Table 2. *Metal–metal and metal–oxygen distances* (Å) *with e.s.d.'s in parentheses*

Pb(1)–Pb(4)	3·910 (3)	Pb(2)–Pb(3)	3·887 (4)
Pb(1)–Pb(5)	3·936 (4)	Pb(2)–Pb(6)	3·865 (4)
Pb(1)–Pb(8)	3·598 (3)	Pb(2)–Pb(7)	3·668 (3)
Pb(4)–Pb(5)	3·628 (3)	Pb(3)–Pb(6)	3·591 (4)
Pb(4)–Pb(8)	3·888 (3)	Pb(3)–Pb(7)	3·892 (3)
Pb(5)–Pb(8)	3·836 (4)	Pb(6)–Pb(7)	3·906 (4)
Pb(1)–O(8)	2·48 (5)	Pb(2)–O(7)	2·35 (3)
Pb(1)–O(1^{i})	2·33 (6)	Pb(2)–O(4^{ii})	2·48 (4)
Pb(1)–O(3^{ii})	2·29 (3)	Pb(2)–O(5^{iii})	2·36 (4)
Pb(1)–O(22^{ii})	2·53 (4)	Pb(2)–O(41)	2·77 (6)
Pb(1)–O(422)	2·96 (6)	Pb(2)–O(42^{iii})	2·98 (7)
Pb(3)–O(7)	2·44 (3)	Pb(4)–O(8)	2·38 (4)
Pb(3)–O(2^{iv})	2·30 (4)	Pb(4)–O(6^{i})	2·37 (6)
Pb(3)–O(4^{ii})	2·33 (3)	Pb(4)–O(3^{ii})	2·55 (4)
Pb(3)–O(11^{ii})	2·61 (5)	Pb(4)–O(32)	2·72 (6)
Pb(5)–O(8)	2·41 (6)	Pb(4)–O(31^{v})	2·74 (4)
Pb(5)–O(1^{i})	2·47 (5)	Pb(6)–O(2^{iv})	2·34 (3)
Pb(5)–O(6^{i})	2·43 (4)	Pb(6)–O(4^{ii})	2·36 (4)
Pb(5)–O(31^{v})	2·56 (3)	Pb(6)–O(5^{iii})	2·49 (4)
Pb(5)–O(121^{i})	2·77 (7)	Pb(6)–O(11^{ii})	2·73 (4)
Pb(5)–O(12^{i})	2·82 (5)	Pb(6)–O(12^{iv})	2·77 (4)
Pb(5)–O(232^{vi})	2·76 (4)	Pb(6)–O(122^{ii})	2·85 (7)
Pb(5)–O(322^{v})	2·83 (5)	Pb(6)–O(432^{ii})	2·91 (4)
Pb(7)–O(7)	2·37 (5)	Pb(8)–O(1^{i})	2·30 (4)
Pb(7)–O(2^{iv})	2·65 (4)	Pb(8)–O(6^{i})	2·39 (4)
Pb(7)–O(5^{iii})	2·40 (4)	Pb(8)–O(3^{ii})	2·46 (7)
Pb(7)–O(42^{iii})	2·46 (4)	Pb(8)–O(21^{i})	2·77 (5)
Pb(7)–O(422^{iii})	2·88 (4)	Pb(8)–O(29^{ii})	2·78 (3)
Pb(7)–O(132^{vii})	2·92 (4)	Pb(8)–O(311^{ii})	2·73 (4)

Symmetry code: (i) $-x, 1-y, -z$; (ii) $x, 1-y, z-\frac{1}{2}$; (iii) $1-x, y, \frac{1}{2}-z$; (iv) $1-x, 1-y, -z$; (v) $-x, 1-y, -z$; (vi) $-x, 1+y, \frac{1}{2}-z$; (vii) $1-x, 1+y, \frac{1}{2}-z$.

Pb–Pb distances range from 3·591 (4) to 3·936 (4) Å (average 3·80 Å) and are similar to those found in the basic lead perchlorates. Metallic lead has Pb–Pb = 3·50 Å (*Tables of Interatomic Distances and Configuration in Molecules and Ions*, 1958).

Fig. 3 illustrates the packing arrangement within half a unit cell ($b/2$). The Pb tetrahedra form sheets linked by styphnate ions. Each styphnate ion is linked to two or more Pb clusters. In other styphnate structures studied there has been a tendency for the anions to lie in parallel planes. This is not observed in this compound.

