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Thiourea Coordination Complexes of Pb(II) Salts. II. Octahedral Coordination in Tetrakis(thiourea)lead(II) Picrate

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Pb(tu)₄(picrate)₂ is monoclinic with $a = 39.51$ (8), $b = 4.74$ (5), $c = 20.13$ (8) Å, $\beta = 125$ (1)°, space group *Cc* or *C2/c* (latter established by successful structure analysis), 4 formula units per cell. The structure was solved by Patterson and Fourier methods and refined by full-matrix least-squares to a final conventional *R*-value of 12.4%. The crystal contains polymeric chains of edge-sharing distorted Pb(tu)₆ octahedra and stacks of picrate ions. Both the polymeric chains and the stacks of anions are arranged in infinite sheets parallel to (100), the crystal structure thus being made up of interleaved parallel cationic and anionic sheets with hydrogen bonds between them. The distances from Pb to shared S are 2.87 and 3.23 Å and the distance of Pb to unshared S is 2.96 Å. The analogous polymeric chains of edge-sharing octahedra found in Cd(tu)₂(HCOO)₂, Ni(tu)₂(NCS)₂ and Pb(tu)₂Cl₂ are more regular than the polymeric chains in Pb(tu)₄(picrate)₂. The overall packing is similar to that found in the equimolar molecular complex of hexabromobenzene and 1,2,4,5-tetrabromobenzene.

Introduction

The coordination complexes of Pb(II) salts with thiourea (tu) show a variety of compositions and types of coordination but no clear principles have yet emerged allowing one to connect composition and coordination type. Sixfold coordination of thiourea about lead has been found in triclinic Pb(ClO₄)₂·6tu (Goldberg & Herbstein, 1972), eightfold coordination in tetragonal Pb(ClO₄)₂·6tu (Boeyens & Herbstein, 1967) and sevenfold (mixed ligand) coordination in PbCl₂·2tu (Nardelli & Fava, 1959). Although some PbX₂·4tu complexes are known (Boeyens & Herbstein, 1967) on structures have yet been reported. We have determined the crystal structure of tetrakis(thiourea)lead(II) picrate (Yatsimirsky & Astasheva, 1952) and find that there is octahedral coordination of sulphur about lead, the octahedra being arranged in infinite polymeric chains and joined by shared edges. Preliminary reports have been published (Goldberg, Herbstein & Kaftory, 1968; Herbstein & Kaftory, 1969). It would be premature to generalize from this result as the cell dimensions and space groups of the PbX₂·4tu complexes investigated so far do not show any marked similarities.

Experimental

Yellow needles, elongated along [010], were obtained by slow cooling of an aqueous solution containing thiourea and lead(II) picrate.

Crystal data (from oscillation and Weissenberg photographs)

Pb(C₆H₂N₃O₇)₂ [SC(NH₂)₂]₄,
m.p. 159° (decomp.), $M = 963.6$, $\mu = 134$ cm⁻¹ (for Cu $K\alpha$),
monoclinic, $a = 39.51$ (8), $b = 4.74$ (5), $c = 20.13$ (8) Å,
 $\beta = 125$ (1)°,
 $U = 2918$ Å³,

$D_m = 2.09$ g.cm⁻³, $D_x = 2.07$ g.cm⁻³ for $Z = 4$.

Reflexion conditions hkl for $h+k=2n$, $h0l$ for $l=2n$ ($h=2n$), $0k0$ for ($k=2n$).

Possible space groups: *Cc* (No. 9) or *C2/c* (No. 14) (*C2/c* was confirmed by the structure analysis).

Intensities of 1842 $h0l$, $h1l$ and $h2l$ independent reflexions were measured by the visual method from equi-inclination Weissenberg photographs (Cu $K\alpha$). Geometrical corrections were applied by standard programs; an absorption correction was not made ($\mu R = 1.07$ for the crystal used).

Computing facilities used and other details are described in part I (Goldberg & Herbstein, 1972).

