

Crystal structures of three lead(II) acetate-bridged diaminobenzene coordination polymers

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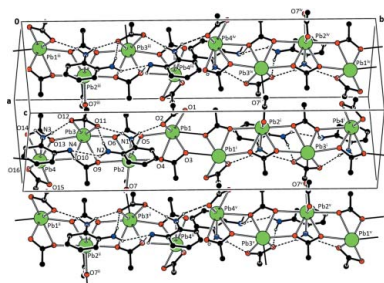
Keywords: crystal structure; coordination polymer; lead(II); hydrogen bonds; benzene-1,2-diamine; hemidirected coordination**CCDC references:** 1035089; 1035088; 1035087**Supporting information:** this article has supporting information at journals.iucr.org/e

Poly[tris(acetato- $\kappa^2 O, O'$)(μ_2 -acetato- $\kappa^3 O, O': O$)tetrakis(μ_3 -acetato- $\kappa^4 O, O': O: O'$)bis(benzene-1,2-diamine- κN)tetralead(II)], [Pb₄(CH₃COO)₈(C₆H₈N₂)₂]_n, (I), poly[(acetato- $\kappa^2 O, O'$)(μ_3 -acetato- $\kappa^4 O, O': O: O'$)(4-chlorobenzene-1,2-diamine- κN)lead(II)], [Pb(CH₃COO)₂(C₆H₇ClN₂)]_n, (II), and poly[($\kappa^2 O, O'$)(μ_3 -acetato- $\kappa^4 O, O': O: O'$)(3,4-diaminobenzonitrile- κN)lead(II)], [Pb(CH₃COO)₂(C₇H₇N₃)]_n, (III), have polymeric structures in which monomeric units are joined by bridging acetate ligands. All of the Pb^{II} ions exhibit hemidirected coordination. The repeating unit in (I) is composed of four Pb^{II} ions having O₆, O₆N, O₇ and O₆N coordination spheres, respectively, where N represents a monodentate benzene-1,2-diamine ligand and O acetate O atoms. Chains along [010] are joined by bridging acetate ligands to form planes parallel to (10 $\bar{1}$). (II) and (III) are isotopic and have one Pb^{II} ion in the asymmetric unit that has an O₆N coordination sphere. Pb₂O₂ units result from a symmetry-imposed inversion center. Polymeric chains parallel to [100] exhibit hydrogen bonding between the amine and acetate ligands. In (III), additional hydrogen bonds between cyano groups and non-coordinating amines join the chains by forming R₂²(14) rings.

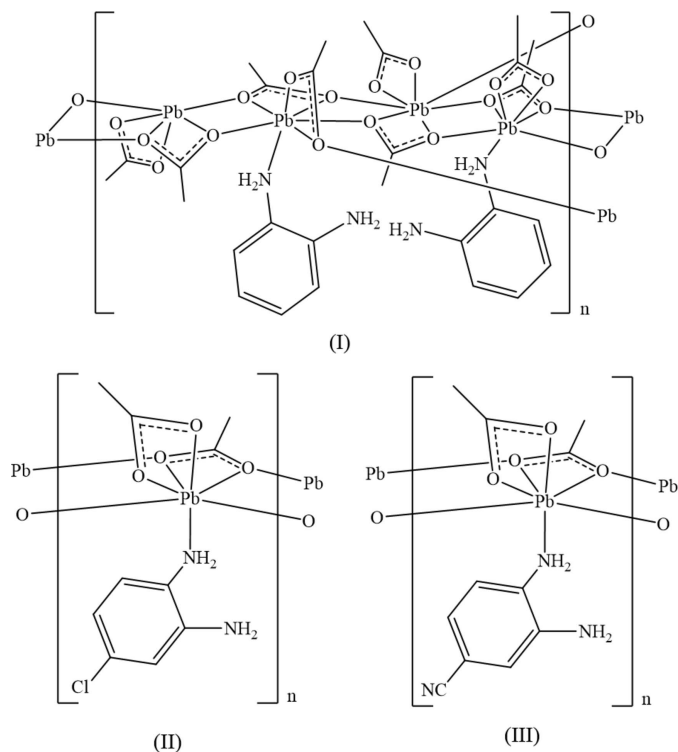
1. Chemical context

Metal–organic frameworks (MOFs) are of inherent interest in areas such as gas storage, catalysis, chemical sensors and molecular separation (Dey *et al.*, 2014; Kreno *et al.*, 2012; Farha & Hupp, 2010). Recently, we reported the synthesis and structural characterization of two zinc MOFs possessing bridging acetate ligands and monodentate chloro- or cyano-substituted *o*-phenylenediamine ligands (Geiger & Parsons, 2014). These complexes possess a ladder–chain structure with an ethanol molecule that occupies a void with a volume of approximately 224 Å³. The results presented here expand the structural study to Pb^{II} analogues.

Pb^{II} compounds often exhibit a distorted coordination sphere or open coordination site that has been attributed to stereoactive ‘lone-pair’ electrons (Morsali, 2004; Wang & Liebau, 2007; Park & Barbier, 2001). Indeed, hemidirected geometry is favored over halodirected geometry for Pb^{II} when hard ligands are present, which corresponds to a greater ionic character in the metal–ligand bonding (Shimoni-Livny *et al.*, 1998), or when one or more of the ligands is anionic (Esteban-Gómez *et al.*, 2006). However, hemidirected lead(II) complexes in a soft sulfur-rich environment are also known (Imran *et al.*, 2014). The results of a reduced variational space (RVS) analysis suggest that more sterically crowded, hemidirected structures are stabilized by polarization of the lead(II) ion induced by the ligand arrangement (Devereux *et al.*, 2011). The possibility of a distorted coordination sphere



enhancing the volume of void space between chains found in coordination polymers provided the impetus for the synthesis and structural characterization of the compounds reported herein.



2. Structural commentary

Fig. 1 shows the three acetate coordination modes displayed by (I), (II), and (III). The three modes will be referred to hereafter as types (a), (b) and (c). As seen in Fig. 2, the asymmetric unit of (I) has four symmetry-independent Pb atoms. The Pb atoms are linked by bridging acetate ligands of type (b) to form a ladder-chain parallel to [010]. Each is also coordinated to a bidentate acetate ligand of type (a) and Pb2 and Pb4 have an amine nitrogen in their coordination spheres. Finally, atoms Pb3 and Pb4 are linked by an acetato ligand of type (c). The two benzene-1,2-diamine ligands are approximately coplanar. The angle formed by the benzene mean planes is $6.1(4)^\circ$, with N1, N2, N3 and N4 being $0.051(16)$,

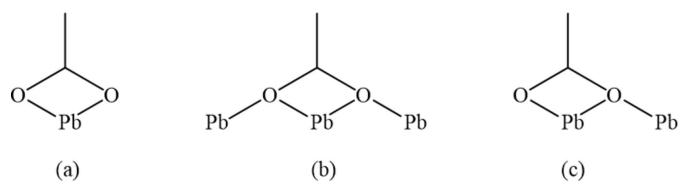


Figure 1
The three acetate coordination modes observed in (I), (II), and (III), showing (a) acetato- κ^2O,O' , (b) μ_3 -acetato- $\kappa^4O,O':O':O'$, and (c) μ_2 -acetato- $\kappa^3O,O':O$.

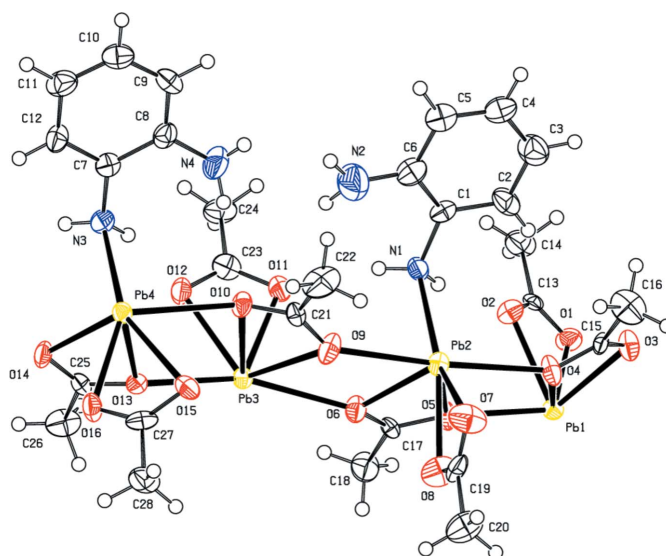


Figure 2
The atom-labeling scheme for (I). Anisotropic displacement parameters are drawn at the 50% probability level.

$0.013(19)$, $0.074(16)$, and $0.034(16)$ Å from their respective planes.

The asymmetric unit of (I) possesses pseudo-translational symmetry as a result of the similarity in the coordination geometries exhibited by Pb1 and Pb3 and by Pb2 and Pb4. $Pb1 \cdots Pb3 = 7.4548(10)$ Å and $Pb2 \cdots Pb4 = 7.5372(10)$ Å, approximately half of the $Pb1 \cdots Pb4^i = 14.989(2)$ Å distance (see Table 1 for symmetry codes). Fig. 3 shows a representation of (I) in which the two pseudo-translationally related halves of the asymmetric unit are color coded. Primary differences in the two halves involve the orientation of the two

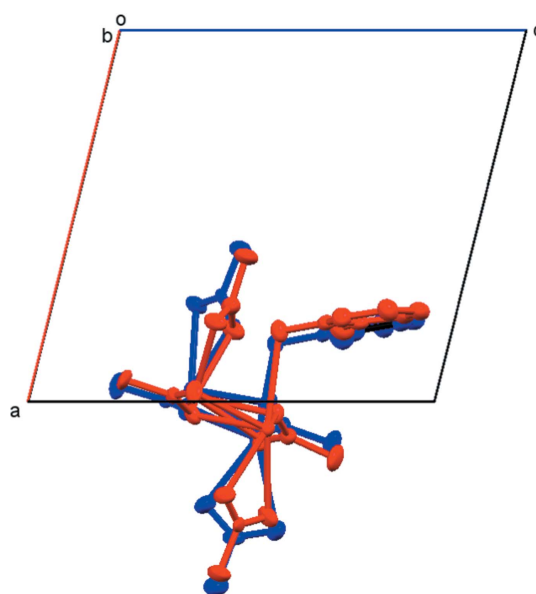


Figure 3
A view of (I) in which the two halves of the asymmetric unit related by the pseudo-translation are color coded. H atoms have been omitted for clarity.

Table 1
Selected bond lengths (Å) for (I).