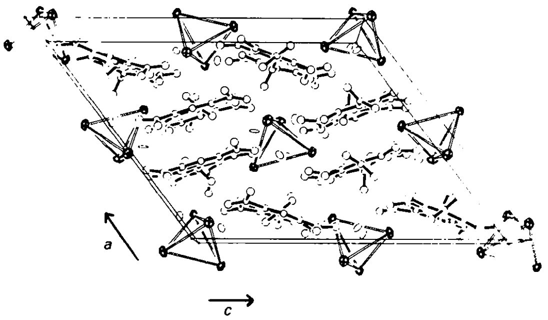

Fig. 3. The packing arrangement within half a unit cell ($a \times b/2 \times c$), viewed along **b**.

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Structure of 2-Hydroxyethanethiolatolead(II) Nitrate, $Pb(SCH_2CH_2OH)(NO_3)$, a Sheet Polymer

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Abstract. $M_r = 346·32$, orthorhombic, $Pbca$, $a = 17·969\ (5)$, $b = 11·860\ (3)$, $c = 6·043\ (2)$ Å, $U = 1287·8$ Å^3, $Z = 8$, $D_x = 3·572$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0·71069$ Å, $\mu = 26·7$ mm^{-1}, $F(000) = 1232$, $T = 291$ K, $R = 0·038$ for 935 observed reflections. The structure consists of polymeric sheets. Pb has an

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irregular sevenfold coordination, with nitrate and 2-hydroxyethanethiolato both acting as bidentate ligands and as bridges between Pb atoms.

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($\text{Å}^2 \times 10^3$)

$U_{\rm eq} = \frac{1}{3}$ (trace of the orthogonalized U_{ij} matrix).

	x	*y*	*z*	$U_{\rm eq}$
Pb	2361 (1)	1327 (1)	1837 (1)	20 (1)
O(1)	3726 (5)	773 (6)	1879 (9)	34 (2)
C(1)	4151 (6)	603 (8)	3882 (16)	27 (3)
C(2)	3849 (7)	1412 (7)	5638 (17)	30 (3)
S	2847 (2)	1259 (2)	6214 (5)	22 (1)
N	887 (5)	1872 (7)	4257 (13)	25 (3)
O(2)	1080 (5)	871 (5)	3731 (11)	32 (2)
O(3)	1346 (4)	2648 (5)	3828 (10)	30 (2)
O(4)	293 (5)	2066 (7)	5126 (12)	46 (3)

Table 2. *Bond lengths* (Å) *and angles* (°)

Pb–O(1)	2·539 (8)	Pb–S	2·786 (3)
Pb–O(2)	2·628 (8)	Pb–O(3)	2·688 (7)
Pb–S$^{\rm i}$	3·017 (3)	Pb–S$^{\rm ii}$	3·113 (2)
Pb–O(3$^{\rm i}$)	2·848 (7)	O(1)–C(1)	1·446 (12)
C(1)–C(2)	1·530 (14)	C(2)–S	1·842 (13)
N–O(2)	1·276 (10)	N–O(3)	1·263 (11)
N–O(4)	1·211 (12)		
O(1)–Pb–S	71·4 (1)	O(1)–Pb–O(2)	142·1 (2)
S–Pb–O(2)	81·7 (2)	O(1)–Pb–O(3)	143·3 (2)
S–Pb–O(3)	78·7 (2)	O(2)–Pb–O(3)	48·0 (2)
O(1)–Pb–S$^{\rm i}$	88·1 (2)	S–Pb–S$^{\rm i}$	93·2 (1)
O(2)–Pb–S$^{\rm i}$	120·2 (2)	O(3)–Pb–S$^{\rm i}$	72·5 (2)
O(1)–Pb–S$^{\rm ii}$	82·1 (2)	S–Pb–S$^{\rm ii}$	97·1 (1)
O(2)–Pb–S$^{\rm ii}$	75·2 (1)	O(3)–Pb–S$^{\rm ii}$	123·1 (2)
S$^{\rm i}$–Pb–S$^{\rm ii}$	162·7 (1)	O(1)–Pb–O(3$^{\rm i}$)	137·3 (2)
S–Pb–O(3$^{\rm i}$)	145·0 (1)	O(2)–Pb–O(3$^{\rm i}$)	78·7 (2)
O(3)–Pb–O(3$^{\rm i}$)	66·6 (1)	S$^{\rm i}$–Pb–O(3$^{\rm i}$)	72·6 (1)
S$^{\rm ii}$–Pb–O(3$^{\rm i}$)	105·4 (1)	Pb–O(1)–C(1)	123·7 (5)
O(1)–C(1)–C(2)	107·8 (8)	C(1)–C(2)–S	114·6 (7)
Pb–S–C(2)	97·1 (3)	Pb–S–Pb$^{\rm iii}$	90·0 (1)
C(2)–S–Pb$^{\rm iii}$	102·3 (3)	Pb–S–Pb$^{\rm iv}$	96·1 (1)
C(2)–S–Pb$^{\rm iv}$	103·7 (3)	Pb$^{\rm iii}$–S–Pb$^{\rm iv}$	152·3 (1)
O(2)–N–O(3)	116·7 (8)	O(2)–N–O(4)	121·6 (8)
O(3)–N–O(4)	121·7 (8)	Pb–O(2)–N	98·9 (5)
Pb–O(3)–N	96·3 (5)	Pb–O(3)–Pb$^{\rm iii}$	95·7 (2)
N–O(3)–Pb$^{\rm iii}$	126·8 (5)		