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Determination and refinement of structure

As a working hypothesis it was assumed that the crystals were centrosymmetric. Accordingly the lead atoms must be at Wyckoff positions (*a*) (000 *etc.*) or (*e*) ($0y\frac{1}{2}$ *etc.*) of space group *C2/c*. Reflexions with $k=4n+2$, $l=2n$ and $k=4n$, $l=2n+1$ were noticeably weaker than the other reflexions; this suggests that the lead atoms are at positions (*e*), with $y_{\text{Pb}} \approx \frac{1}{2}$. This was confirmed by a three-dimensional Patterson synthesis, which also gave directly the coordinates of most of the other atoms in the asymmetric unit. The positions of the remaining atoms were obtained from a three-dimensional Fourier synthesis. Refinement was effected by least-squares, using anisotropic temperature factors for Pb and S atoms, and isotropic temperature factors for the other atoms. Hydrogen atoms were not included at any stage. The atomic scattering factors and dispersion corrections (real part only) were taken from standard sources (*International Tables for X-ray Crystallography*, 1962).

Refinement was stopped with the conventional *R*

value equal to 12.4%, at which stage convergence appeared to have been attained. Final atomic parameters are given in Table 1. Observed and calculated structure factors can be obtained on request.*

The estimated standard deviations of the atomic coordinates have the following values: Pb: $\sigma(y) \approx 0.0015 \text{ \AA}$, S: $\sigma(x) \approx \sigma(z) \approx 0.005$, $\sigma(y) \approx 0.011 \text{ \AA}$; O, N, C: $\sigma(x) \approx \sigma(z) \approx 0.025$, $\sigma(y) \approx 0.02-0.04 \text{ \AA}$. The larger values of $\sigma(y)$ are a consequence of the anisotropic distribution of measured reflexions in reciprocal space. Although *h3l* and *h4l* layers are accessible to *Cu K α* , intensities of these reflexions were not measured as it was felt that only a marginal improvement of accuracy would be obtained by their inclusion. The temperature factors are of limited accuracy and no attempt has been

* Table 2 has been deposited as Document NAPS 01655 with the National Auxiliary Publications Service, CCM Information Corporation, 867 Third Avenue, New York, N.Y. 10022, U.S.A. A copy may be secured by citing the Document No. and remitting \$5.00 for photocopy or \$2.00 for microfiche copy. Advance payment is required. Make checks or money orders payable to: CCMIC-NAPS.

Table 1. Fractional atomic coordinates, temperature factors and their estimated standard deviations

	Isotropic temperature factor $T = \exp(-8\pi^2 U \sin^2 \theta / \lambda^2)$			
	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> (Å ²)
Pb	0	-0.1326 (3)	$\frac{1}{2}$	†
Thiourea molecules				
S(1)	0.0588 (1)	0.318 (2)	0.3090 (3)	†
C(1)	0.0907 (6)	0.236 (9)	0.4116 (11)	0.034 (4)
N(1)	0.1185 (6)	0.025 (8)	0.4369 (11)	0.046 (5)
N(2)	0.0899 (6)	0.357 (5)	0.4628 (13)	0.039 (5)
S(2)	0.0113 (1)	-0.126 (1)	0.4087 (3)	†
C(8)	-0.0359 (6)	-0.276 (9)	0.3707 (12)	0.037 (4)
N(6)	-0.0707 (6)	-0.125 (5)	0.3110 (13)	0.038 (5)
N(7)	-0.0404 (6)	-0.512 (8)	0.4032 (11)	0.045 (4)
Picrate ion				
C(2)	0.2319 (6)	0.348 (6)	0.3853 (12)	0.029 (4)
C(3)	0.2435 (6)	0.138 (5)	0.3587 (12)	0.027 (4)
C(4)	0.2178 (5)	0.057 (7)	0.2789 (10)	0.026 (4)
C(7)	0.1782 (6)	0.155 (5)	0.2287 (12)	0.026 (4)
C(5)	0.1621 (6)	0.368 (5)	0.2525 (12)	0.026 (4)
C(6)	0.1934 (6)	0.446 (7)	0.3368 (12)	0.033 (4)
O(4)	0.1277 (5)	0.470 (7)	0.2079 (10)	0.053 (4)
Nitro groups				
N(3)	0.1802 (5)	0.674 (5)	0.3734 (10)	0.034 (4)
O(1)	0.1447 (6)	0.742 (8)	0.3416 (13)	0.069 (5)
O(2)	0.2077 (6)	0.790 (7)	0.4312 (11)	0.055 (5)
N(4)	0.2840 (5)	0.017 (8)	0.4132 (10)	0.036 (4)
O(3)	0.3076 (5)	0.105 (4)	0.4797 (11)	0.041 (4)
O(6)	0.2930 (5)	-0.215 (6)	0.3871 (10)	0.050 (4)
N(5)	0.1523 (5)	0.030 (7)	0.1468 (10)	0.035 (4)
O(5)	0.1210 (7)	0.136 (5)	0.0951 (14)	0.060 (6)
O(7)	0.1656 (6)	-0.164 (5)	0.1314 (12)	0.049 (5)

