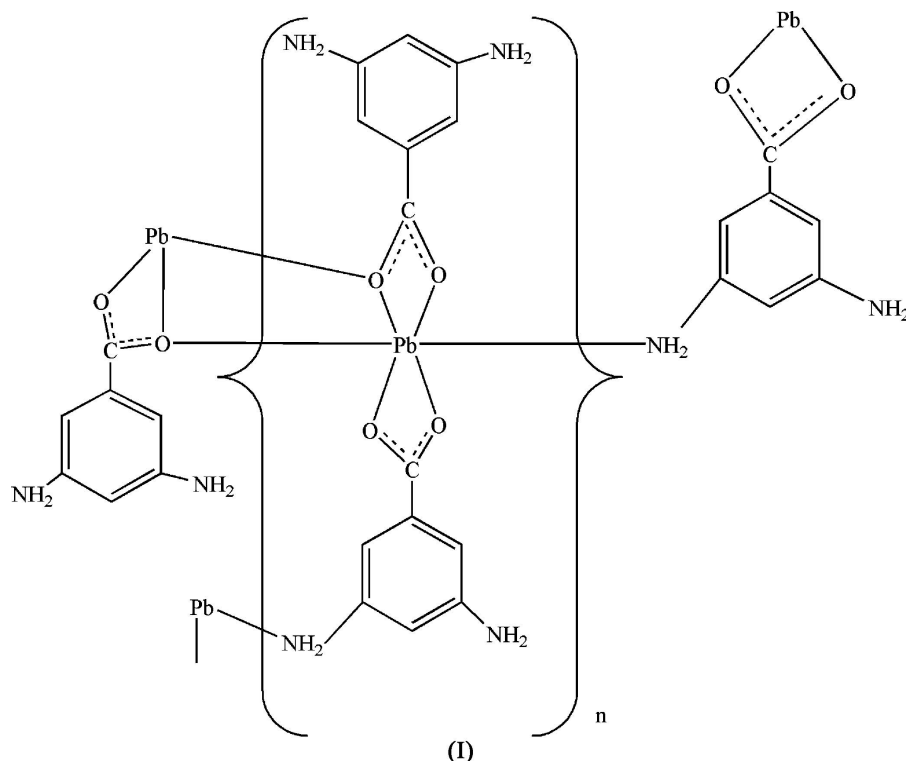


An-Zhi Tan,[‡] You-Huan Wei,[§]
Fu-Pei Liang,* Zi-Lu Chen and
Rui-Xiang HuCollege of Chemistry and Chemical Engineering,
Guangxi Normal University, Guilin 541004,
People's Republic of China[‡] Permanent address: Department of Chemistry
and Biology, Yulin Normal College, Yulin,
Guangxi 537000, People's Republic of China.[§] Permanent address: Department of Chemistry
and Life Science, Nanning Teachers College,
Longzhou, Guangxi 532400, People's Republic
of China.Correspondence e-mail:
fliangoffice@yahoo.com**Key indicators**Single-crystal X-ray study
 $T = 294$ K
Mean $\sigma(\text{C}-\text{C}) = 0.009$ Å
 R factor = 0.036
 wR factor = 0.086
Data-to-parameter ratio = 14.7For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.**Poly[bis(μ_2 -3,5-diaminobenzoato)- $\kappa^3\text{N}:\text{O},\text{O}'$;-
 $\kappa^3\text{O}:\text{O},\text{O}'$ -lead(II)]**

The asymmetric unit of the title compound, $[\text{Pb}(\text{C}_7\text{H}_7\text{N}_2\text{O}_2)_2]_n$, consists of a Pb^{II} ion and two 3,5-diaminobenzoate anions. The Pb^{II} ion is coordinated by one N and five O atoms from four different 3,5-diaminobenzoate ligands in a hemi-directed polyhedral geometry, with a $\text{Pb}-\text{N}$ distance of 2.625 (5) Å and $\text{Pb}-\text{O}$ distances in the range 2.354–2.719 Å. The μ_2 -bridging 3,5-diaminobenzoate anions connect the Pb^{II} ions into a two-dimensional layer. These two-dimensional layers are linked further by $\text{N}-\text{H}\cdots\text{N}$ and $\text{N}-\text{H}\cdots\text{O}$ intermolecular hydrogen-bond interactions, forming an extensive three-dimensional network.

