C(4)—O(1)	1.314 (13)	C(16)—C(15)	1.400 (15)
C(5)—O(2)	1.238 (12)	C(17)—C(16)	1.319 (15)
C(3)—O(3)	1.354 (14)	C(24)—O(21)	1.317 (11)
C(7)—O(3)	1.331 (14)	C(25)—O(22)	1.240 (12)
C(2)—C(1)	1.450 (18)	C(23)—O(23)	1.342 (14)
C(3)—C(2)	1.489 (16)	C(27)—O(23)	1.341 (16)
C(4)—C(3)	1.363 (15)	C(22)—C(21)	1.38 (3)
C(5)—C(4)	1.445 (14)	C(23)—C(22)	1.495 (17)
C(6)—C(5)	1.420 (16)	C(24)—C(23)	1.330 (14)
C(7)—C(6)	1.326 (16)	C(25)—C(24)	1.440 (14)
C(14)—O(11)	1.346 (14)	C(26)—C(25)	1.424 (14)
C(15)—O(12)	1.258 (11)	C(27)—C(26)	1.290 (19)
Bi···Bi^{i}	3.792 (2)	Bi···O(1^{i})	3.126 (7)
O(2)—Bi—O(1)	69.6 (2)	C(4)—O(1)—Bi	117.6 (6)
O(11)—Bi—O(1)	72.6 (3)	C(5)—O(2)—Bi	113.0 (7)
O(11)—Bi—O(2)	139.2 (2)	C(7)—O(3)—C(3)	119.3 (9)
O(12)—Bi—O(1)	142.8 (3)	C(3)—C(2)—C(1)	118.1 (13)
O(12)—Bi—O(2)	144.5 (2)	C(2)—C(3)—O(3)	110.7 (10)
O(12)—Bi—O(11)	70.3 (2)	C(4)—C(3)—C(2)	126.1 (12)
O(21)—Bi—O(1)	85.3 (3)	C(6)—C(5)—O(2)	123.6 (10)
O(21)—Bi—O(2)	79.4 (3)	C(13)—C(12)—C(11)	112.3 (13)
O(21)—Bi—O(11)	82.8 (3)	C(12)—C(13)—O(13)	113.3 (11)
O(21)—Bi—O(12)	88.3 (3)	C(14)—C(13)—C(12)	126.0 (13)
O(22)—Bi—O(1)	139.1 (3)	C(23)—C(22)—C(21)	113.8 (16)
O(22)—Bi—O(2)	73.3 (3)	C(22)—C(23)—O(23)	111.3 (11)
O(22)—Bi—O(11)	133.8 (3)	C(24)—C(23)—C(22)	124.8 (12)
O(22)—Bi—O(12)	71.2 (3)	C(26)—C(25)—O(22)	124.0 (11)
O(22)—Bi—O(21)	71.4 (3)	C(26)—C(25)—C(24)	115.0 (11)

Symmetry code: (i) $-x, -y, -z$.

Data collection and cell refinement: *XSCANS* (Fait, 1991). Data reduction and structure solution: *SHELXTL/PC* (Sheldrick, 1990). Refinement: *SHELX76* (Sheldrick, 1976). Molecular graphics: *SHELXTL/PC XP*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HU1084). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Diaquadiphenylbis(*p*-toluenesulfonato)lead–Water (1/1)

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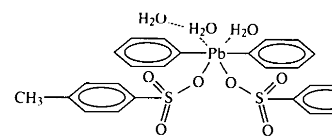
Abstract

The polyhedron around the Pb atom in $[Pb(C_7H_7SO_3)_2(C_6H_5)_2(H_2O)_2].H_2O$ is a slightly distorted pentagonal bipyramid, with two C(phenyl) atoms in the apical positions and three sulfonate O atoms and two O atoms of the coordinated water molecules forming the pentagonal plane. One sulfonate ligand is monodentate, the second is bidentate and links a neighbouring molecule to form a chain {*i.e. catena*-poly[diaquadiphenyl(*p*-toluenesulfonato-*O*)lead-μ-(*p*-toluenesulfonato)-*O*:*O'*]}. The chains are linked by the non-coordinated water molecules to form a two-dimensional network.

Comment

The present work continues structural studies on organometal organosulfonates (Huber, Westhof & Preut, 1987; Preut, Rüther & Huber, 1986; Rüther, Huber & Preut, 1985, 1986, 1988) and in this paper we present the first structure determination of an organolead organosulfonate, diaquadiphenylbis(*p*-toluenesulfonato)lead–water (1/1), (I), by single-crystal X-ray diffraction.