Pb1—O2	2.380 (6)	Pb3—O12	2.509 (7)
Pb1—O3	2.474 (7)	Pb3—O10	2.590 (6)
Pb1—O4	2.576 (7)	Pb3—O9	2.604 (7)
Pb1—O1	2.636 (7)	Pb3—O7 ⁱⁱ	2.675 (7)
Pb1—O5	2.667 (6)	Pb3—O13	2.688 (6)
Pb1—O3 ⁱ	2.792 (7)	Pb3—O6	2.696 (7)
Pb2—O8	2.448 (8)	Pb4—O16	2.427 (7)
Pb2—O6	2.470 (7)	Pb4—O13	2.482 (7)
Pb2—O5	2.485 (6)	Pb4—O14	2.563 (7)
Pb2—O9	2.654 (7)	Pb4—O14 ⁱⁱⁱ	2.609 (7)
Pb2—O4	2.696 (6)	Pb4—O15	2.713 (7)
Pb2—O7	2.747 (8)	Pb4—O10	2.901 (6)
Pb2—N1	2.797 (9)	Pb4—N3	2.862 (10)
Pb3—O11	2.443 (7)		

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

Table 2
Selected bond lengths (Å) for (II).

Pb1—O1	2.467 (6)	Pb1—O2	2.678 (8)
Pb1—O3	2.504 (6)	Pb1—O3 ⁱⁱ	2.734 (6)
Pb1—O4	2.512 (6)	Pb1—N1	2.800 (8)
Pb1—O4 ⁱ	2.632 (6)		

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

Table 3
Selected bond lengths (Å) for (III).

Pb1—O3	2.431 (7)	Pb1—O4	2.667 (8)
Pb1—O2	2.485 (8)	Pb1—O1 ⁱⁱ	2.727 (7)
Pb1—O1	2.505 (7)	Pb1—N1	2.906 (10)
Pb1—O2 ⁱ	2.635 (7)		

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

non-coordinating amine groups, one less acetate type (*c*) on Pb1 than on Pb3, and a type (*c*) acetate ligand on Pb2 replaced by a type (*a*) acetate ligand on Pb4.

(II) and (III) are isotopic if the nitrile function in (III) is considered as a large one-atomic group and replaces the Cl atom in (II). Fig. 4 shows the atom-labeling scheme for (II) and Fig. 5 shows the atom-labeling scheme for (III). Each Pb atom has two bidentate acetate ligands, one of type (*a*) and one of type (*b*). The type (*b*) ligands result in chains parallel to [100], with Pb₂O₂ cores related by inversion centers. The substituted benzene-1,2-diamine ligands are essentially planar. For (II), N1 and N2 are below the plane by 0.056 (14) and 0.066 (18) Å, respectively, and Cl1 is 0.020 (14) Å above the plane. In (III), N1 and N2 are 0.073 (17) and 0.05 (2) Å out of the plane. The C7—N3—C4 angle is 177.7 (16)° and N3 is 0.12 (2) Å out of the plane.

The coordination spheres are O₆, O₆N, O₇, and O₆N for Pb1, Pb2, Pb3, and Pb4, respectively, for (I), and O₆N for (II) and (III). Representations of the coordination spheres are shown in Fig. 6 and pertinent bond distances are found in Tables 1, 2 and 3. The coordination is clearly hemidirected for each Pb and the Pb—O bond lengths are asymmetrical, as is often found for hemidirected compounds (Shimoni-Livny *et al.*, 1998). The average Pb—O bond lengths are 2.60 (13), 2.59 (11), and 2.58 (12) Å for (I), (II) and (III), respectively, or

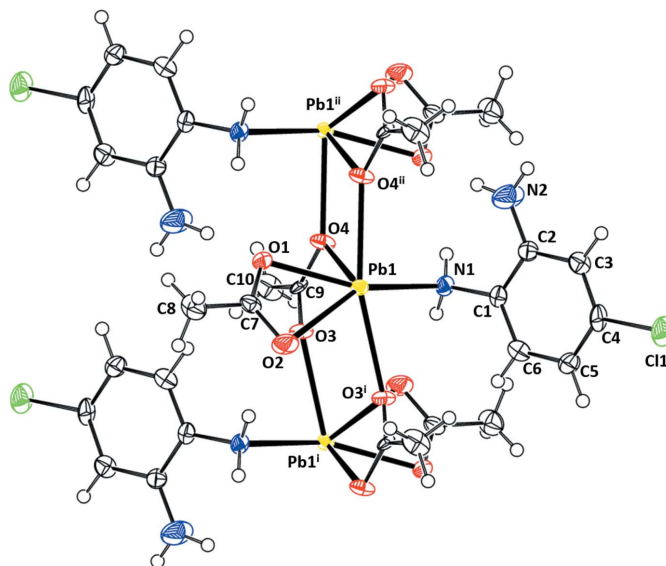


Figure 4
The atom-labeling scheme for (II). Anisotropic displacement parameters are drawn at the 50% probability level. [Symmetry identifiers: (i) $-x + 1, -y, -z + 1$; (ii) $-x + 2, -y, -z + 1$.]

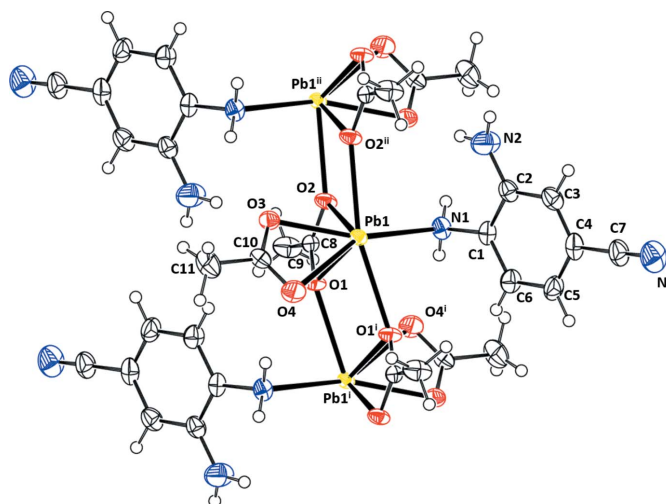


Figure 5
The atom-labeling scheme for (III). Anisotropic displacement parameters are drawn at the 50% probability level. [Symmetry identifiers: (i) $-x + 1, -y, -z + 1$; (ii) $-x + 2, -y, -z + 1$.]

2.59 (12) Å overall, and range from 2.380 (6) to 2.901 (6) Å. The average Pb—N bond length for the three compounds is 2.84 (5) Å. In all cases, the Pb—O(N) bond lengths are longer for those ligand atoms adjacent to the open coordination site. This is consistent with structural results for other hemidirected coordination modes involving O- and N-donor atoms (*cf.* Shimoni-Livny *et al.*, 1998; Morsali *et al.*, 2005; Esteban-Gómez *et al.*, 2006; Morsali, 2004).

3. Supramolecular features

The one-dimensional asymmetric unit chain of (I) propagates *via* inversion centers and is extended into two dimensions *via*

Table 4
Hydrogen-bond geometry (Å, °) for (I).

<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—H1A···O2	0.89 (2)	2.20 (3)	3.061 (11)	163 (7)
N1—H1B···O11	0.88 (2)	2.25 (4)	3.079 (11)	156 (8)
N2—H2A···O11	0.90 (2)	2.38 (5)	3.238 (13)	159 (11)
N2—H2B···N4	0.90 (2)	2.57 (5)	3.229 (14)	131 (5)
N3—H3A···O12	0.87 (2)	2.32 (4)	3.151 (11)	160 (8)
N3—H3B···O16 ⁱⁱⁱ	0.88 (2)	2.33 (4)	3.145 (11)	154 (7)
N4—H4B···O10	0.88 (2)	2.38 (4)	3.236 (12)	163 (10)

Symmetry code: (iii) $-x + 2, -y, -z + 1$.

an acetate ligand of type (*c*) that bridges Pb2 and Pb3ⁱⁱⁱ, as shown in Fig. 7, where the symmetry designators are defined. The result is an extended structure composed of planes parallel to (10 $\bar{1}$). N—H···O and N—H···N hydrogen bonding is observed along the chains parallel to [010] (see Table 4).

In compounds (II) and (III), chains parallel to [100] are observed. An extensive N—H···O hydrogen-bonding network is found along the chains (see Tables 5 and 6). For (III), the nitrile group affords the opportunity for additional hydrogen bonding. As seen in Fig. 8, this results in $R_2^2(14)$ rings involving N—H···N≡C hydrogen bonds between adjacent chains.

Based on calculations performed with *PLATON* (Spek, 2009), no solvent-accessible voids are found in (I), (II), or (III).

4. Database survey

Numerous examples of polymeric lead(II) compounds with bridging carboxylate ligands possessing a range of coordination modes have been reported (for examples, see Lyczko & Bak, 2008; Dai *et al.*, 2009; Mohammadnezhad *et al.*, 2010; Yilmaz *et al.*, 2003; Foreman *et al.*, 2001). A zinc metal organic framework with bridging acetate ligands and a monodentate 4-

Table 5
Hydrogen-bond geometry (Å, °) for (II).

<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—H1A···O2 ⁱⁱ	0.88 (2)	2.38 (3)	3.261 (11)	172 (9)
N1—H1B···O1 ⁱ	0.88 (2)	2.39 (5)	3.201 (10)	153 (8)
N2—H2A···O1 ⁱ	0.87 (2)	2.19 (6)	2.998 (11)	155 (13)

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

Table 6
Hydrogen-bond geometry (Å, °) for (III).

<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—H1A···O4 ⁱⁱ	0.88 (2)	2.46 (4)	3.310 (14)	164 (12)
N1—H1B···O3 ⁱ	0.87 (2)	2.40 (8)	3.139 (12)	143 (11)
N2—H2A···O3 ⁱ	0.88 (2)	2.25 (9)	3.044 (14)	150 (16)
N2—H2B···N3 ⁱⁱⁱ	0.88 (2)	2.62 (11)	3.355 (18)	142 (14)

Symmetry codes: (i) $-x + 2, -y + 1, -z + 1$; (ii) $-x + 1, -y + 1, -z + 1$; (iii) $-x + 1, -y + 2, -z + 2$.

chlorobenzene-1,2-diamine ligand has been reported (Geiger & Parsons, 2014).

5. Synthesis and crystallization

5.1. Preparation of (I)

Benzene-1,2-diamine (0.109 g, 0.93 mmol) was stirred into a solution of lead(II) acetate trihydrate (0.175 g, 0.46 mmol) in ethanol (10 ml). The solution was heated to a gentle reflux for 2 h and then cooled to room temperature. The solvent was reduced in volume by slow evaporation. After 5 d, crystals suitable for X-ray analysis had formed. Further solvent reduction resulted in precipitation of excess diamine, so the overall yield was not determined. Selected IR bands (diamond anvil, cm⁻¹): 3353 (*br*), 1505 (*s*), 1932 (*s*), 1284 (*s*) 1045 (*w*), 1018 (*w*), 939 (*w*).