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The coordination chemistry of lead has been of general interest for decades owing to its wide applications in technology, its widespread occurrence in the environment, and the interesting structural features of its complexes (Parr, 1997). Many lead complexes of carboxylic acids with various structures have been reported (Foreman *et al.*, 2001). We describe here the structure of a lead coordination polymer with 3,5-diaminobenzoate as a bridging ligand, (I).



The coordination environment of Pb is shown in Fig. 1. The Pb atom is coordinated by five O atoms and one N atom that

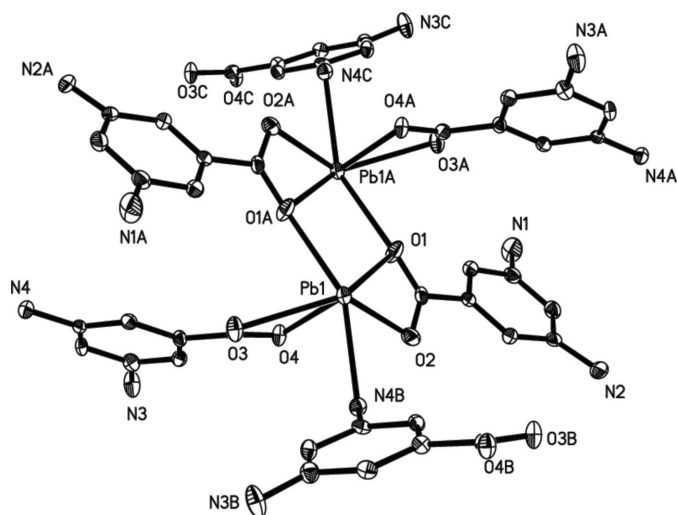


Figure 1

A view of part of the polymeric structure of (I). Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity. Atoms labeled with the suffixes A–C are at the symmetry positions $(-x, 1 - y, 2 - z)$, $(\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z)$ and $(x - \frac{1}{2}, -y + \frac{1}{2}, z - \frac{1}{2})$, respectively.

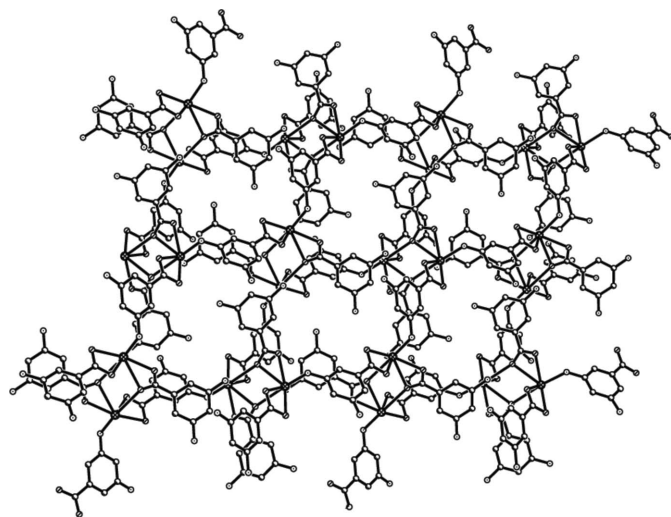


Figure 2

A view of the two-dimensional network in the *bc* plane. All H atoms have been omitted for clarity.

belong to four different 3,5-diaminobenzoate ligands. The average Pb–O bond length is 2.570 Å, which is very close to the sum of the ionic radii of Pb²⁺ and O²⁻ (2.59 Å; Shannon, 1976) and is comparable to those found for Pb^{II}–carboxylate compounds with similar coordination modes (Lacouture *et al.*, 2001; Foreman *et al.*, 2001). The Pb–N distance of 2.625 (5) Å is slightly longer than that found in the polymeric Pb–*p*-aminobenzoate complex (2.605 Å; Amiraslanov *et al.*, 1980). The six coordination bonds in (I) are all within a hemisphere centered at the Pb atom, leaving a distinct void in the coordination sphere around the metal center. The presence of this void, together with the fact that there exists a shortened distance between Pb and O4 relative to the other Pb–O

distances, indicates the presence of a stereochemically active lone pair of electrons on the Pb atom (Shimoni-Livny *et al.*, 1998).

The 3,5-diaminobenzoate anions act in two different bridging modes. In one bridging mode the carboxylate group of the ligand chelates to one Pb atom and bridges a symmetry-related Pb centre *via* one of its O atoms, linking two symmetry-related Pb atoms in this manner into a four-membered Pb₂O₂ ring. In the other bridging mode the carboxylate coordinates to a Pb atom in a chelating fashion and one of the amino groups of the ligand connects to a Pb atom of the adjacent four-membered Pb₂O₂ ring. These connections link the Pb₂O₂ units into a two-dimensional layer in the *bc* plane (Fig. 2).