Symmetry operators: (i) $x, \frac{1}{2}-y, -\frac{1}{2}+z$; (ii) $\frac{1}{2}-x, -y, -\frac{1}{2}+z$; (iii) $x, \frac{1}{2}-y, \frac{1}{2}+z$; (iv) $\frac{1}{2}-x, -y, \frac{1}{2}+z$.

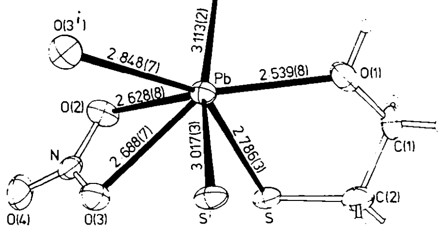

Fig. 1. The asymmetric unit of the structure, together with additional symmetry-related atoms completing the coordination sphere of Pb. Thermal motion is depicted as 50% probability ellipsoids. For symmetry-operator codes, see Table 2. (Distances in Å.)

Introduction. The development of lead(II) thiolate chemistry has been restricted by the insolubility of Pb(S*R*)$_2$ complexes of simple thiolates, and by the paucity of definitive structural data (Borgstrom, Ellis & Reid, 1929; Shaw & Woods, 1971). Freeman, Stevens & Taylor (1974) and Dance & Guerney (1981) have used thiolates containing other functional groups, which increase solubility and allow a more controlled crystallization of the lead(II) complexes, thus producing crystals suitable for diffraction study. We report here the preparation and structure of another such complex, $Pb(SCH_2CH_2OH)(NO_3)$.

The complex was obtained by addition of 2-mercaptoethanol (0·1 cm^3, 1·4 mmol) to an aqueous solution (10 cm^3) of lead nitrate (0·33 g, 2 mmol) at 313 K. Cooling to room temperature produced pale-yellow thin plate crystals, which gave satisfactory analyses.

Experimental. Crystal mounted on glass fibre, 0·61 × 0·27 × 0·02 mm. Stoe–Siemens AED diffractometer. Unit-cell parameters from 2θ values of 32 reflections centred at $\pm\omega$ ($20 < 2\theta < 25°$). 1062 non-extinguished unique reflections with $2\theta < 50°$ and all indices ≥ 0. Profile analysis on-line (Clegg, 1981). No significant variation for three standard reflections. Semiempirical absorption corrections based on azimuthal scan data for sets of equivalent reflections, transmission 0·069 to 0·683 ($R_{\rm int}$ for azimuthal scan data reduced from 0·359 to 0·040). 935 reflections with $F > 4\sigma(F)$. Pb atom from Patterson synthesis, others from difference syntheses. Blocked-cascade refinement on *F*. $w^{-1} = \sigma^2(F) + 0{\cdot}00092F^2$. H atoms constrained to give C–H = 0·96 Å, H–C–H = 109·5°, O–H = 0·96 Å staggered with respect to neighbouring CH$_2$, U(H) = $1{\cdot}2U_{\rm eq}$(C) or $1{\cdot}2U_{\rm eq}$(O). Anisotropic thermal parameters for non-H atoms. Extinction coefficient $x = 6\,(1) \times 10^{-7}$ $[F'_c = F_c/(1 + xF_c^2/\sin 2\theta)^{1/4}]$. Scattering factors from *International Tables for X-ray Crystallography* (1974). 83 parameters, $R = 0{\cdot}038$, $wR = 0{\cdot}049$. Slope of normal probability plot = 1·22. Max. $\Delta/\sigma = 0{\cdot}011$, mean = 0·002. Largest peak in final difference map 3·05 e Å^{-3} close to S, largest hole −1·56 e Å^{-3}. Programs: *SHELXTL* (Sheldrick, 1978), diffractometer program by WC.

