$[Rh_2(C_2H_3O_2)_4(C_{13}H_{10}N_2S)_2]$

N10—C15—C14—S5	−10 (2)	C13—C12—C11—N10	−48 (2)
C15—C14—S5—C13	−62 (1)	C12—C11—N10—C15	−3 (2)
C14—S5—C13—C12	67 (1)	C11—N10—C15—C14	60 (2)
S5—C13—C12—C11	−1 (2)		

Code de symétrie: (i) $\frac{1}{2} - x, \frac{1}{2} - y, 1 - z$.

Les intensités ont été corrigées des facteurs de Lorentz et de polarisation. La structure a été déterminée par méthodes directes avec *SHELXS86* (Sheldrick, 1985). L'affinement des paramètres x, y, z, β_{ij} pour les atomes non-H, basé sur les F, a été effectué à l'aide du programme *SHELX76* (Sheldrick, 1976). Les H ont été placés suivant une géométrie standard. Facteur de température U_{iso} global affiné pour les H des méthyls où U_{iso} global = 0,15 Å². Le dessin de la structure a été obtenu à l'aide du programme *MACORTEP* (Michalowicz & André, 1991). Les angles dièdres ont été calculés à l'aide du programme *BEST PLANES* (Ito & Sugawara, 1983).

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Metal–Hydroxy Ribbons in Pb(OH)(NCS)

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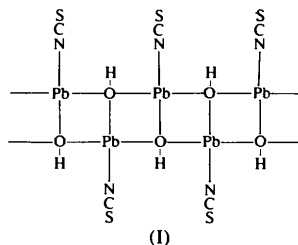
Abstract

The crystal structure of hydroxy(isothiocyanato)lead is orthorhombic *Pbnm*, with the asymmetric unit lying in a mirror plane. The Pb atom is surrounded by an N atom at 2.63 (1) Å and three O atoms, one at 2.372 (9) Å and two at 2.453 (5) Å, in a pyramidal arrangement. The interactions of Pb with O produce ribbons of double –O—Pb—O—Pb– chains running along the z axis.

Comment

During research concerning the interactions of lead(II) with sulfur-containing ligands, basic lead thiocyanate was obtained as crystals suitable for X-ray single-crystal structure analysis.

The NCS group of the title compound, (I), is coordinated through the N atom to Pb. Each Pb atom has one N atom, at 2.63 (1) Å, and three O atoms, one at 2.372 (9) Å and two at 2.453 (5) Å, in a pyramidal arrangement leaving room for the metal 'inert pair' (Fig. 2). The IR spectrum shows ν(CS) and δ(NCS) absorptions at 2071 and 448 cm^{-1}, respectively, in agreement with the terminal position of the group (Nakamoto, 1970), which is in the isothiocyanate form and collinear with the Pb—N bond. Each O atom has a tetrahedral environment involving one Pb atom at a distance of 2.372 (9) Å, two Pb atoms at 2.453 (5) Å and an H atom. The second coordination sphere of Pb involves two S atoms at 3.305 (4) Å and two at 3.490 (4) Å. Two O—H···N hydrogen-bonding interactions to N atoms at $-x,-y,z-\frac{1}{2}$ and $-x,-y,z+\frac{1}{2}$ have O···N distances of 2.97 (1) Å.

(I)

It is interesting to note that all the atoms of the SCN—Pb—OH moiety lie in a mirror plane perpendicular to the c direction at $z = \frac{1}{4}$. The interactions of Pb with O cause the formation of ribbons running along the z axis.

