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Poly[bis(μ-pentafluorobenzenethiolato)lead(II)]

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Key indicators

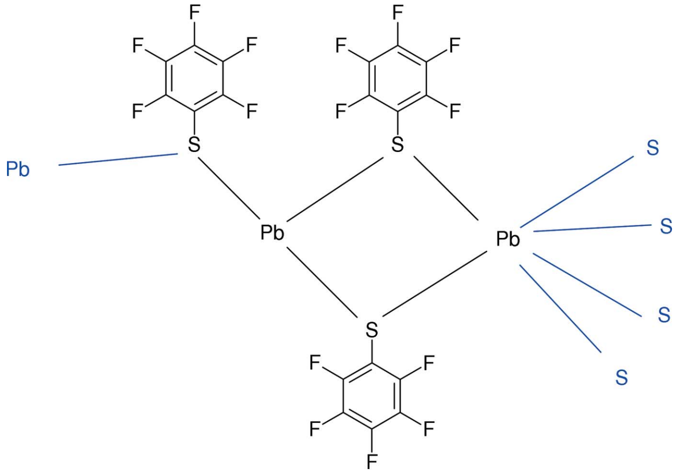
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.015 Å
R factor = 0.051
wR factor = 0.149
Data-to-parameter ratio = 15.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $[Pb(SC_6F_5)_2]_n$, exhibits a layered structure containing two differently hexacoordinated Pb^{II} atoms. One is situated on a centre of inversion and is coordinated by six S atoms, its valence lone pair of electrons being consequently stereochemically inactive. The other Pb atom is pyramidally coordinated by three S atoms and forms three contacts to adjacent F atoms, the stereochemically active lone pair pointing towards the F_3 plane.

Comment

Pb^{II} thiolates without additional donor atoms tend to form thiolate-bridged polymeric solid state structures and hence are soluble only in strongly polar solvents (Dance, 1986). The title compound, (I), which is of interest for synthetic purposes (Peach & Spinney, 1971; Redón *et al.*, 2001; Morales-Morales *et al.*, 2001), is very soluble in acetone and methanol. It was thus of interest to explore its crystal structure and to see whether intra- or intermolecular Pb···F contacts are present.



(I)

Two quite different hexacoordinate Pb^{II} centres are present in (I). Pb1 is situated on an inversion centre, its valence lone pair being consequently stereochemically inactive (Fig. 1). It is coordinated by six S atoms and exhibits a distorted octahedral coordination geometry, with Pb1—S distances between 2.971 (2) and 3.036 (2) Å and *cis* S—Pb1—S angles between 66.49 (6) and 113.51 (6)°.

Atom Pb2 is coordinated by three SC_6F_5 ligands *via* their S atoms. Each ligand additionally forms a Pb···F contact with the F atom in the position *ortho* to the S atom (Fig. 2). The Pb2—S distances range between 2.704 (2) and 2.737 (2) Å and are thus significantly shorter than the Pb1—S bonds, while the

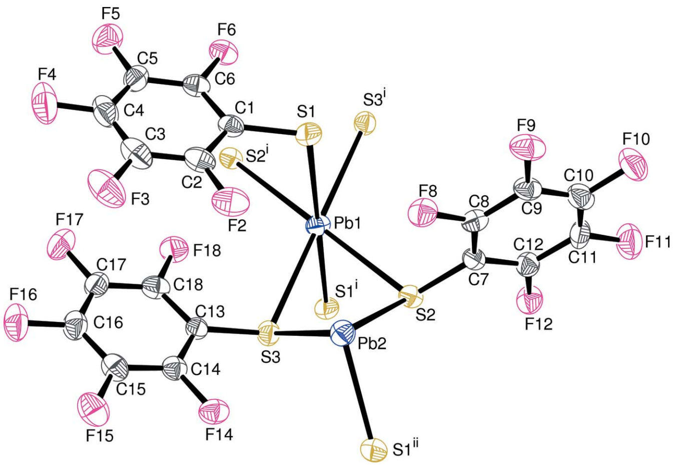


Figure 1
Detail of (I), showing the Pb coordination environments (30% displacement ellipsoids). [Symmetry codes: (i) $1 - x, 1 - y, 1 - z$; (ii) $1 - x, y - \frac{1}{2}, \frac{1}{2} - z$.]

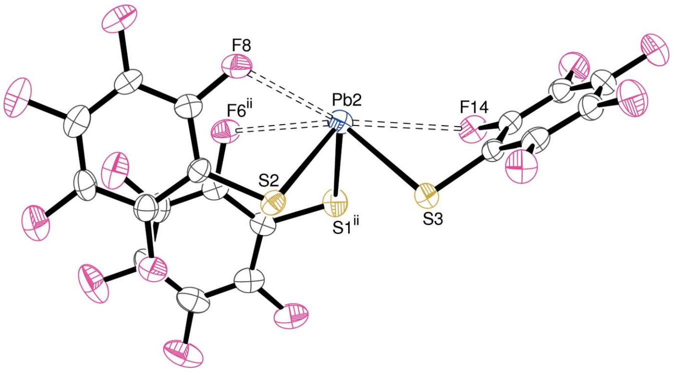


Figure 2
Environment of atom Pb2 in (I), showing the Pb$\cdots$F interactions as dashed lines (30% displacement ellipsids). The symmetry codes are as in Fig. 1.