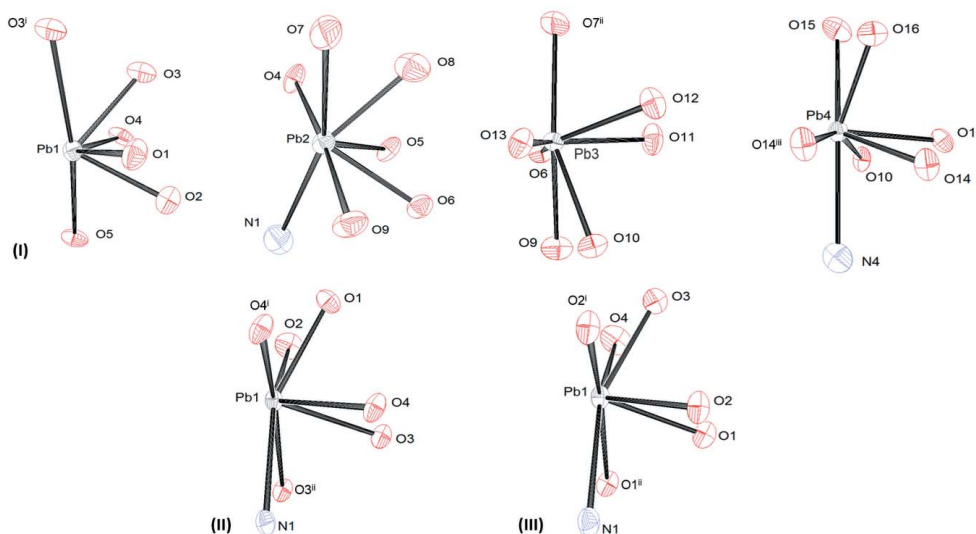
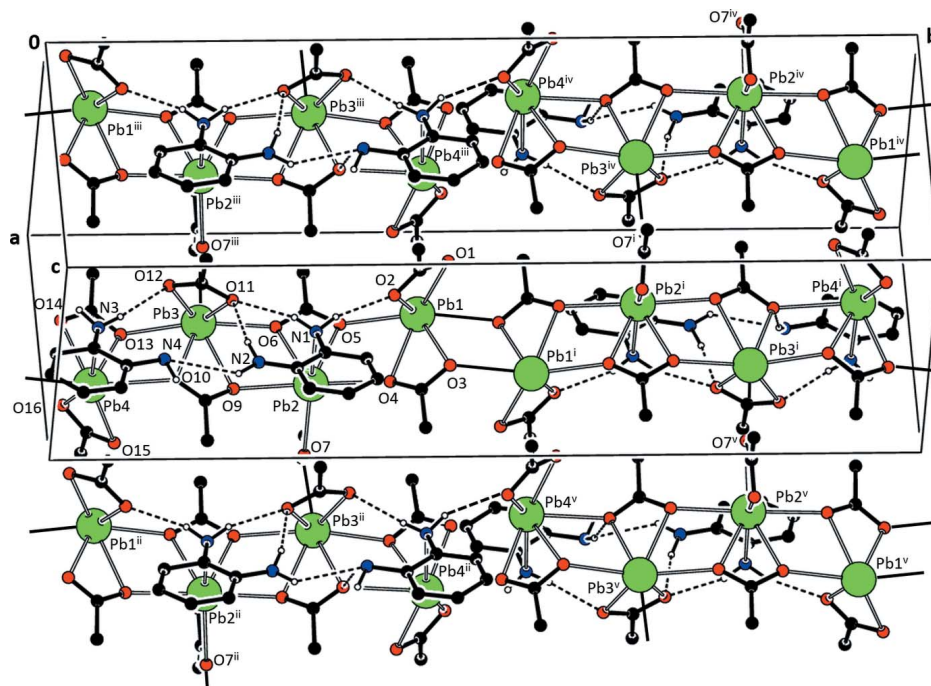
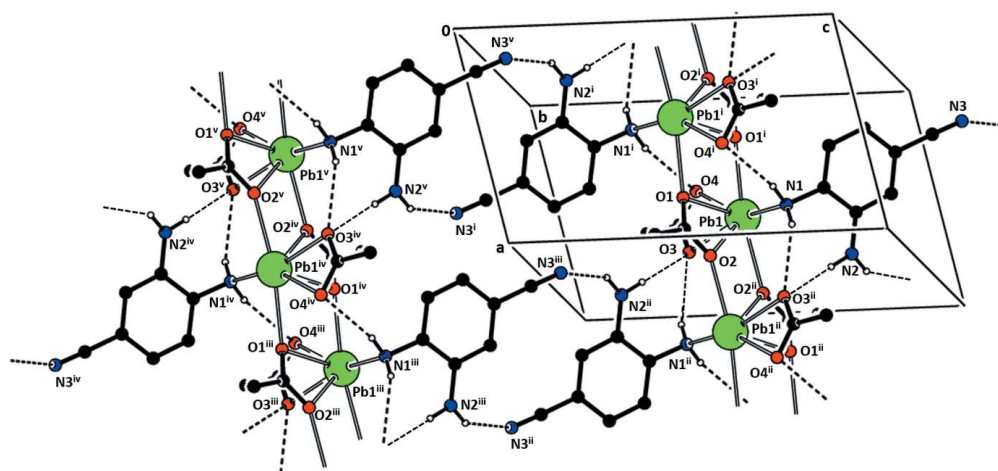


Figure 6
Representation of the Pb^{II} coordination environments observed in (I), (II), and (III). Symmetry identifiers are those used in Tables 1, 2 and 3.


Figure 7

Packing diagram for (I), showing the linked chains. Hydrogen bonds are represented by dashed lines. H atoms not involved in the hydrogen-bonding network are not shown. [Symmetry identifiers: (i) $-x + 2, -y + 1, -z + 1$; (ii) $x + \frac{1}{2}, y + \frac{1}{2}, z + \frac{1}{2}$; (iii) $x - \frac{1}{2}, -y + \frac{1}{2}, z - \frac{1}{2}$; (iv) $-x + \frac{3}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$; (v) $-x + \frac{3}{2}, y + \frac{1}{2}, -z + \frac{3}{2}$.]


Figure 8

Packing diagram for (III), showing the chains joined by $N-H \cdots N \equiv C$ hydrogen bonds. Hydrogen bonds are represented by dashed lines. H atoms not involved in the hydrogen-bonding network are not shown. [Symmetry identifiers: (i) $-x + 1, -y + 1, -z + 1$; (ii) $-x + 2, -y + 1, -z + 1$; (iii) $x + 1, y - 1, z - 1$; (iv) $-x + 2, -y, -z$; (v) $x, y - 1, z - 1$.]

5.2. Preparation of (II)

4-Chlorobenzene-1,2-diamine (0.106 g, 0.75 mmol) was dissolved in boiling ethanol (10 ml) and lead(II) acetate trihydrate (0.134 g, 0.35 mmol) was added with stirring. The resulting solution was refluxed for 4 h, removed from the heat and the solvent was allowed to slowly evaporate. The residue obtained was dissolved in hot methanol and passed through a 45 μm pore filter. Crystals suitable for X-ray analysis were obtained after slow evaporation of the solvent. Further solvent reduction resulted in precipitation of excess diamine and so the overall yield was not determined. Selected IR bands

(diamond anvil, cm^{-1}): 3334 (*br*), 1537 (*s*), 1393 (*s*), 1337 (*s*), 1018 (*s*).

5.3. Preparation of (III)

To a solution of lead(II) acetate trihydrate (0.149 g, 0.39 mmol) in ethanol (10 ml) was added 3,4-diaminobenzonitrile (0.104 g, 0.75 mmol). The resulting solution was stirred at a gentle reflux for 1 h. The solvent was allowed to slowly evaporate over a period of 3 d, resulting in crystals suitable for X-ray analysis. Further solvent reduction resulted in precipitation of excess diamine and so the overall yield was not

Table 7
Experimental details.

	(I)	(II)	(III)
Crystal data			
Chemical formula	$[\text{Pb}_4(\text{C}_2\text{H}_3\text{O}_2)_8(\text{C}_6\text{H}_8\text{N}_2)_2]$	$[\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2(\text{C}_6\text{H}_7\text{ClN}_2)]$	$[\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2(\text{C}_7\text{H}_7\text{N}_3)]$
M_r	1517.40	467.86	458.43
Crystal system, space group	Monoclinic, $P2_1/n$	Triclinic, $P\bar{1}$	Triclinic, $P\bar{1}$
Temperature (K)	200	200	200
a, b, c (Å)	11.1447 (14), 29.694 (4), 11.8597 (14)	7.3623 (10), 7.6177 (10), 13.1413 (17)	7.3724 (8), 7.6349 (8), 13.4069 (15)
α, β, γ (°)	90, 103.941 (4), 90	89.762 (4), 76.405 (4), 66.691 (4)	88.839 (3), 78.330 (3), 66.035 (3)
V (Å ³)	3809.1 (8)	654.63 (15)	673.71 (13)
Z	4	2	2
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
μ (mm ⁻¹)	17.70	13.10	12.54
Crystal size (mm)	0.50 × 0.30 × 0.10	0.30 × 0.10 × 0.10	0.30 × 0.20 × 0.05
Data collection			
Diffractometer	Bruker SMART X2S benchtop	Bruker SMART X2S benchtop	Bruker SMART X2S benchtop
Absorption correction	Multi-scan (SADABS; Bruker, 2013)	Multi-scan (SADABS; Bruker, 2013)	Multi-scan (SADABS; Bruker, 2013)
$T_{\text{min}}, T_{\text{max}}$	0.12, 0.27	0.11, 0.35	0.12, 0.57
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	26670, 7708, 5239	6572, 2572, 2262	8223, 2819, 2498
R_{int}	0.080	0.057	0.053
$(\sin \theta/\lambda)_{\text{max}}$ (Å ⁻¹)	0.624	0.625	0.641
Refinement			
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.042, 0.093, 0.96	0.038, 0.100, 1.04	0.041, 0.145, 1.14
No. of reflections	7708	2572	2819
No. of parameters	502	178	187
No. of restraints	122	6	96
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å ⁻³)	2.12, -1.97	3.38, -3.08	3.41, -2.68

Computer programs: APEX2 and SAINT (Bruker, 2013), SHELXS97 and SHELXL2014 (Sheldrick, 2008), PLATON (Spek, 2009), Mercury (Macrae *et al.*, 2006), ORTEP-3 for Windows (Farrugia, 2012) and publCIF (Westrip, 2010).

determined. Selected IR bands (diamond anvil, cm⁻¹): 3432 (*w*), 3316 (*w*), 2213 (*s*), 1581 (*s*), 1557 (*s*), 1394 (*s*), 1301 (*s*), 1020 (*s*).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 7. All H atoms were observed in difference Fourier maps. C-bonded H atoms were refined using a riding model, with C–H = 0.98 Å for the methyl groups and 0.95 Å for the aromatic ring. The C–H hydrogen isotropic displacement parameters were fixed using the approximation $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for the methyl H atoms and $1.2U_{\text{eq}}(\text{C})$ for the aromatic H atoms. The atomic coordinates for the amine H atoms were refined using an N–H bond-distance restraint of 0.88 (2) Å and the H-atom isotropic displacement parameters were set using the approximation $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{N})$. Late in the refinement, a correction for extinction was applied for each of the structures. For (I), the highest residual electron-density peak is 0.94 Å from Pb2 and the deepest hole is 1.20 Å from Pb3. The highest residual electron-density peak is 0.89 Å and the deepest hole is 0.91 Å from Pb1 in (II). For (III), the highest residual electron-

density peak and the deepest hole are 0.92 Å and 0.82 Å, respectively, from Pb1.