An analysis of thermal motion, carried out in terms of the Schomaker & Trueblood (1968) TLS rigid-body approximation using the *THMV* program (Trueblood,
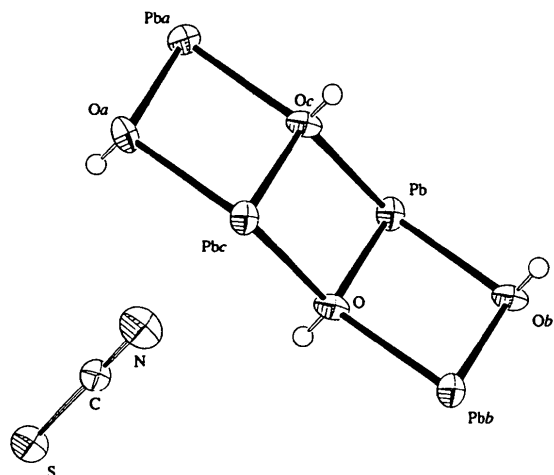
1984), gave 0.022 for the overall residual error index, R_{wU}, indicating that the assumed approximation is quite satisfactory.

Experimental

The compound was prepared according to Gmelin (1969). Crystals were obtained from an aqueous solution containing $Pb(NCS)_2$ and sodium acetate.

Fig. 1. *PLATON* (Spek, 1990) drawing of the asymmetric unit of Pb(OH)(NCS). Displacement ellipsoids are shown at the 50% probability level.
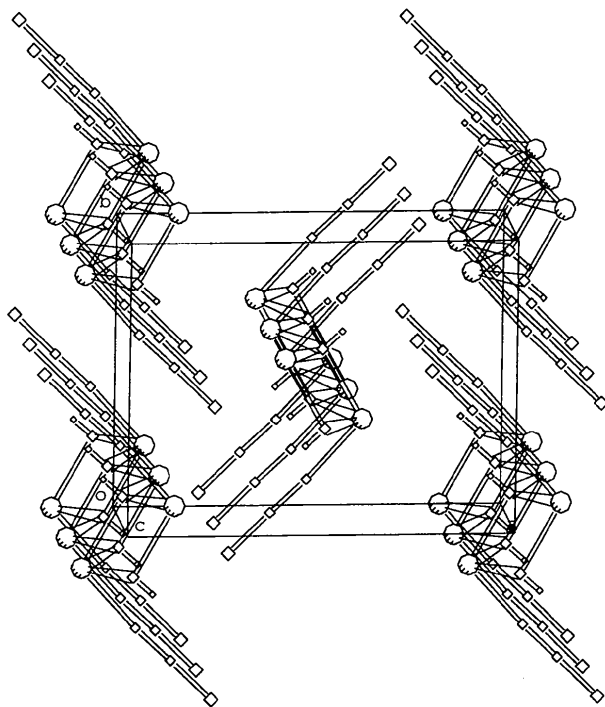
Crystal data

Pb(OH)(NCS)	Mo $K\alpha$ radiation
$M_r = 282.29$	$\lambda = 0.71069$ Å
Orthorhombic	Cell parameters from 29
Pbnm	reflections
$a = 11.250$ (7) Å	$\theta = 12.07-17.76°$
$b = 8.564$ (5) Å	$\mu = 42.44$ mm^{-1}
$c = 4.157$ (2) Å	$T = 293$ (2) K
$V = 400.5$ (4) Å^3	Slender needle
$Z = 4$	$0.16 \times 0.03 \times 0.03$ mm
$D_x = 4.682$ Mg m^{-3}	Colourless

Data collection

Siemens AED single-crystal	$R_{int} = 0.0386$
diffractometer	$\theta_{max} = 26.97°$
$\theta-2\theta$ scans	$h = -14 \rightarrow 14$
Absorption correction:	$k = -10 \rightarrow 10$
cylindrical	$l = 0 \rightarrow 5$
$T_{min} = 0.341$, $T_{max} =$	3 standard reflections
0.354	monitored every 50
1636 measured reflections	reflections
497 independent reflections	intensity decay: within
383 observed reflections	statistical fluctuation
$[I > 2\sigma(I)]$	