† Anisotropic factors

$$T = \exp \left\{ -2\pi^2 \sum_{i=1}^3 \sum_{j=1}^3 U_{ij} h_i h_j a_i^* a_j^* \right\}$$

where U_{ij} are the coefficients of the (symmetric) atomic thermal vibration tensors and a_i^* are the reciprocal lattice parameters.

	U_{11}	U_{22}	U_{33}	U_{12}	U_{23}	U_{13}
Pb	0.0307 (7)	-0.008 (7)	0.0209 (7)	0	0	0.0035 (5)
S(1)	0.034 (2)	0.03 (1)	0.020 (2)	-0.00 (0)	0.00 (0)	0.01 (0)
S(2)	0.020 (2)	0.01 (1)	0.038 (3)	-0.00 (0)	0.00 (0)	0.01 (0)

made to analyse the thermal motion of the ions in the crystal; the negative value of U_{22} for Pb is probably also due to the limitation placed on k .

Description of the structure

The structure is shown in projection on (010) in Fig. 1. The lead ions are in chains along [010], and thus extend normal to the projection shown in Fig. 1. The lead ions are 4.74 Å apart and are surrounded by distorted octahedra of sulphur atoms of thiourea molecules. These octahedra share edges (Fig. 2). The parallel lead–thiourea chains form sheets which lie in the (100) planes and are separated by $\frac{1}{2}d_{100}$ (=16.18 Å). The intervening space is occupied by staggered stacks of picrate ions, whose ring planes make angles of $\pm 46^\circ$ with (010); the mean perpendicular distance between ring planes in any stack is 3.4 Å.

The shorter distances between atoms in different groups are indicated in Fig. 1. The lead–thiourea chains are separated by normal van der Waals distances. There are hydrogen bonds between picrate ions and thiourea molecules and the picrate ions are oriented with their negatively charged oxygen atoms pointed towards the amino groups that constitute the surface of the lead–thiourea chains.

The structure can thus be described as being made up of parallel positive and negative sheets of material lying in the (100) planes. The lead–thiourea chains

within the positive sheets, and the picrate ions in the stacks which make up the negative sheets, are bonded by van der Waals forces. The sheets as a whole presumably interact both ionically and through hydrogen bonds.

The geometrical features of the polymeric chains of edge-sharing octahedra and the dispositions of the thiourea molecules about a lead atom are shown in Fig. 2. The chain of composition $[\text{Pb}(\text{tu})_4]^{2+}$ has a twofold axis of symmetry along its length and is polar. The crystal as a whole is non-polar because the individual polar chains are related by centres of symmetry (the individual anions are also polar but are related by centres of symmetry). The distances between lead atoms and shared sulphur atoms have alternating values of 2.87 and 3.23 Å; the S–Pb–S internal angles also alternate between 83.6 and 72.4°. However within the chains the Pb···Pb and S···S distances (shared sulphur atoms) are constant at 4.74 and 3.82 Å respectively. The distance lead–unshared sulphur is 2.96 Å. The two shorter Pb–S contacts lie in the range usually ascribed to ionic interactions, whereas the longer distance of 3.23 Å approaches that ascribed to van der Waals interactions (3.4 Å).

Discussion

One can search for structural analogies either *via* similarities in structural formula, or through known