Acknowledgements

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Crystal structures of three lead(II) acetate-bridged diaminobenzene coordination polymers

David K. Geiger, Dylan E. Parsons and Patricia L. Zick

Computing details

For all compounds, data collection: *APEX2* (Bruker, 2013); cell refinement: *APEX2* (Bruker, 2013); data reduction: *SAINTE* (Bruker, 2013); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009), *Mercury* (Macrae *et al.*, 2006) and *ORTEP-3* for Windows (Farrugia, 2012); software used to prepare material for publication: *publCIF* (Westrip, 2010).

(I) Poly[tris(acetato- κ^2O,O')(μ_3 -acetato- $\kappa^3O,O':O$)tetrakis(μ_2 -acetato- $\kappa^4O,O':O:O'$)bis(benzene-1,2-diamine- κN)tetralead(II)]

Crystal data

[Pb₄(C₂H₃O₂)₈(C₆H₈N₂)₂]

$M_r = 1517.40$

Monoclinic, $P2_1/n$

$a = 11.1447$ (14) Å

$b = 29.694$ (4) Å

$c = 11.8597$ (14) Å

$\beta = 103.941$ (4)°

$V = 3809.1$ (8) Å³

$Z = 4$

$F(000) = 2768$

$D_x = 2.646$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 426 reflections

$\theta = 0.1$ – 26.5°

$\mu = 17.70$ mm⁻¹

$T = 200$ K

Plate, clear colorless

$0.50 \times 0.30 \times 0.10$ mm

Data collection

Bruker SMART X2S benchtop
diffractometer

Radiation source: sealed microfocus tube

Doubly curved silicon crystal monochromator

ω scans

Absorption correction: multi-scan
(*SADABS*; Bruker, 2013)

$T_{\min} = 0.12$, $T_{\max} = 0.27$

26670 measured reflections

7708 independent reflections

5239 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.080$

$\theta_{\max} = 26.3^\circ$, $\theta_{\min} = 1.9^\circ$

$h = -13 \rightarrow 12$

$k = -36 \rightarrow 35$

$l = -9 \rightarrow 14$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.042$

$wR(F^2) = 0.093$

$S = 0.96$

7708 reflections

502 parameters

122 restraints

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map

Hydrogen site location: mixed

H atoms treated by a mixture of independent
and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0015P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 2.12 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -1.97 \text{ e } \text{\AA}^{-3}$

Extinction correction: *SHELXL2014* (Sheldrick, 2008)
 Extinction coefficient: 0.000185 (12)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Pb1	0.96723 (3)	0.43662 (2)	0.39419 (3)	0.02150 (11)
Pb2	1.09865 (4)	0.31230 (2)	0.58736 (3)	0.02407 (11)
Pb3	0.98507 (3)	0.18567 (2)	0.40741 (3)	0.02108 (11)
Pb4	1.07290 (3)	0.05882 (2)	0.60542 (3)	0.02225 (11)
O1	0.7399 (6)	0.4682 (2)	0.3553 (6)	0.0339 (18)
O2	0.7962 (6)	0.4054 (2)	0.4545 (6)	0.0276 (17)
O3	1.0040 (7)	0.4700 (2)	0.5906 (6)	0.0332 (18)
O4	1.0871 (7)	0.4030 (2)	0.5891 (6)	0.0326 (18)
O5	1.0131 (6)	0.3484 (2)	0.3958 (6)	0.0277 (17)
O6	1.0227 (7)	0.2751 (2)	0.3972 (6)	0.0298 (17)
O7	1.3471 (7)	0.3072 (3)	0.6898 (6)	0.051 (2)
O8	1.2763 (7)	0.3133 (3)	0.4997 (6)	0.047 (2)
O9	1.1040 (7)	0.2231 (2)	0.6007 (6)	0.037 (2)
O10	1.0341 (6)	0.1551 (2)	0.6181 (5)	0.0277 (17)
O11	0.8067 (7)	0.2193 (2)	0.4613 (7)	0.039 (2)
O12	0.7851 (6)	0.1481 (2)	0.4133 (6)	0.0344 (18)
O13	1.0352 (6)	0.0970 (2)	0.4139 (6)	0.0278 (17)
O14	0.9730 (7)	0.0274 (2)	0.4033 (6)	0.0331 (19)
O15	1.3094 (7)	0.0892 (2)	0.6606 (6)	0.039 (2)
O16	1.2513 (6)	0.0321 (2)	0.5413 (6)	0.0328 (18)
N1	0.8445 (8)	0.3134 (3)	0.5734 (7)	0.0268 (19)
H1A	0.825 (8)	0.3367 (16)	0.525 (6)	0.032*
H1B	0.811 (8)	0.2888 (15)	0.538 (6)	0.032*
N2	0.8160 (13)	0.2434 (4)	0.7296 (9)	0.069 (4)
H2A	0.796 (12)	0.2323 (18)	0.657 (3)	0.082*
H2B	0.818 (12)	0.2193 (12)	0.776 (5)	0.082*
N3	0.8106 (9)	0.0629 (3)	0.5758 (7)	0.034 (2)
H3A	0.794 (9)	0.0893 (12)	0.543 (7)	0.04*
H3B	0.774 (8)	0.0418 (19)	0.527 (6)	0.04*
N4	0.8000 (10)	0.1348 (3)	0.7272 (10)	0.052 (3)
H4A	0.827 (9)	0.146 (3)	0.798 (4)	0.062*
H4B	0.851 (8)	0.142 (3)	0.684 (7)	0.062*
C1	0.8190 (9)	0.3205 (3)	0.6868 (8)	0.026 (2)
C2	0.8133 (10)	0.3672 (4)	0.7200 (9)	0.040 (3)
H2	0.8224	0.3908	0.6687	0.048*
C3	0.7946 (11)	0.3763 (4)	0.8272 (10)	0.049 (3)

H3	0.7895	0.4067	0.8512	0.059*
C4	0.7832 (10)	0.3414 (4)	0.9007 (10)	0.040 (3)
H4	0.7714	0.3487	0.9753	0.048*
C5	0.7879 (10)	0.2975 (4)	0.8724 (9)	0.038 (3)
H5	0.7771	0.2745	0.9248	0.045*
C6	0.8096 (10)	0.2864 (4)	0.7613 (9)	0.039 (3)
C8	0.7861 (9)	0.0904 (4)	0.7650 (9)	0.033 (2)
C7	0.7876 (9)	0.0548 (3)	0.6853 (9)	0.028 (2)
C12	0.7734 (10)	0.0105 (4)	0.7208 (9)	0.035 (3)
H12	0.7772	-0.0137	0.6695	0.042*
C11	0.7537 (11)	0.0016 (4)	0.8298 (9)	0.043 (3)
H11	0.7385	-0.0283	0.851	0.051*
C10	0.7565 (10)	0.0366 (4)	0.9073 (10)	0.038 (3)
H10	0.7481	0.0302	0.9835	0.046*
C9	0.7711 (9)	0.0805 (4)	0.8758 (9)	0.031 (2)
H9	0.7709	0.1042	0.9297	0.037*
C13	0.7171 (9)	0.4340 (3)	0.4121 (8)	0.021 (2)
C14	0.5894 (9)	0.4294 (4)	0.4293 (10)	0.044 (3)
H14A	0.5768	0.3986	0.4536	0.066*
H14B	0.5292	0.4361	0.3562	0.066*
H14C	0.5783	0.4506	0.4894	0.066*
C15	1.0659 (9)	0.4370 (3)	0.6435 (8)	0.021 (2)
C16	1.1129 (12)	0.4395 (4)	0.7736 (9)	0.052 (4)
H16A	1.1738	0.4638	0.7937	0.077*
H16B	1.1519	0.4108	0.8026	0.077*
H16C	1.0437	0.4454	0.8092	0.077*
C17	0.9977 (8)	0.3114 (3)	0.3407 (7)	0.019 (2)
C18	0.9578 (11)	0.3100 (4)	0.2115 (8)	0.043 (3)
H18A	1.0306	0.3075	0.1792	0.065*
H18B	0.9126	0.3377	0.183	0.065*
H18C	0.9038	0.2839	0.1872	0.065*
C19	1.3646 (10)	0.3104 (4)	0.5901 (10)	0.033 (3)
C20	1.4962 (10)	0.3101 (4)	0.5755 (10)	0.053 (3)
H20A	1.5138	0.3389	0.5423	0.08*
H20B	1.5059	0.2855	0.5234	0.08*
H20C	1.5538	0.3058	0.6514	0.08*
C21	1.0930 (9)	0.1891 (4)	0.6614 (8)	0.024 (2)
C22	1.1513 (12)	0.1894 (4)	0.7902 (8)	0.053 (4)
H22A	1.2099	0.2145	0.8089	0.08*
H22B	1.1951	0.1609	0.8125	0.08*
H22C	1.0867	0.1929	0.8329	0.08*
C23	0.7422 (10)	0.1834 (4)	0.4410 (9)	0.036 (3)
C24	0.6089 (10)	0.1854 (4)	0.4486 (10)	0.045 (3)
H24A	0.5571	0.1956	0.374	0.068*
H24B	0.6011	0.2066	0.5098	0.068*
H24C	0.5821	0.1555	0.4669	0.068*
C25	0.9838 (9)	0.0635 (3)	0.3540 (8)	0.024 (2)
C26	0.9410 (10)	0.0680 (4)	0.2243 (8)	0.042 (3)