(I)

The coordination polyhedron around Pb is a slightly distorted pentagonal bipyramid (Fig. 1). The central atom is surrounded by two C(phenyl) atoms in apical positions with three sulfonate O atoms and two water O atoms forming the pentagonal plane. One of the sulfonate ligands is monodentately bonded to Pb, the distance Pb(1)—O(8) [2.447 (6) Å] being the shortest Pb—O distance observed in the molecule. The distance between Pb and O of the bidentate sulfonate [Pb(1)—O(1) 2.478 (6) Å] is consistently longer. A similar distance is found between Pb and O of one of the coordinated water molecules [Pb(1)—O(22) 2.494 (5) Å], while the distance to the second coordinated water molecule is somewhat longer [Pb(1)—O(33) 2.518 (6) Å]. Both Pb—O distances are longer than the sum of the covalent radii (2.20 Å; Bondi, 1964), but appreciably shorter than the sum of the van der Waals radii (3.5 Å; Bondi, 1964). In diphenyllead 2,6-pyridinedicarboxylate–water, the distance between Pb and O of the coordinated water was found to be 2.472 (5) Å (Preut, Huber & Hoffmann, 1988). The pentagonal plane is completed by the O(3*a*) atom of the bidentate sulfonate from a neighbouring molecule, the distance of 2.617 (5) Å being substantially shorter than 3.5 Å. This interaction links the molecules to form a linear chain. The deviations from planarity of the atoms constituting the pentagonal plane are rather small: Pb(1) 0.030 (2), O(1) −0.058 (4), O(3*a*) −0.068 (4), O(8) 0.004 (4), O(22) 0.064 (4) and O(33) 0.028 (4) Å.

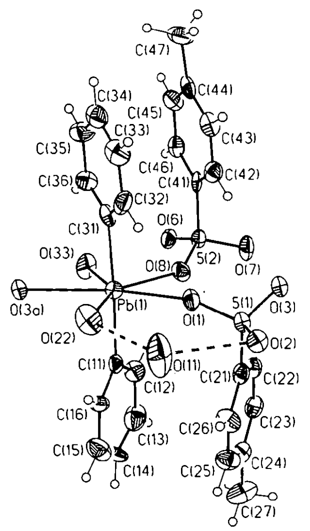


Fig. 1. View of part of the crystal structure, showing 50% probability displacement ellipsoids and the atom-numbering scheme.

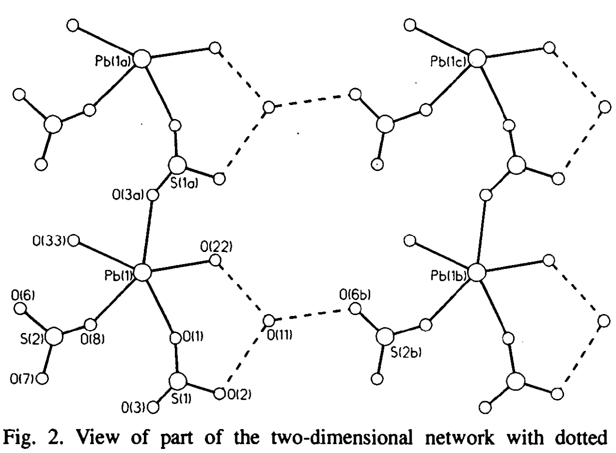


Fig. 2. View of part of the two-dimensional network with dotted lines indicating inter- and intramolecular hydrogen bonds. The aryl groups have been omitted for clarity.

The third water molecule, $H_2O(11)$, is connected by three-centre hydrogen bonds to one chain [O(11)···O(22) 2.740 (10), O(11)···O(2) 2.935 (10) Å, O(2)···O(11)···O(22) 103.1 (3)°] (Fig. 2) and also forms a hydrogen bond to O(6*b*) of the monodentate sulfonate ligand of a neighbouring chain [O(11)···O(6*b*) 2.973 (9) Å, O(22)···O(11)···O(6*b*) 124.5 (4) and O(2)···O(11)···O(6*b*) 131.7 (4)°], thereby bridging the chains.

An interesting feature of the structure is the alignment of parallel alternating phenyl and tolyl groups on one side of each chain. The second phenyl and tolyl groups of each molecule are also parallel, forming staggered pairs on the opposite side of the chain.

Experimental

The title compound was obtained by a redistribution reaction of (acetonyl)(phenyl)bis(4-tolylsulfonato)lead in CD_3OD/D_2O. Single crystals were obtained from the same solvent system.