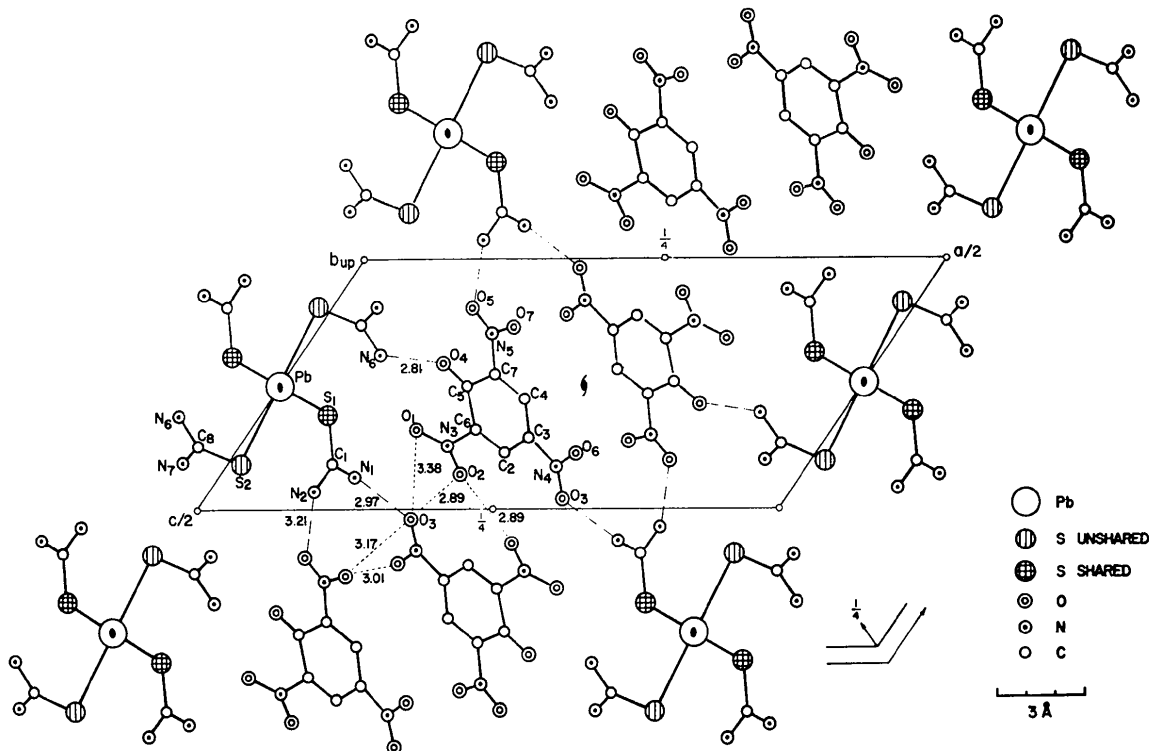


Fig. 1. The structure projected onto (010). The hydrogen bonds are shown by dashed lines and other shorter distances by dotted lines.

similarities in structural arrangement. Four different complexes of formula $Pb(tu)_4X_2$ have been reported:

$X \equiv Cl$: Boeyens & Herbststein (1967); Nardelli, Cavalca & Braibanti (1956);

$X \equiv NCS$ (thiocyanate): Boeyens & Herbststein (1967). (The complex was incorrectly described as an isothiocyanate in this reference);

$X \equiv$ picrate: present paper.

However only the structure of the latter is known.

There are a number of complexes of formula $M^{II}(L)_4X_2$ whose structures have been reported. Discrete octahedral molecules, connected by hydrogen bonds, are found in $Ni(tu)_4Cl_2$ (Lopez-Castro & Truter, 1963), in $Co(tu)_4Cl_2$ and the isomorphous $Mn(II)$, $Fe(II)$ and Cd complexes (O'Connor & Amma, 1969), in $Ni(tmu)_4Cl_2$ (Luth & Truter, 1968; tmu = trimethylenethiourea). Discrete octahedral molecules, without definite evidence of hydrogen bonding, are found in $Ni(etu)_4Cl_2$ (Robinson, Holt & Carpenter, 1967; triclinic and monoclinic polymorphs; etu = ethylenethiourea), in $Ni(pz)_4Cl_2$ (Reimann, Mighell & Maurer, 1967) and $Ni(pz)_4Br_2$ (Mighell, Reimann & Santoro, 1969) (pz = pyrazole), in $Ni(py)_4Cl_2$ and $Co(py)_4Cl_2$ (Porai-Koshits, 1954) (py = pyridine) and in $Ni(py)_4Br_2$ and $Co(py)_4Br_2$ (Antsishkina & Porai-Koshits, 1958). An ionic structure is found in $Pd(tu)_4Cl_2$ with square planar $[Pd(tu)_4]^{2+}$ cations and Cl^- anions (Berta, Spofford, Boldrini & Amma, 1970).