Pb$\cdots$F contacts are rather long [2.968 (6)–3.245 (6) Å]. The PbS_3 unit exhibits a trigonal–pyramidal configuration with a fairly narrow spread of S—Pb2—S angles between 75.61 (7) and 86.77 (8)°. The stereochemically active lone pair of electrons points towards the F_3 plane, from which Pb2 is displaced by 0.339 (3) Å.

Atoms Pb1 and Pb2 are connected *via* μ^2-S bridges and form a sheet structure consisting of PbS_6 octahedra and PbS_3 pyramids sharing corners and edges (Fig. 3). It is interesting to compare the crystal structure of (I) to that of $Pb(SAr)_2$ (Ar = $C_6H_2{}^tBu_3$-2,4,6; Hitchcock *et al.* 1983). In both, pyramidal PbS_3 units form μ^2-S bridges to another crystallographically different Pb atom. In $Pb(SAr)_2$, this second Pb atom exhibits a pseudo-trigonal–bipyramidal PbS_4 coordination mode, with a stereochemically active lone pair in an equatorial position, and is thus different from the octahedrally coordinated Pb1 in $Pb(SC_6F_5)_2$.

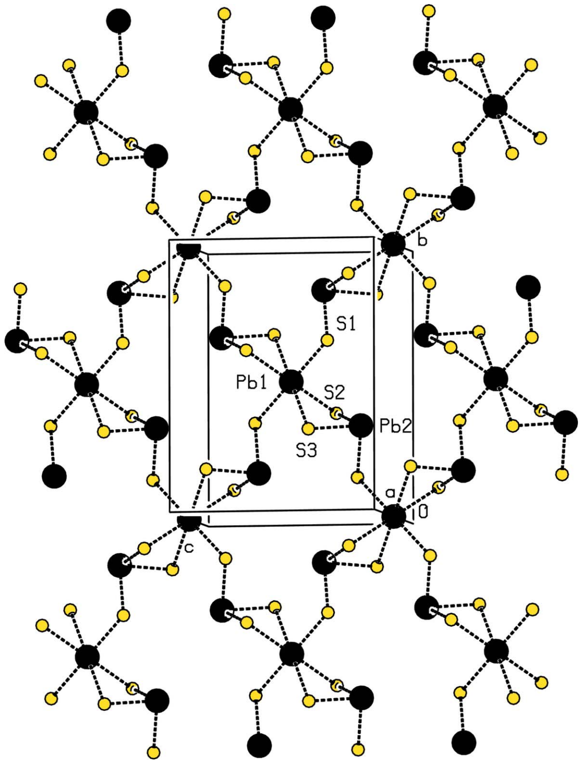


Figure 3
Packing diagram of the Pb and S atoms in (I), showing the sheet structure.

The Pb—S bonds in (I) are longer [mean Pb—S = 3.012 (3) Å for the six-coordinate site and mean Pb—S = 2.721 (3) Å for the three-coordinate site] and hence weaker than those in $Pb(SAr)_2$ [mean Pb—S = 2.788 (3) Å for the four-coordinate site and mean Pb—S = 2.669 (3) Å for the three-coordinate site]. This may explain the enhanced solubility of $Pb(SC_6F_5)_2$ in acetone and methanol, despite the polymeric nature of its structure in the solid state.

Experimental

Compound (I) was prepared according to the literature procedure of Peach (1968). Its purity was confirmed by elemental (C and S) analysis and ^{19}F NMR spectroscopy. Crystals suitable for single-crystal X-ray diffraction were obtained by cooling a saturated solution of the compound in acetone from room temperature to 278 K.

Crystal data

$[Pb(C_6F_5S)_2]$
M_r = 605.43
Monoclinic, $P2_1/c$
a = 14.7217 (5) Å
b = 13.7581 (5) Å
c = 10.5960 (4) Å
β = 95.647 (1)°
V = 2135.73 (13) Å^3
Z = 6
D_x = 2.824 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 12.25 mm^{-1}
T = 298 (2) K
Plate, yellow
0.28 × 0.24 × 0.09 mm

Data collection

Bruker SMART CCD diffractometer
ω scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
T_{min} = 0.03, T_{max} = 0.30 (expected range = 0.033–0.332)
32252 measured reflections
5306 independent reflections
3532 reflections with $I > 2\sigma(I)$
R_{int} = 0.161
θ_{max} = 28.3°

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Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.076P)^2 + 4.1862P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.051$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.149$	$\Delta\rho_{max} = 2.22$ e Å^{-3}
$S = 1.01$	$\Delta\rho_{min} = -1.58$ e Å^{-3}
5306 reflections	
340 parameters	

Table 1
Selected bond lengths (Å).

Pb1—S1	2.971 (2)	Pb2—S2	2.704 (2)
Pb1—S2	3.030 (2)	Pb2—S3	2.721 (2)
Pb1—S3	3.036 (2)	Pb2—S1^{i}	2.737 (3)

Symmetry code: (i) $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$.

The highest peak is located 0.87 Å from atom Pb2 and the deepest hole is located 0.69 Å from the same atom. The high value of R_{int} is due to the poor quality of the crystal used for analysis.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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