Crystal data

$[Pb(C_7H_7SO_3)_2(C_6H_5)_2(H_2O)_2].H_2O$
$M_r = 757.81$
Orthorhombic
$P2_12_12_1$
$a = 7.144$ (2) Å
$b = 11.328$ (3) Å
$c = 32.658$ (9) Å
$V = 2642.9$ (13) Å^3
$Z = 4$
$D_x = 1.905$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 7.7–13.9°$
$\mu = 6.595$ mm^{-1}
$T = 291$ (1) K
Block
0.24 × 0.20 × 0.18 mm
Colourless

Data collection

Nicolet *R*3*m*/*V* diffractometer
$\omega/2\theta$ scans (1.5–15.0° min^{-1} in θ)
Absorption correction: empirical
$T_{min} = 0.923$, $T_{max} = 1.000$
6187 measured reflections
4689 independent reflections
3724 observed reflections [$I > 2\sigma(I)$]
$R_{int} = 0.0317$
$\theta_{max} = 25.06°$
$h = -8 \rightarrow 1$
$k = 0 \rightarrow 13$
$l = -38 \rightarrow 38$
6 standard reflections monitored every 300 reflections intensity variation: $< \pm 3.4\%$

Refinement

Refinement on F^2
$R(F) = 0.0360$
$wR(F^2) = 0.0670$
$S = 0.99$
4689 reflections
345 parameters
Only H-atom *U*'s refined
$w = 1/[\sigma^2(F_o^2) + (0.0293P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.151$ e Å^{-3}
$\Delta\rho_{min} = -0.732$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C) *Tables* 4.2.6.8 and 6.1.1.4

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Pb(1)	−0.10660 (5)	0.12863 (3)	0.123961 (10)	0.02401 (9)
S(1)	0.3925 (4)	0.2386 (2)	0.13672 (6)	0.0238 (5)
S(2)	0.1973 (3)	−0.1410 (2)	0.11534 (6)	0.0250 (5)
O(1)	0.2039 (8)	0.2252 (5)	0.1191 (2)	0.0279 (14)
O(2)	0.4548 (7)	0.3619 (6)	0.1341 (2)	0.0295 (15)
O(3)	0.5307 (7)	0.1572 (5)	0.1184 (2)	0.031 (2)
O(6)	0.0617 (8)	−0.2324 (5)	0.1272 (2)	0.031 (2)
O(7)	0.3875 (9)	−0.1669 (5)	0.1283 (2)	0.0389 (15)
O(8)	0.1355 (9)	−0.0234 (5)	0.1295 (2)	0.031 (2)
C(11)	−0.1162 (13)	0.1392 (8)	0.1897 (2)	0.024 (2)
C(12)	−0.0615 (4)	0.0455 (9)	0.2124 (3)	0.039 (3)
C(13)	−0.0692 (15)	0.0548 (11)	0.2557 (3)	0.052 (3)
C(14)	−0.1339 (16)	0.1546 (10)	0.2731 (3)	0.044 (3)
C(15)	−0.1805 (16)	0.2497 (10)	0.2508 (3)	0.048 (3)
C(16)	−0.1678 (413)	0.2459 (9)	0.2087 (3)	0.033 (2)
C(21)	0.3791 (15)	0.2012 (7)	0.1893 (3)	0.026 (2)
C(22)	0.4201 (14)	0.0896 (7)	0.2023 (3)	0.026 (2)
C(23)	0.4151 (15)	0.0645 (8)	0.2439 (3)	0.034 (2)
C(24)	0.3679 (14)	0.1489 (8)	0.2725 (2)	0.029 (2)
C(25)	0.3228 (14)	0.2605 (9)	0.2586 (3)	0.038 (3)
C(26)	0.3331 (14)	0.2875 (8)	0.2176 (3)	0.037 (3)
C(27)	0.3606 (17)	0.1197 (11)	0.3176 (3)	0.058 (3)
C(31)	−0.1146 (13)	0.1218 (8)	0.0583 (2)	0.026 (2)
C(32)	−0.0342 (13)	0.2100 (9)	0.0365 (3)	0.036 (2)
C(33)	−0.0435 (15)	0.2094 (9)	−0.0057 (3)	0.042 (3)
C(34)	−0.1318 (14)	0.1172 (9)	−0.0257 (3)	0.040 (2)
C(35)	−0.2112 (7)	0.0310 (9)	−0.0038 (3)	0.041 (3)
C(36)	−0.2078 (14)	0.0306 (8)	0.0387 (3)	0.032 (3)
C(41)	0.1998 (11)	−0.1354 (9)	0.0613 (2)	0.024 (2)
C(42)	0.2786 (14)	−0.0408 (8)	0.0412 (3)	0.032 (2)
C(43)	0.2731 (15)	−0.0339 (8)	−0.0008 (3)	0.034 (2)
C(44)	0.1846 (12)	−0.1208 (10)	−0.0243 (3)	0.030 (2)
C(45)	0.1106 (17)	−0.2162 (8)	−0.0036 (3)	0.035 (2)
C(46)	0.1134 (14)	−0.2234 (7)	0.0390 (2)	0.027 (2)
C(47)	0.1669 (14)	−0.1127 (10)	−0.0700 (2)	0.048 (3)
O(22)	−0.1622 (9)	0.3451 (5)	0.1171 (2)	0.044 (2)
O(33)	−0.2576 (9)	−0.0714 (5)	0.1304 (2)	0.039 (2)
O(11)	0.1202 (12)	0.5082 (6)	0.1219 (4)	0.076 (3)