The uncoordinated amino N atoms (N1, N2 and N3) and the coordinated amino N atom (N4), as well as the coordinated carboxylate O atoms of the 3,5-diaminobenzoates, are involved in hydrogen bonding. The two-dimensional layers of (I) are linked by the hydrogen bonds into a three-dimensional network. The geometry of the hydrogen bonding is given in Table 2.

Experimental

A mixture of 3,5-diaminobenzoic acid (0.15 g, 1 mmol), Pb(CH₃COO)₂·3H₂O (0.19 g, 0.5 mmol), NaOH (0.04 g, 1 mmol) and water (14 ml) in a Teflon-lined stainless steel Parr vessel (25 ml) autoclave was heated at 413 K in an oven for 4 d. The autoclave was then removed from the oven and allowed to cool naturally to room temperature. The resulting mixture was filtered, and the filtrate was allowed to stand at room temperature to allow the solvent to evaporate. Brown block-shaped crystals of (I) were obtained in 38% yield after 20 h. Analysis calculated for C₁₄H₁₄N₄O₄Pb: C 33.01, H 2.77, N 10.99%; found: C 33.12, H 2.75, N 11.08%.

Crystal data

[Pb(C₇H₇N₂O₂)₂]
M_r = 509.48
 Monoclinic, *P*2₁/*n*
a = 8.7573 (12) Å
b = 11.9212 (16) Å
c = 14.758 (2) Å
 β = 102.227 (2)°
V = 1505.8 (4) Å³

Z = 4
D_x = 2.247 Mg m⁻³
 Mo K α radiation
 μ = 11.23 mm⁻¹
T = 294 (2) K
 Block, brown
 0.18 × 0.14 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
T_{min} = 0.237, *T_{max}* = 0.346
 (expected range = 0.178–0.260)

8313 measured reflections
 3059 independent reflections
 2440 reflections with *I* > 2 σ (*I*)
R_{int} = 0.069
 θ_{\max} = 26.3°

Refinement

Refinement on *F*²
R[*F*² > 2 σ (*F*²)] = 0.036
wR(*F*²) = 0.086
S = 0.99
 3059 reflections
 208 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0433P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.002$
 $\Delta\rho_{\max} = 1.71 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -2.44 \text{ e } \text{Å}^{-3}$

Table 1

Selected geometric parameters (Å, °).

Pb1—O4	2.354 (4)	Pb1—O3	2.673 (5)
Pb1—O2	2.420 (5)	Pb1—O1 ⁱⁱ	2.686 (5)
Pb1—N4 ⁱ	2.625 (5)	Pb1—O1	2.719 (5)
O4—Pb1—O3	51.65 (14)	N4 ⁱ —Pb1—O1	119.44 (16)
O4—Pb1—O1	87.23 (17)	O3—Pb1—O1	133.34 (15)
O2—Pb1—O1	50.04 (15)	O1 ⁱⁱ —Pb1—O1	67.92 (16)

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

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1B...N3 ⁱⁱⁱ	0.90	2.37	3.241 (9)	163
N2—H2A...O4 ^{iv}	0.90	2.13	2.981 (7)	159
N3—H3B...O3 ^v	0.90	2.15	2.945 (8)	147
N4—H4A...O4 ^{vi}	0.90	2.45	3.207 (7)	142
N4—H4B...O2 ^{vi}	0.90	2.25	3.074 (7)	152

Symmetry codes: (iii) $x - \frac{1}{2}, -y + \frac{3}{2}, z + \frac{1}{2}$; (iv) $x + \frac{1}{2}, -y + \frac{3}{2}, z + \frac{1}{2}$; (v) $-x + \frac{1}{2}, y + \frac{1}{2}, -z + \frac{3}{2}$; (vi) $-x + \frac{1}{2}, y - \frac{1}{2}, -z + \frac{3}{2}$.

All H atoms were positioned geometrically and treated as riding atoms, with C—H distances of 0.93 Å and N—H distances of 0.90 Å, and with U_{iso} (H) values of $1.2U_{eq}$ (C,N). The highest peak and deepest hole are located 1.01 and 0.76 Å, respectively, from atom Pb1.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT*; data reduction: *SAINT* (Bruker, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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