H26A	0.9203	0.0381	0.1897	0.063*
H26B	1.007	0.0813	0.1936	0.063*
H26C	0.8677	0.0873	0.2048	0.063*
C27	1.3316 (9)	0.0598 (4)	0.5943 (8)	0.028 (3)
C28	1.4623 (9)	0.0562 (4)	0.5755 (10)	0.045 (3)
H28A	1.4851	0.0847	0.5449	0.068*
H28B	1.4649	0.0319	0.52	0.068*
H28C	1.5207	0.0495	0.6497	0.068*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pb1	0.0228 (2)	0.0223 (2)	0.0190 (2)	0.00101 (17)	0.00423 (15)	0.00183 (17)
Pb2	0.0257 (2)	0.0232 (2)	0.0211 (2)	-0.00045 (18)	0.00127 (16)	0.00155 (19)
Pb3	0.0208 (2)	0.0210 (2)	0.02038 (19)	-0.00022 (17)	0.00290 (15)	-0.00038 (18)
Pb4	0.0243 (2)	0.0220 (2)	0.0198 (2)	-0.00163 (17)	0.00413 (16)	-0.00230 (17)
O1	0.030 (4)	0.031 (4)	0.045 (5)	0.005 (4)	0.018 (4)	0.006 (4)
O2	0.024 (4)	0.026 (4)	0.031 (4)	0.005 (3)	0.004 (3)	0.009 (3)
O3	0.042 (5)	0.024 (4)	0.030 (4)	0.001 (4)	0.000 (4)	-0.004 (3)
O4	0.050 (5)	0.020 (4)	0.024 (4)	0.005 (4)	0.002 (4)	-0.005 (3)
O5	0.039 (5)	0.016 (4)	0.024 (4)	0.006 (3)	-0.001 (3)	0.000 (3)
O6	0.038 (5)	0.023 (4)	0.026 (4)	-0.003 (3)	0.004 (3)	0.000 (3)
O7	0.037 (5)	0.084 (7)	0.029 (4)	0.002 (5)	0.001 (4)	-0.005 (5)
O8	0.030 (5)	0.068 (6)	0.038 (4)	-0.002 (4)	-0.003 (4)	0.012 (4)
O9	0.046 (5)	0.028 (4)	0.033 (4)	-0.014 (4)	-0.001 (4)	0.003 (4)
O10	0.041 (5)	0.018 (4)	0.023 (4)	-0.004 (3)	0.005 (3)	0.004 (3)
O11	0.038 (5)	0.032 (5)	0.054 (5)	-0.005 (4)	0.022 (4)	-0.014 (4)
O12	0.030 (4)	0.024 (4)	0.051 (5)	-0.006 (3)	0.014 (4)	-0.009 (4)
O13	0.036 (4)	0.022 (4)	0.031 (4)	-0.002 (3)	0.017 (3)	0.000 (3)
O14	0.049 (5)	0.022 (4)	0.028 (4)	-0.017 (4)	0.009 (4)	-0.006 (3)
O15	0.041 (5)	0.040 (5)	0.043 (5)	-0.004 (4)	0.022 (4)	-0.017 (4)
O16	0.028 (4)	0.026 (4)	0.043 (5)	-0.004 (3)	0.004 (4)	-0.014 (4)
N1	0.033 (5)	0.023 (5)	0.025 (4)	0.000 (4)	0.008 (4)	-0.002 (4)
N2	0.095 (10)	0.047 (5)	0.069 (8)	0.000 (5)	0.031 (8)	0.000 (5)
N3	0.047 (6)	0.026 (5)	0.031 (4)	-0.004 (5)	0.016 (4)	0.003 (4)
N4	0.062 (8)	0.032 (5)	0.066 (7)	-0.005 (4)	0.023 (6)	0.002 (5)
C1	0.021 (5)	0.034 (5)	0.020 (4)	-0.002 (4)	0.001 (4)	0.003 (3)
C2	0.045 (7)	0.044 (5)	0.031 (5)	0.000 (5)	0.009 (5)	-0.006 (4)
C3	0.055 (8)	0.048 (6)	0.046 (5)	0.007 (5)	0.016 (5)	0.000 (4)
C4	0.028 (6)	0.051 (5)	0.040 (6)	0.001 (5)	0.005 (5)	0.006 (4)
C5	0.026 (6)	0.049 (5)	0.035 (5)	0.003 (5)	0.001 (4)	0.006 (4)
C6	0.038 (7)	0.042 (5)	0.037 (5)	-0.001 (4)	0.007 (4)	0.008 (4)
C8	0.023 (6)	0.033 (5)	0.041 (5)	-0.003 (4)	0.006 (4)	-0.002 (4)
C7	0.022 (5)	0.033 (4)	0.027 (4)	-0.002 (4)	0.003 (4)	0.001 (3)
C12	0.036 (6)	0.031 (5)	0.037 (5)	-0.006 (4)	0.007 (4)	0.000 (4)
C11	0.045 (7)	0.041 (6)	0.044 (5)	0.002 (5)	0.016 (5)	0.008 (4)
C10	0.031 (6)	0.043 (5)	0.041 (5)	0.004 (5)	0.010 (5)	0.005 (4)
C9	0.019 (5)	0.040 (5)	0.031 (5)	0.005 (4)	0.002 (4)	-0.003 (4)

C13	0.022 (5)	0.028 (6)	0.016 (5)	-0.005 (5)	0.009 (4)	-0.009 (5)
C14	0.026 (6)	0.061 (9)	0.045 (7)	-0.005 (6)	0.010 (5)	-0.015 (7)
C15	0.029 (6)	0.014 (5)	0.019 (5)	-0.007 (5)	0.004 (4)	0.000 (5)
C16	0.064 (9)	0.058 (9)	0.027 (7)	0.013 (7)	-0.001 (6)	0.003 (6)
C17	0.018 (5)	0.020 (5)	0.019 (5)	0.005 (4)	0.003 (4)	-0.004 (5)
C18	0.064 (9)	0.040 (7)	0.024 (6)	-0.006 (7)	0.006 (6)	0.000 (6)
C19	0.024 (6)	0.025 (6)	0.048 (7)	-0.007 (5)	0.005 (5)	0.001 (6)
C20	0.035 (8)	0.067 (9)	0.055 (8)	-0.008 (7)	0.005 (6)	0.010 (8)
C21	0.025 (6)	0.020 (6)	0.027 (5)	0.002 (5)	0.005 (4)	-0.004 (5)
C22	0.065 (9)	0.068 (9)	0.022 (6)	-0.014 (8)	0.001 (6)	0.017 (7)
C23	0.030 (7)	0.042 (8)	0.033 (6)	0.001 (6)	0.006 (5)	-0.003 (6)
C24	0.025 (7)	0.057 (8)	0.056 (8)	0.001 (6)	0.015 (6)	-0.001 (7)
C25	0.026 (6)	0.025 (6)	0.021 (5)	0.001 (5)	0.005 (4)	-0.006 (5)
C26	0.037 (7)	0.068 (9)	0.016 (5)	-0.004 (6)	-0.004 (5)	0.007 (6)
C27	0.023 (6)	0.048 (8)	0.013 (5)	0.002 (5)	0.003 (4)	0.013 (5)
C28	0.025 (6)	0.061 (8)	0.053 (8)	-0.004 (6)	0.014 (6)	-0.018 (7)

Geometric parameters (Å, °)

Pb1—O2	2.380 (6)	N4—H4A	0.88 (2)
Pb1—O3	2.474 (7)	N4—H4B	0.88 (2)
Pb1—O4	2.576 (7)	C1—C6	1.366 (13)
Pb1—O1	2.636 (7)	C1—C2	1.445 (14)
Pb1—O5	2.667 (6)	C2—C3	1.364 (14)
Pb1—O3 ⁱ	2.792 (7)	C2—H2	0.95
Pb2—O8	2.448 (8)	C3—C4	1.380 (14)
Pb2—O6	2.470 (7)	C3—H3	0.95
Pb2—O5	2.485 (6)	C4—C5	1.352 (14)
Pb2—O9	2.654 (7)	C4—H4	0.95
Pb2—O4	2.696 (6)	C5—C6	1.434 (14)
Pb2—O7	2.747 (8)	C5—H5	0.95
Pb2—N1	2.797 (9)	C8—C9	1.395 (14)
Pb3—O11	2.443 (7)	C8—C7	1.421 (14)
Pb3—O12	2.509 (7)	C7—C12	1.401 (13)
Pb3—O10	2.590 (6)	C12—C11	1.388 (14)
Pb3—O9	2.604 (7)	C12—H12	0.95
Pb3—O7 ⁱⁱ	2.675 (7)	C11—C10	1.382 (14)
Pb3—O13	2.688 (6)	C11—H11	0.95
Pb3—O6	2.696 (7)	C10—C9	1.377 (14)
Pb4—O16	2.427 (7)	C10—H10	0.95
Pb4—O13	2.482 (7)	C9—H9	0.95
Pb4—O14	2.563 (7)	C13—C14	1.492 (13)
Pb4—O14 ⁱⁱⁱ	2.609 (7)	C14—H14A	0.98
Pb4—O15	2.713 (7)	C14—H14B	0.98
Pb4—O10	2.901 (6)	C14—H14C	0.98
Pb4—N3	2.862 (10)	C15—C16	1.507 (13)
O1—C13	1.279 (11)	C16—H16A	0.98
O2—C13	1.239 (11)	C16—H16B	0.98

O3—C15	1.273 (11)	C16—H16C	0.98
O4—C15	1.252 (11)	C17—C18	1.490 (12)
O5—C17	1.270 (10)	C18—H18A	0.98
O6—C17	1.263 (11)	C18—H18B	0.98
O7—C19	1.248 (12)	C18—H18C	0.98
O7—Pb3 ^{iv}	2.675 (7)	C19—C20	1.517 (14)
O8—C19	1.272 (12)	C20—H20A	0.98
O9—C21	1.262 (11)	C20—H20B	0.98
O10—C21	1.246 (11)	C20—H20C	0.98
O11—C23	1.276 (12)	C21—C22	1.509 (13)
O12—C23	1.230 (12)	C22—H22A	0.98
O13—C25	1.276 (11)	C22—H22B	0.98
O14—C25	1.240 (11)	C22—H22C	0.98
O14—Pb4 ⁱⁱⁱ	2.609 (7)	C23—C24	1.511 (14)
O15—C27	1.239 (12)	C24—H24A	0.98
O16—C27	1.266 (12)	C24—H24B	0.98
N1—C1	1.456 (11)	C24—H24C	0.98
N1—H1A	0.89 (2)	C25—C26	1.503 (13)
N1—H1B	0.88 (2)	C26—H26A	0.98
N2—C6	1.336 (14)	C26—H26B	0.98
N2—H2A	0.899 (19)	C26—H26C	0.98
N2—H2B	0.898 (19)	C27—C28	1.530 (13)
N3—C7	1.403 (12)	C28—H28A	0.98
N3—H3A	0.87 (2)	C28—H28B	0.98
N3—H3B	0.88 (2)	C28—H28C	0.98
N4—C8	1.415 (14)		
O2—Pb1—O3	80.6 (2)	C8—N4—H4A	95 (7)
O2—Pb1—O4	81.9 (2)	C8—N4—H4B	124 (7)
O3—Pb1—O4	51.5 (2)	H4A—N4—H4B	110 (5)
O2—Pb1—O1	52.1 (2)	C6—C1—C2	121.3 (10)
O3—Pb1—O1	87.5 (2)	C6—C1—N1	123.5 (9)
O4—Pb1—O1	124.3 (2)	C2—C1—N1	115.0 (9)
O2—Pb1—O5	77.4 (2)	C3—C2—C1	118.2 (10)
O3—Pb1—O5	113.5 (2)	C3—C2—H2	120.9
O4—Pb1—O5	63.6 (2)	C1—C2—H2	120.9
O1—Pb1—O5	121.7 (2)	C2—C3—C4	119.8 (11)
O8—Pb2—O6	75.6 (2)	C2—C3—H3	120.1
O8—Pb2—O5	77.2 (2)	C4—C3—H3	120.1
O6—Pb2—O5	52.2 (2)	C5—C4—C3	123.6 (11)
O8—Pb2—O9	91.6 (2)	C5—C4—H4	118.2
O6—Pb2—O9	66.8 (2)	C3—C4—H4	118.2
O5—Pb2—O9	118.9 (2)	C4—C5—C6	118.4 (11)
O8—Pb2—O4	92.1 (2)	C4—C5—H5	120.8
O6—Pb2—O4	116.5 (2)	C6—C5—H5	120.8
O5—Pb2—O4	64.4 (2)	N2—C6—C1	120.6 (11)
O9—Pb2—O4	175.6 (2)	N2—C6—C5	120.7 (10)
O8—Pb2—O7	49.9 (2)	C1—C6—C5	118.7 (11)