The sought-after resemblance must therefore come from similarities in some or all aspects of appropriate crystal structures (apart from the as yet unknown $Pb(II)$ complexes listed above). Polymeric chains of edge-sharing octahedra (shared sulphur atoms) are found in a number of coordination complexes of formula $M^{2+}L_2X_2$; three examples where sulphur atoms of thiourea groups are shared are known. In these three examples the polymeric chains are formally neutral whereas the $[Pb(tu)_4]^{2+}$ chain is cationic; the consequences for the overall crystal structures are discussed below. The chain in $Ni(tu)_2(NCS)_2$ (Nardelli, Fava Gasparri, Giraldi Battistini & Domiano, 1966; (see especially Fig. 1 of this paper); Domiano, Gaetani Manfredotti, Grossoni, Nardelli & Vidoni Tani, 1969) has centres of symmetry at the Ni atoms, while those in $Pb(tu)_4(picrate)_2$ and $Cd(tu)_2(HCOO)_2$ (Nardelli, Fava Gasparri & Boldrini, 1965; (see especially Fig. 2 of this paper) have twofold axes running along their lengths through the metal atoms. The chain in $Pb(tu)_2Cl_2$ (Nardelli & Fava, 1959 [see especially Fig. 3 of this paper]) does not have any symmetry. The two chains with twofold axes and the one without symmetry will be chiral. Both enantiomers will be found in $Pb(tu)_4(picrate)_2$ (space group $C2/c$), $Ni(tu)_2(NCS)_2$ (space group $P\bar{1}$) and $Pb(tu)_2Cl_2$ (space group $Pna2_1$) but the crystals of $Cd(tu)_2(HCOO)_2$ (space group $P2_12_12$) will contain only one enantiomer and it should be possible, at least in principle, to determine its absolute configuration. The alternation of $Pb-S$

distances within the polymeric chains of the present compound may be due to the stereochemical influence of the $Pb 6s^2$ lone pair (Orgel, 1959). [We add that polymeric chains of edge-sharing octahedra are also found in $Cd[OC(NH_2)_2]_2Cl_2$ (Nardelli, Cavalca & Fava, 1957). Here the chlorines act as bridging atoms. As the

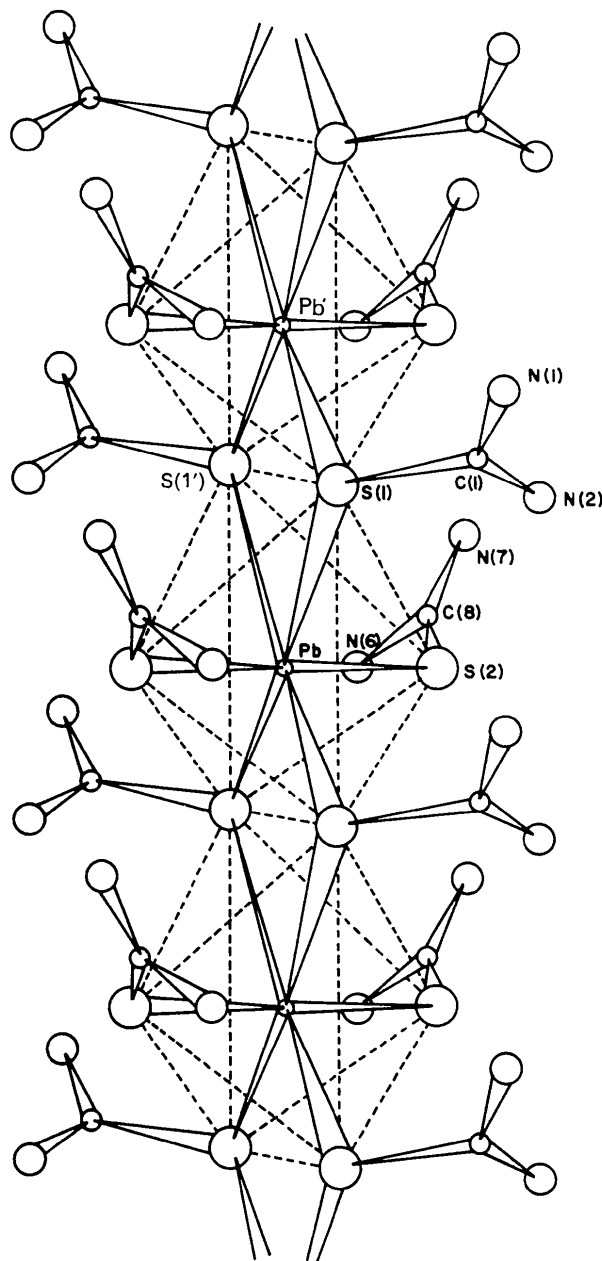


Fig. 2. Clinographic projection of the polymeric chain of edge-sharing octahedra in $Pb(tu)_4(picrate)_2$.

