layers of salts **1—4**, **5**, and **6** manifest themselves in the structures of their conducting layers. Thus, in salts **1—4** ET radical cations are packed in the same fashion as in the $(ET)_2ClO_4(TCE)_{0.5}$ salt,[7] *i.e.*, the packing is of the ClO_4 type. In salts **5** and **6**, the ET cations are packed in stacks of the α type. In our opinion, different conducting properties of salts **1—6** are to a large extent accounted for by the difference in the modes of packing of ET radical cations in conducting layers of these salts.

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