O6—Pb2—O7	117.8 (2)	C9—C8—N4	122.8 (10)
O5—Pb2—O7	123.5 (2)	C9—C8—C7	119.6 (10)
O9—Pb2—O7	85.0 (2)	N4—C8—C7	117.6 (10)
O4—Pb2—O7	95.5 (2)	C12—C7—N3	119.9 (9)
O8—Pb2—N1	152.3 (2)	C12—C7—C8	118.5 (10)
O6—Pb2—N1	80.3 (2)	N3—C7—C8	121.5 (9)
O5—Pb2—N1	77.3 (2)	C11—C12—C7	121.0 (10)
O9—Pb2—N1	91.3 (2)	C11—C12—H12	119.5
O4—Pb2—N1	86.5 (2)	C7—C12—H12	119.5
O7—Pb2—N1	157.7 (2)	C10—C11—C12	119.5 (11)
O11—Pb3—O12	52.5 (2)	C10—C11—H11	120.3
O11—Pb3—O10	83.5 (2)	C12—C11—H11	120.3
O12—Pb3—O10	78.4 (2)	C9—C10—C11	121.1 (11)
O11—Pb3—O9	82.5 (2)	C9—C10—H10	119.4
O12—Pb3—O9	115.6 (2)	C11—C10—H10	119.4
O10—Pb3—O9	49.9 (2)	C10—C9—C8	120.2 (10)
O11—Pb3—O7 ⁱⁱ	84.6 (3)	C10—C9—H9	119.9
O12—Pb3—O7 ⁱⁱ	75.7 (2)	C8—C9—H9	119.9
O10—Pb3—O7 ⁱⁱ	153.7 (2)	O2—C13—O1	123.1 (9)
O9—Pb3—O7 ⁱⁱ	150.2 (2)	O2—C13—C14	119.4 (9)
O11—Pb3—O13	124.7 (2)	O1—C13—C14	117.4 (9)
O12—Pb3—O13	75.1 (2)	C13—C14—H14A	109.5
O10—Pb3—O13	68.6 (2)	C13—C14—H14B	109.5
O9—Pb3—O13	109.5 (2)	H14A—C14—H14B	109.5
O7 ⁱⁱ —Pb3—O13	99.9 (2)	C13—C14—H14C	109.5
O11—Pb3—O6	75.7 (2)	H14A—C14—H14C	109.5
O12—Pb3—O6	126.0 (2)	H14B—C14—H14C	109.5
O10—Pb3—O6	112.9 (2)	O4—C15—O3	120.9 (9)
O9—Pb3—O6	64.4 (2)	O4—C15—C16	120.2 (9)
O7 ⁱⁱ —Pb3—O6	86.5 (2)	O3—C15—C16	118.8 (9)
O13—Pb3—O6	158.8 (2)	C15—C16—H16A	109.5
O16—Pb4—O13	80.4 (2)	C15—C16—H16B	109.5
O16—Pb4—O14	78.0 (2)	H16A—C16—H16B	109.5
O13—Pb4—O14	51.0 (2)	C15—C16—H16C	109.5
O16—Pb4—O14 ⁱⁱⁱ	80.4 (2)	H16A—C16—H16C	109.5
O13—Pb4—O14 ⁱⁱⁱ	115.1 (2)	H16B—C16—H16C	109.5
O14—Pb4—O14 ⁱⁱⁱ	64.5 (3)	O6—C17—O5	118.7 (8)
O16—Pb4—O15	50.4 (2)	O6—C17—C18	119.7 (9)
O13—Pb4—O15	90.5 (2)	O5—C17—C18	121.5 (9)
O14—Pb4—O15	121.6 (2)	C17—C18—H18A	109.5
O14 ⁱⁱⁱ —Pb4—O15	120.4 (2)	C17—C18—H18B	109.5
O16—Pb4—N3	149.9 (2)	H18A—C18—H18B	109.5
O13—Pb4—N3	85.7 (2)	C17—C18—H18C	109.5
O14—Pb4—N3	72.5 (2)	H18A—C18—H18C	109.5
O14 ⁱⁱⁱ —Pb4—N3	81.5 (2)	H18B—C18—H18C	109.5
O15—Pb4—N3	156.9 (2)	O7—C19—O8	122.5 (10)
C13—O1—Pb1	85.9 (5)	O7—C19—C20	119.0 (10)
C13—O2—Pb1	98.9 (6)	O8—C19—C20	118.5 (10)

C15—O3—Pb1	95.9 (6)	C19—C20—H20A	109.5
C15—O4—Pb1	91.6 (6)	C19—C20—H20B	109.5
C15—O4—Pb2	145.7 (6)	H20A—C20—H20B	109.5
Pb1—O4—Pb2	113.3 (2)	C19—C20—H20C	109.5
C17—O5—Pb2	94.1 (5)	H20A—C20—H20C	109.5
C17—O5—Pb1	147.7 (6)	H20B—C20—H20C	109.5
Pb2—O5—Pb1	117.5 (2)	O10—C21—O9	121.7 (9)
C17—O6—Pb2	95.0 (5)	O10—C21—C22	118.5 (9)
C17—O6—Pb3	147.9 (6)	O9—C21—C22	119.8 (9)
Pb2—O6—Pb3	115.0 (3)	C21—C22—H22A	109.5
C19—O7—Pb3 ^{iv}	136.3 (7)	C21—C22—H22B	109.5
C19—O7—Pb2	87.0 (6)	H22A—C22—H22B	109.5
Pb3 ^{iv} —O7—Pb2	134.9 (3)	C21—C22—H22C	109.5
C19—O8—Pb2	100.5 (6)	H22A—C22—H22C	109.5
C21—O9—Pb3	93.6 (6)	H22B—C22—H22C	109.5
C21—O9—Pb2	145.9 (6)	O12—C23—O11	121.8 (10)
Pb3—O9—Pb2	112.0 (3)	O12—C23—C24	120.0 (11)
C21—O10—Pb3	94.7 (6)	O11—C23—C24	118.2 (10)
C23—O11—Pb3	93.8 (6)	C23—C24—H24A	109.5
C23—O12—Pb3	91.8 (6)	C23—C24—H24B	109.5
C25—O13—Pb4	96.2 (6)	H24A—C24—H24B	109.5
C25—O13—Pb3	133.3 (6)	C23—C24—H24C	109.5
Pb4—O13—Pb3	117.5 (3)	H24A—C24—H24C	109.5
C25—O14—Pb4	93.3 (6)	H24B—C24—H24C	109.5
C25—O14—Pb4 ⁱⁱⁱ	150.4 (6)	O14—C25—O13	119.5 (9)
Pb4—O14—Pb4 ⁱⁱⁱ	115.5 (3)	O14—C25—C26	121.0 (9)
C27—O15—Pb4	86.8 (6)	O13—C25—C26	119.4 (9)
C27—O16—Pb4	99.5 (6)	C25—C26—H26A	109.5
C1—N1—Pb2	111.6 (6)	C25—C26—H26B	109.5
C1—N1—H1A	114 (6)	H26A—C26—H26B	109.5
Pb2—N1—H1A	98 (6)	C25—C26—H26C	109.5
C1—N1—H1B	114 (6)	H26A—C26—H26C	109.5
Pb2—N1—H1B	109 (6)	H26B—C26—H26C	109.5
H1A—N1—H1B	109 (4)	O15—C27—O16	123.2 (9)
C6—N2—H2A	127 (4)	O15—C27—C28	119.0 (10)
C6—N2—H2B	126 (4)	O16—C27—C28	117.8 (9)
H2A—N2—H2B	105 (4)	C27—C28—H28A	109.5
C7—N3—Pb4	106.7 (6)	C27—C28—H28B	109.5
C7—N3—H3A	119 (6)	H28A—C28—H28B	109.5
Pb4—N3—H3A	101 (7)	C27—C28—H28C	109.5
C7—N3—H3B	109 (6)	H28A—C28—H28C	109.5
Pb4—N3—H3B	110 (7)	H28B—C28—H28C	109.5
H3A—N3—H3B	110 (5)		

Symmetry codes: (i) $-x+2, -y+1, -z+1$; (ii) $x-1/2, -y+1/2, z-1/2$; (iii) $-x+2, -y, -z+1$; (iv) $x+1/2, -y+1/2, z+1/2$.

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—H1 <i>A</i> ...O2	0.89 (2)	2.20 (3)	3.061 (11)	163 (7)
N1—H1 <i>B</i> ...O11	0.88 (2)	2.25 (4)	3.079 (11)	156 (8)
N2—H2 <i>A</i> ...O11	0.90 (2)	2.38 (5)	3.238 (13)	159 (11)
N2—H2 <i>B</i> ...N4	0.90 (2)	2.57 (5)	3.229 (14)	131 (5)
N3—H3 <i>A</i> ...O12	0.87 (2)	2.32 (4)	3.151 (11)	160 (8)
N3—H3 <i>B</i> ...O16 ⁱⁱⁱ	0.88 (2)	2.33 (4)	3.145 (11)	154 (7)
N4—H4 <i>B</i> ...O10	0.88 (2)	2.38 (4)	3.236 (12)	163 (10)
C2—H2...O2	0.95	2.52	3.309 (13)	140
C12—H12...O16 ⁱⁱⁱ	0.95	2.5	3.305 (13)	142
C9—H9...O8 ^v	0.95	2.58	3.474 (13)	156
C16—H16 <i>B</i> ...O12 ^{iv}	0.98	2.46	3.413 (13)	165
C18—H18 <i>B</i> ...O15 ⁱⁱ	0.98	2.44	3.403 (14)	167

Symmetry codes: (ii) $x-1/2, -y+1/2, z-1/2$; (iii) $-x+2, -y, -z+1$; (iv) $x+1/2, -y+1/2, z+1/2$; (v) $x-1/2, -y+1/2, z+1/2$.