Some important interatomic distances and angles are:

$Pb-S(2)$	2.96 Å	$S(1')-Pb'-S(1)$	83.6
$Pb-S(1)$	3.23	$S(1')-Pb-S(1)$	72.4
$Pb'-S(1)$	2.87	$S(2)-Pb-Pb'$	90.7
$Pb-Pb'$	4.74	$S(1)-Pb-S(2)$	87.7
$S(1)-S(1')$	3.82	$Pb \cdots S(1)-Pb'$	102.0

space group was not established beyond doubt, it is not possible to usefully discuss the symmetry of the polymer chains].

The dimensions found here for the picrate ion are similar to those reported by Maartmann-Moe (1969) from (more accurate) studies of ammonium and potassium picrates. Detailed results are not reproduced but it is worth noting that the internal angle at the carbon atom to which oxygen is bonded is reduced to $109(3)^\circ$ while the internal angles at the adjacent carbon atoms in the ring are increased to $123(4)$ and $128(3)^\circ$. Similar changes from 120° are found in ammonium and potassium picrates. Dimensions have been given for the picric acid molecule in the π -molecular compound 1-bromo-2-naphthylamine:picric acid (Carstensen-Oeser, Göttlicher & Habermehl, 1968): the internal angle at the carbon to which the hydroxyl is bonded was found to be 119° , appreciably different from the value in the picrate ion. There is some disorder in the positions of the molecules of the molecular compound so confirmation of this difference from an independent source is desirable. The picrate ion is planar except for the nitro groups; the atoms C(2)–C(7), N(3)–N(5), O(4) do not deviate more than 0.06 \AA from the plane

$$0.791X + 0.698Y - 0.759Z = 2.210$$

(X, Y, Z measured in Å along the *crystal axes*). These deviations are not statistically significant. However two of the nitro groups are twisted about the C–N bonds out of the mean-plane (the torsion angles, defined according to Klyne & Prelog (1960), are O(2)–N(3)–O(4): -20° ; O(5)–N(5)–O(7): $+10^\circ$). The third nitro group lies in the plane given above.

In $\text{Cd}(\text{tu})_2(\text{HCOO})_2$ and $\text{Pb}(\text{tu})_2\text{Cl}_2$ hydrogen-bond acceptor groups are part of the chain structure and thus intrachain hydrogen bonding is important in determining the internal geometry of the chains, and interchain hydrogen bonding is important in determining the crystal structure as a whole. Hydrogen bonding does not appear to play any role in $\text{Ni}(\text{tu})_2(\text{NCS})_2$ nor does it appear to affect the structure of the chains in $\text{Pb}(\text{tu})_4(\text{picrate})_2$. However it does appear to play an important part in binding together the cationic and anionic sheets in $\text{Pb}(\text{tu})_4(\text{picrate})_2$. Despite the importance of the hydrogen bonding in $\text{Pb}(\text{tu})_4(\text{picrate})_2$ the most similar structure in overall type that we have found is that of the equimolar molecular complex of hexabromobenzene and 1,2,4,5-tetrabromobenzene (Gafner & Herbstein, 1964). Here the two component molecules are segregated into stacks, with only a small angle between the normals to the molecular planes and the stack axes. The hexabromobenzene stacks are arranged in sheets one molecule broad but of infinite lateral extent in two dimensions; these sheets are interleaved by similar sheets made up of stacks of 1,2,4,5-tetrabromobenzene molecules. Dispersion forces presumably make the major contribution both to the bonding between similar molecules within the sheets, and between the sheets as a whole.

An intriguing question that remains to be answered is why $\text{Pb}(\text{tu})_4(\text{picrate})_2$ forms an ionic polymeric structure while the other ML_4X_2 crystals have molecular structures with octahedral molecules [$\text{Pd}(\text{tu})_4\text{Cl}_2$ is the only exception to this generalisation that we have encountered]. One possible explanation is the bulkiness of the picrate ion; another is the reluctance of lead to form covalent Pb–O bonds.

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