Table 2. *Selected geometric parameters* (Å, °)

Pb(1)—C(31)	2.146 (7)	O(11)···O(6^{ii})	2.973 (9)
Pb(1)—C(11)	2.151 (7)	S(1)—O(2)	1.469 (6)
Pb(1)—O(8)	2.447 (6)	S(1)—O(1)	1.473 (6)
Pb(1)—O(1)	2.478 (6)	S(1)—O(3)	1.477 (6)
Pb(1)—O(22)	2.494 (5)	S(2)—O(7)	1.453 (6)
Pb(1)—O(33)	2.518 (6)	S(2)—O(6)	1.469 (6)
Pb(1)—O(3^{i})	2.617 (5)	S(2)—O(8)	1.478 (6)
O(11)···O(2)	2.935 (10)	S(1)—C(21)	1.770 (8)
O(11)···O(22)	2.740 (10)	S(2)—C(41)	1.765 (8)
C(31)—Pb(1)—C(11)	176.5 (3)	O(1)—Pb(1)—O(22)	72.7 (2)
C(31)—Pb(1)—O(8)	93.9 (3)	O(3^{i})—Pb(1)—O(22)	73.4 (2)
C(11)—Pb(1)—O(8)	89.3 (3)	O(3^{i})—Pb(1)—O(33)	72.1 (2)
C(31)—Pb(1)—O(1)	88.6 (3)	O(8)—Pb(1)—O(33)	70.3 (2)
C(11)—Pb(1)—O(1)	93.9 (3)	Pb(1)—O(1)—S(1)	147.0 (4)
C(31)—Pb(1)—O(22)	86.7 (3)	Pb(1)—O(8)—S(2)	145.3 (4)
C(11)—Pb(1)—O(22)	91.7 (3)	Pb(1)—O(3^{i})—S(1^{i})	135.3 (3)
C(31)—Pb(1)—O(33)	92.2 (3)	S(1)—O(2)—O(11)	107.4 (3)
C(11)—Pb(1)—O(33)	87.4 (3)	O(22)···O(11)···O(2)	103.1 (3)
C(31)—Pb(1)—O(3^{i})	84.8 (3)	O(22)···O(11)···O(6^{ii})	124.5 (4)
C(11)—Pb(1)—O(3^{i})	91.7 (3)	O(2)···O(11)···O(6^{ii})	131.7 (4)
O(1)—Pb(1)—O(8)	71.5 (2)		

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

Systematic absences ($h00$) $h = 2n + 1$ and ($0k0$) $k = 2n + 1$ indicated space group $P2_12_12$ (No. 18), but the structure was solved by standard Patterson and difference Fourier methods (*SHELXTL-Plus*; Sheldrick, 1987) and refined satisfactorily in space group $P2_12_12_1$ (No. 19) by full-matrix least-squares calculations (*SHELXL*93; Sheldrick, 1994). The absolute structure of the crystal used for the investigation was established with the Flack (1983) parameter $x = -0.021$ (9). The H atoms, except those of the water molecules, were placed in geometrically calculated positions and refined with common isotropic temperature factors for different C—H types [H_{aryl}: C—H 0.93 Å, U_{iso} 0.032 (9) Å^2; H_{alkyl}: C—H 0.96 Å, U_{iso} 0.119 (20) Å^2].

As well as *SHELXTL-Plus* and *SHELXL*93, programs used include *PARST* (Nardelli, 1983), *PLATON* (Spek, 1990) and *MISSYM* (Le Page, 1987).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and least-squares-planes data have been deposited with the IUCr (Reference: KA1085). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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