(II) Poly[acetato- κ^2 O,O'](μ_3 -acetato- κ^4 O,O':O:O')(4-chlorobenzene-1,2-diamine- κ N)lead(II)]

Crystal data

[Pb(C₂H₃O₂)₂(C₆H₇ClN₂)₂]

$M_r = 467.86$

Triclinic, $P\bar{1}$

$a = 7.3623$ (10) Å

$b = 7.6177$ (10) Å

$c = 13.1413$ (17) Å

$\alpha = 89.762$ (4)°

$\beta = 76.405$ (4)°

$\gamma = 66.691$ (4)°

$V = 654.63$ (15) Å³

$Z = 2$

$F(000) = 436$

$D_x = 2.374$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 120 reflections

$\theta = 3.6$ – 25.7 °

$\mu = 13.10$ mm⁻¹

$T = 200$ K

Needle, clear orange

$0.30 \times 0.10 \times 0.10$ mm

Data collection

Bruker SMART X2S benchtop
diffractometer

Radiation source: sealed microfocus tube

Doubly curved silicon crystal monochromator

ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2013)

$T_{\min} = 0.11, T_{\max} = 0.35$

6572 measured reflections

2572 independent reflections

2262 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.057$

$\theta_{\max} = 26.4$ °, $\theta_{\min} = 2.9$ °

$h = -9 \rightarrow 9$

$k = -9 \rightarrow 9$

$l = -15 \rightarrow 16$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.038$

$wR(F^2) = 0.100$

$S = 1.04$

2572 reflections

178 parameters

6 restraints

Hydrogen site location: mixed

H atoms treated by a mixture of independent
and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.054P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 3.38$ e Å⁻³

$\Delta\rho_{\min} = -3.08$ e Å⁻³

Extinction correction: *SHELXL2014* (Sheldrick,
2008)

Extinction coefficient: 0.0042 (8)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Pb1	0.67144 (4)	0.16792 (4)	0.53160 (2)	0.01693 (17)
Cl1	0.2021 (4)	0.3992 (4)	1.0948 (2)	0.0413 (7)
O1	0.8134 (9)	0.2240 (10)	0.3510 (5)	0.0263 (15)
O2	0.4752 (10)	0.3657 (10)	0.3929 (6)	0.0342 (17)
O3	0.6797 (9)	-0.1072 (9)	0.4242 (5)	0.0239 (15)
O4	0.9660 (9)	-0.1498 (9)	0.4639 (5)	0.0246 (15)
N1	0.7013 (12)	-0.0788 (11)	0.6912 (6)	0.0223 (17)
H1A	0.652 (11)	-0.160 (9)	0.675 (8)	0.027*
H1B	0.827 (6)	-0.145 (10)	0.696 (8)	0.027*
N2	0.8707 (15)	0.0961 (18)	0.8079 (9)	0.057 (3)
H2A	0.951 (13)	0.031 (17)	0.749 (5)	0.068*
H2B	0.933 (14)	0.138 (18)	0.844 (8)	0.068*
C1	0.5832 (14)	0.0307 (12)	0.7902 (7)	0.0204 (19)
C2	0.6734 (14)	0.1165 (13)	0.8450 (7)	0.022 (2)
C3	0.5512 (14)	0.2296 (14)	0.9401 (8)	0.027 (2)
H3	0.608	0.2878	0.9803	0.033*
C4	0.3504 (14)	0.2563 (14)	0.9749 (7)	0.028 (2)
C5	0.2584 (14)	0.1756 (15)	0.9208 (8)	0.030 (2)
H5	0.1189	0.196	0.946	0.036*
C6	0.3793 (15)	0.0641 (14)	0.8284 (8)	0.028 (2)
H6	0.3202	0.0073	0.789	0.034*
C7	0.6442 (13)	0.3293 (14)	0.3291 (7)	0.023 (2)
C8	0.6519 (17)	0.4058 (15)	0.2235 (8)	0.036 (3)
H8A	0.542	0.3983	0.1962	0.054*
H8B	0.7843	0.3291	0.1748	0.054*
H8C	0.6344	0.5399	0.2306	0.054*
C9	0.8652 (12)	-0.2138 (13)	0.4197 (7)	0.0166 (18)
C10	0.9620 (17)	-0.4105 (15)	0.3656 (9)	0.040 (3)
H10A	0.9409	-0.5006	0.4156	0.06*
H10B	1.1089	-0.4461	0.3375	0.06*
H10C	0.9003	-0.415	0.3077	0.06*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pb1	0.0120 (2)	0.0172 (2)	0.0188 (2)	-0.00311 (14)	-0.00369 (13)	-0.00168 (14)
Cl1	0.0378 (15)	0.0373 (15)	0.0300 (14)	-0.0009 (12)	0.0004 (11)	-0.0101 (12)
O1	0.016 (3)	0.037 (4)	0.023 (3)	-0.006 (3)	-0.005 (3)	0.003 (3)
O2	0.019 (3)	0.035 (4)	0.043 (4)	-0.004 (3)	-0.010 (3)	0.006 (3)

O3	0.013 (3)	0.023 (4)	0.032 (4)	-0.004 (3)	-0.007 (3)	-0.003 (3)
O4	0.017 (3)	0.020 (3)	0.037 (4)	-0.005 (3)	-0.012 (3)	-0.004 (3)
N1	0.019 (4)	0.022 (4)	0.020 (4)	-0.003 (3)	-0.003 (3)	-0.002 (3)
N2	0.030 (5)	0.074 (8)	0.059 (7)	-0.021 (5)	0.005 (5)	-0.031 (6)
C1	0.023 (5)	0.015 (5)	0.019 (5)	-0.004 (4)	-0.005 (4)	0.005 (4)
C2	0.027 (5)	0.020 (5)	0.024 (5)	-0.015 (4)	-0.004 (4)	0.001 (4)
C3	0.029 (5)	0.023 (5)	0.029 (5)	-0.009 (4)	-0.011 (4)	-0.001 (4)
C4	0.027 (5)	0.020 (5)	0.017 (5)	0.006 (4)	-0.001 (4)	-0.005 (4)
C5	0.021 (5)	0.035 (6)	0.027 (5)	-0.008 (4)	-0.001 (4)	-0.004 (5)
C6	0.030 (5)	0.028 (5)	0.032 (5)	-0.013 (4)	-0.015 (4)	0.006 (4)
C7	0.016 (4)	0.022 (5)	0.025 (5)	0.000 (4)	-0.008 (4)	-0.008 (4)
C8	0.046 (6)	0.032 (6)	0.041 (6)	-0.022 (5)	-0.022 (5)	0.016 (5)
C9	0.010 (4)	0.021 (5)	0.017 (4)	-0.008 (3)	0.005 (3)	-0.004 (4)
C10	0.036 (6)	0.031 (6)	0.047 (7)	-0.003 (5)	-0.016 (5)	-0.014 (6)

Geometric parameters (Å, °)

Pb1—O1	2.467 (6)	N2—H2B	0.87 (2)
Pb1—O3	2.504 (6)	C1—C6	1.383 (13)
Pb1—O4	2.512 (6)	C1—C2	1.400 (13)
Pb1—O4 ⁱ	2.632 (6)	C2—C3	1.407 (13)
Pb1—O2	2.678 (7)	C3—C4	1.372 (13)
Pb1—O3 ⁱⁱ	2.734 (6)	C3—H3	0.95
Pb1—N1	2.800 (8)	C4—C5	1.379 (14)
Cl1—C4	1.767 (9)	C5—C6	1.374 (13)
O1—C7	1.286 (10)	C5—H5	0.95
O2—C7	1.252 (12)	C6—H6	0.95
O3—C9	1.270 (10)	C7—C8	1.500 (14)
O3—Pb1 ⁱⁱ	2.734 (6)	C8—H8A	0.98
O4—C9	1.272 (10)	C8—H8B	0.98
O4—Pb1 ⁱ	2.632 (6)	C8—H8C	0.98
N1—C1	1.430 (11)	C9—C10	1.478 (13)
N1—H1A	0.88 (2)	C10—H10A	0.98
N1—H1B	0.88 (2)	C10—H10B	0.98
N2—C2	1.364 (13)	C10—H10C	0.98
N2—H2A	0.87 (2)		
O1—Pb1—O3	77.8 (2)	H2A—N2—H2B	112 (5)
O1—Pb1—O4	77.8 (2)	C6—C1—C2	119.8 (8)
O3—Pb1—O4	51.77 (19)	C6—C1—N1	121.4 (8)
O1—Pb1—O4 ⁱ	76.6 (2)	C2—C1—N1	118.7 (8)
O3—Pb1—O4 ⁱ	114.49 (19)	N2—C2—C1	122.1 (9)
O4—Pb1—O4 ⁱ	64.3 (2)	N2—C2—C3	120.4 (8)
O1—Pb1—O2	50.8 (2)	C1—C2—C3	117.5 (8)
O3—Pb1—O2	81.2 (2)	C4—C3—C2	120.2 (9)
O4—Pb1—O2	117.4 (2)	C4—C3—H3	119.9
O4 ⁱ —Pb1—O2	121.3 (2)	C2—C3—H3	119.9
O1—Pb1—O3 ⁱⁱ	119.4 (2)	C3—C4—C5	123.0 (9)