Refinement

Refinement on F^2	$\Delta\rho_{max} = 2.637$ e Å^{-3}
$R(F) = 0.0264$	$\Delta\rho_{min} = -2.660$ e Å^{-3}
$wR(F^2) = 0.0579$	Extinction correction:
$S = 1.171$	$F_c^* = kF_c[1 + (0.001x$
496 reflections	$\times F_c^2\lambda^3/\sin2\theta)]^{-1/4}$
35 parameters	Extinction coefficient:
All H-atom parameters	0.005 (1)
refined	Atomic scattering factors
$w = 1/[\sigma^2(F_o^2) + (0.0324P)^2]$	from *International Tables*
where $P = (F_o^2 + 2F_c^2)/3$	*for Crystallography* (1992,
$(\Delta/\sigma)_{max} < 0.001$	Vol. C, Tables 4.2.6.8 and
	6.1.1.4)

Fig. 2. *PLUTO* (Motherwell & Clegg, 1978) drawing of the packing in Pb(OH)(NCS).

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	0.10941 (4)	0.12978 (6)	1/4	0.0230 (2)
S	−0.2302 (3)	0.5839 (4)	1/4	0.0301 (11)
O	0.0055 (8)	−0.1113 (11)	1/4	0.023 (2)
N	−0.0528 (12)	0.3506 (15)	1/4	0.039 (3)
C	−0.1244 (11)	0.4477 (16)	1/4	0.024 (3)

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

Pb—O	2.372 (9)	S—C	1.667 (14)
Pb—O^i	2.453 (5)	C—N	1.16 (2)
Pb—N	2.629 (13)	O—H	0.76 (14)
N—Pb—O	106.5 (4)	Pb—O—Pb^i	108.4 (2)
N—Pb—O^i	71.4 (2)	Pb—O^i—Pb^i	108.4 (2)
O—Pb—O^i	71.6 (2)	Pb—N—C	179.9 (12)
O^i—Pb—O^{ii}	115.8 (4)	S—C—N	178.5 (13)

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

The Pb(OH)(NCS) crystals were described in the *Pbnm* non-standard setting of the *Pnma* (No. 62) space group; the transformation matrix is (001/100/010) and the coordinates of the equivalent positions are: x,y,z; $x+\frac{1}{2}, -y+\frac{1}{2}, -z$; $x-\frac{1}{2}, -y-\frac{1}{2}, z-\frac{1}{2}$; $x,y,-z-\frac{1}{2}$; together with those related by a centre of symmetry at 0,0,0. The integrated intensities were obtained by a modified version (Belletti, Ugozzoli, Cantoni & Pasquinelli, 1979) of the Lehmann & Larsen (1974) peak-profile analysis procedure. All reflections were corrected for Lorentz and polarization effects, absorption correction was for a cylindrical sample ($\mu R = 2.2$) and coefficients were taken from *International Tables for Crystallography* (1992, Vol. C, Table 6.3.3.2, p. 523).

The structure was solved by the Patterson method and refined by anisotropic full-matrix least squares on F^2 using *SHELXL93* (Sheldrick, 1993). Refinement on F^2 involved all reflections except for one probably affected by systematic errors. Two residual peaks of 2.68 and 2.35 e Å^{-3} were found in the final difference map at *ca* 1 Å from Pb.

Calculations were carried out on the ENCORE91 and GOULD-POWERNODE 6040 computers of the Centro di Studio per la Strutturistica Diffrattometrica del CNR, Parma, and on a COMPAQ 486c portable computer.

Data collection: local programs. Cell refinement: *LQPARM* (Nardelli & Mangia, 1984). Data reduction: local programs. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL93*. Molecular graphics: *PLATON* (Spek, 1990); *PLUTO* (Motherwell & Clegg, 1976). Software used to prepare material for publication: *PARST* (Nardelli, 1983); *PARSTCIF* (Nardelli, 1991).

Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the IUCr (Reference: HA1124). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Novel Structure of (L-Aspartato)(1,10-phenanthroline)copper(II) Hydrate

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Abstract

The title compound, [Cu(C$_4$H$_5$NO$_4$)(C$_{12}$H$_8$N$_2$)].3.5H$_2$O, consists of two types of different pentacoordinate copper complex: one monomeric, with the aspartate group acting as a tridentate ligand, and the other polymeric, with the aspartate group bridging two different copper centers. The whole structure is held together by a complex hydrogen-bonding scheme involving the water molecules of solvation.