Discussion. Atomic coordinates are given in Table 1, bond lengths and angles in Table 2.* Fig. 1 shows the Pb coordination sphere, Fig. 2 the polymeric sheet structure.

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* Tables of structure factors, anisotropic thermal parameters, and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39448 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

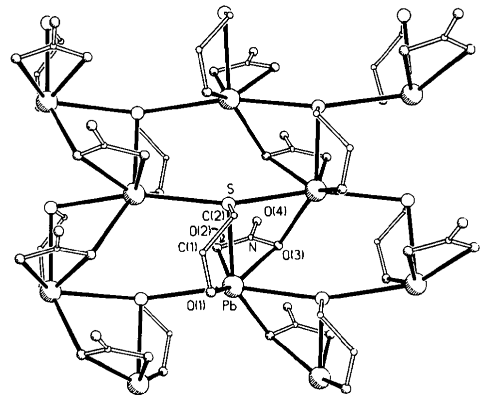

Fig. 2. Part of the polymeric sheet structure viewed along the *a* axis.

The Pb atom is seven-coordinate, but with a wide range of Pb—S and Pb—O bond lengths. The next-nearest S atom is 3·510 (4) Å distant, almost collinear with the 'primary' Pb—S bond (Fig. 2). All other Pb···O distances are >4·3 Å.

The polymeric sheets are based on a $(PbS)_\infty$ array perpendicular to the *a* axis. The nitrate ligands and the other atoms of the SCH_2CH_2OH ligand are arranged above and below the sheet. Each SCH_2CH_2OH ligand is chelating, and forms two additional S—Pb bonds within the sheet; the OH group is non-bridging. Nitrate acts as an essentially symmetrical bidentate ligand for the Pb atom (Addison, Logan, Wallwork & Garner, 1971); in addition, one of the bonded O atoms forms a bridge to another Pb atom.

The sevenfold coordination of Pb, chelating and bridging nitrate, and five-membered $PbSC_2X$ chelate ring are features also observed in 2-(morpholino)-ethanethiolatolead(II) nitrate (Dance & Guerney, 1981). Significant differences between the two structures are the number of bridging O atoms for the nitrate group (one or two), the degree of asymmetry of the Pb—NO_3 bonding (very slight or considerable), and the sheet *versus* chain polymerization. Pb—S bonds are longer, and Pb—O shorter, in the present structure than in 2-(morpholino)ethanethiolatolead(II) nitrate. As in this other Pb-thiolate structure, and in D-penicillaminatolead(II) (Freeman, Stevens & Taylor, 1974), the effect of a stereochemically active non-bonding electron pair on the Pb atom in $Pb(SCH_2CH_2OH)(NO_3)$ is marked: its angular location is approximately in the direction of the next-nearest S atom (Fig. 2).

The tendency of lead–thiolate complexes to polymerize appears to be aided by the propensity of Pb^{II} to have an irregular coordination sphere involving several secondary but significant bonding interactions.

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Structure of *N*-(3-Cyanophenyl)acetohydroxamic Acid Hydrate, $C_9H_8N_2O_2.\frac{1}{3}H_2O$

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Abstract. $M_r = 182\cdot18$, orthorhombic, $Pna2_1$, $a = 25\cdot123$ (7), $b = 3\cdot947$ (1), $c = 26\cdot107$ (7) Å, $V = 2588\cdot8$ (12) Å^3 at 138 (2) K, $a = 25\cdot202$ (6), $b = 4\cdot0433$ (12), $c = 26\cdot268$ (10) Å, $V = 2676\cdot5$ (14) Å^3 at 295 K, $Z = 12$, $D_m = 1\cdot354$, $D_x = 1\cdot356$ g cm^{-3} at 295 K, Ni-filtered Cu $K\bar{\alpha}$, $\lambda = 1\cdot5418$ Å, $\mu = 7\cdot71$ cm^{-1}, $F(000) = 1144$, $T = 138$ (2) K, $R = 0\cdot045$ for 2149 observed data. The three unique hydroxamic acid molecules all have a *trans* conformation, have the cyano groups *anti* to the hydroxyl O atom, have consistent bond distances and angles, and are connected through a network of hydrogen bonds.

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