O3—Pb1—O3 ⁱⁱ	64.1 (2)	C3—C4—C11	118.9 (8)
O4—Pb1—O3 ⁱⁱ	107.7 (2)	C5—C4—C11	118.2 (8)
O4 ⁱ —Pb1—O3 ⁱⁱ	161.4 (2)	C6—C5—C4	116.4 (9)
O2—Pb1—O3 ⁱⁱ	77.2 (2)	C6—C5—H5	121.8
O1—Pb1—N1	148.3 (2)	C4—C5—H5	121.8
O3—Pb1—N1	84.2 (2)	C5—C6—C1	123.1 (9)
O4—Pb1—N1	70.5 (2)	C5—C6—H6	118.4
O4 ⁱ —Pb1—N1	87.7 (2)	C1—C6—H6	118.4
O2—Pb1—N1	150.8 (2)	O2—C7—O1	121.5 (9)
O3 ⁱⁱ —Pb1—N1	73.7 (2)	O2—C7—C8	119.9 (8)
C7—O1—Pb1	98.4 (6)	O1—C7—C8	118.6 (9)
C7—O2—Pb1	89.4 (5)	C7—C8—H8A	109.5
C9—O3—Pb1	94.8 (5)	C7—C8—H8B	109.5
C9—O3—Pb1 ⁱⁱ	133.5 (6)	H8A—C8—H8B	109.5
Pb1—O3—Pb1 ⁱⁱ	115.9 (2)	C7—C8—H8C	109.5
C9—O4—Pb1	94.4 (5)	H8A—C8—H8C	109.5
C9—O4—Pb1 ⁱ	146.7 (5)	H8B—C8—H8C	109.5
Pb1—O4—Pb1 ⁱ	115.7 (2)	O3—C9—O4	119.0 (8)
C1—N1—Pb1	109.3 (5)	O3—C9—C10	120.6 (8)
C1—N1—H1A	111 (6)	O4—C9—C10	120.3 (8)
Pb1—N1—H1A	105 (6)	C9—C10—H10A	109.5
C1—N1—H1B	108 (6)	C9—C10—H10B	109.5
Pb1—N1—H1B	114 (7)	H10A—C10—H10B	109.5
H1A—N1—H1B	108 (5)	C9—C10—H10C	109.5
C2—N2—H2A	124 (7)	H10A—C10—H10C	109.5
C2—N2—H2B	123 (7)	H10B—C10—H10C	109.5

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

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—H1A...O2 ⁱⁱ	0.88 (2)	2.38 (3)	3.261 (11)	172 (9)
N1—H1B...O1 ⁱ	0.88 (2)	2.39 (5)	3.201 (10)	153 (8)
N2—H2A...O1 ⁱ	0.87 (2)	2.19 (6)	2.998 (11)	155 (13)

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

(III) Poly[(acetato- κ^2 O,O')(μ_3 -acetato- κ^4 O,O':O:O')(3,4-diaminobenzonitrile- κ N)lead(II)]

Crystal data

[Pb(C₂H₃O₂)₂(C₇H₇N₃)₂]

M_r = 458.43

Triclinic, *P*1̄

a = 7.3724 (8) Å

b = 7.6349 (8) Å

c = 13.4069 (15) Å

α = 88.839 (3)°

β = 78.330 (3)°

γ = 66.035 (3)°

V = 673.71 (13) Å³

Z = 2

F(000) = 428

D_x = 2.260 Mg m⁻³

Mo *K* α radiation, λ = 0.71073 Å

Cell parameters from 117 reflections

θ = 3.4–27.1°

μ = 12.54 mm⁻¹

T = 200 K

Plate, clear colourless

0.30 × 0.20 × 0.05 mm

Data collection

Bruker SMART X2S benchtop diffractometer	8223 measured reflections
Radiation source: sealed microfocus tube	2819 independent reflections
Doubly curved silicon crystal monochromator	2498 reflections with $I > 2\sigma(I)$
ω scans	$R_{\text{int}} = 0.053$
Absorption correction: multi-scan (SADABS; Bruker, 2013)	$\theta_{\text{max}} = 27.1^\circ$, $\theta_{\text{min}} = 2.9^\circ$
$T_{\text{min}} = 0.12$, $T_{\text{max}} = 0.57$	$h = -9 \rightarrow 9$
	$k = -9 \rightarrow 9$
	$l = -17 \rightarrow 17$

Refinement

Refinement on F^2	Hydrogen site location: mixed
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.041$	$w = 1/[\sigma^2(F_o^2) + (0.0965P)^2]$
$wR(F^2) = 0.145$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.14$	$(\Delta/\sigma)_{\text{max}} < 0.001$
2819 reflections	$\Delta\rho_{\text{max}} = 3.41 \text{ e } \text{\AA}^{-3}$
187 parameters	$\Delta\rho_{\text{min}} = -2.68 \text{ e } \text{\AA}^{-3}$
96 restraints	Extinction correction: <i>SHELXL2014</i> (Sheldrick, 2008)
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0029 (14)
Secondary atom site location: difference Fourier map	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Pb1	0.67380 (5)	0.66477 (5)	0.53022 (2)	0.0203 (2)
N1	0.7227 (14)	0.4007 (13)	0.6920 (7)	0.0275 (19)
H1A	0.670 (16)	0.320 (12)	0.682 (11)	0.041*
H1B	0.845 (8)	0.333 (14)	0.702 (10)	0.041*
N2	0.8921 (17)	0.592 (2)	0.8005 (10)	0.059 (4)
H2A	0.97 (2)	0.54 (2)	0.742 (7)	0.088*
H2B	0.94 (2)	0.67 (2)	0.823 (12)	0.088*
C1	0.6114 (17)	0.5081 (16)	0.7846 (8)	0.027 (2)
C2	0.6936 (17)	0.6094 (15)	0.8374 (8)	0.028 (2)
C3	0.5729 (17)	0.7188 (17)	0.9252 (9)	0.036 (3)
H3	0.6284	0.7802	0.9635	0.043*
C4	0.3739 (18)	0.7430 (18)	0.9601 (9)	0.035 (2)
C5	0.2911 (18)	0.6475 (18)	0.9083 (9)	0.038 (3)
H5	0.156	0.6596	0.9329	0.046*
C6	0.4078 (17)	0.5367 (16)	0.8218 (9)	0.032 (2)
H6	0.3496	0.476	0.7848	0.038*
C7	0.251 (2)	0.870 (2)	1.0495 (9)	0.044 (3)
N3	0.1570 (19)	0.9743 (18)	1.1197 (10)	0.057 (3)

O1	0.6745 (10)	0.3933 (10)	0.4281 (6)	0.0253 (15)
O2	0.9632 (11)	0.3531 (12)	0.4658 (6)	0.0303 (17)
C8	0.8580 (15)	0.2890 (15)	0.4258 (8)	0.0216 (19)
C9	0.9546 (18)	0.0876 (19)	0.3768 (11)	0.041 (3)
H9A	1.1009	0.0496	0.3529	0.062*
H9B	0.8942	0.0826	0.3188	0.062*
H9C	0.9315	-0.0005	0.4269	0.062*
O3	0.8051 (11)	0.7257 (11)	0.3578 (6)	0.0280 (16)
O4	0.4720 (12)	0.8727 (14)	0.3943 (7)	0.042 (2)
C10	0.6369 (18)	0.8350 (18)	0.3345 (9)	0.032 (2)
C11	0.642 (2)	0.916 (2)	0.2316 (10)	0.047 (3)
H11A	0.6577	1.0367	0.2353	0.07*
H11B	0.5144	0.9398	0.2104	0.07*
H11C	0.756	0.8238	0.1818	0.07*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pb1	0.0163 (3)	0.0203 (3)	0.0214 (3)	-0.00385 (19)	-0.00553 (16)	-0.00232 (16)
N1	0.028 (4)	0.015 (4)	0.030 (4)	-0.001 (4)	-0.002 (3)	-0.004 (3)
N2	0.040 (5)	0.073 (9)	0.057 (7)	-0.024 (5)	0.005 (5)	-0.034 (6)
C1	0.030 (4)	0.021 (5)	0.022 (4)	-0.003 (4)	-0.004 (3)	0.001 (3)
C2	0.032 (4)	0.020 (5)	0.027 (4)	-0.002 (4)	-0.012 (3)	0.004 (3)
C3	0.036 (5)	0.033 (6)	0.033 (5)	-0.006 (4)	-0.009 (4)	-0.004 (4)
C4	0.034 (5)	0.029 (6)	0.028 (5)	-0.001 (4)	-0.006 (4)	0.001 (4)
C5	0.033 (5)	0.041 (6)	0.029 (5)	-0.006 (5)	-0.001 (4)	-0.003 (4)
C6	0.033 (4)	0.023 (5)	0.032 (5)	-0.007 (4)	-0.001 (3)	-0.003 (4)
C7	0.044 (7)	0.051 (9)	0.029 (6)	-0.013 (7)	-0.002 (5)	-0.011 (6)
N3	0.062 (8)	0.050 (8)	0.047 (7)	-0.014 (7)	-0.002 (6)	-0.016 (6)
O1	0.017 (3)	0.013 (3)	0.042 (4)	0.000 (3)	-0.013 (3)	0.002 (3)
O2	0.017 (3)	0.034 (5)	0.036 (4)	-0.006 (3)	-0.006 (3)	-0.011 (3)
C8	0.021 (4)	0.020 (4)	0.022 (4)	-0.006 (3)	-0.007 (3)	0.005 (3)
C9	0.027 (5)	0.028 (5)	0.063 (8)	-0.002 (4)	-0.014 (5)	-0.019 (5)
O3	0.023 (3)	0.028 (4)	0.031 (4)	-0.008 (3)	-0.007 (3)	0.002 (3)
O4	0.023 (3)	0.048 (6)	0.043 (4)	-0.001 (4)	-0.012 (3)	0.011 (4)
C10	0.029 (4)	0.036 (6)	0.033 (5)	-0.014 (4)	-0.012 (3)	-0.004 (4)
C11	0.060 (8)	0.042 (8)	0.043 (5)	-0.021 (6)	-0.022 (5)	0.011 (5)

Geometric parameters (Å, °)

Pb1—O3	2.431 (7)	C4—C7	1.449 (17)
Pb1—O2	2.485 (8)	C5—C6	1.358 (15)
Pb1—O1	2.505 (7)	C5—H5	0.95
Pb1—O2 ⁱ	2.635 (7)	C6—H6	0.95
Pb1—O4	2.667 (8)	C7—N3	1.146 (16)
Pb1—O1 ⁱⁱ	2.727 (7)	O1—C8	1.255 (12)
Pb1—N1	2.906 (10)	O1—Pb1 ⁱⁱ	2.727 (7)
N1—C1	1.403 (13)	O2—C8	1.271 (12)

N1—H1A	0.88 (2)	O2—Pb1 ⁱ	2.635 (7)
N1—H1B	0.87 (2)	C8—C9	1.505 (15)
N2—C2	1.398 (15)	C9—H9A	0.98
N2—H2A	0.88 (2)	C9—H9B	0.98
N2—H2B	0.88 (2)	C9—H9C	0.98
C1—C6	1.409 (15)	O3—C10	1.279 (13)
C1—C2	1.430 (15)	O4—C10	1.239 (14)
C2—C3	1.373 (15)	C10—C11	1.500 (18)
C3—C4	1.382 (17)	C11—H11A	0.98
C3—H3	0.95	C11—H11B	0.98
C4—C5	1.393 (17)	C11—H11C	0.98
O3—Pb1—O2	77.0 (3)	C4—C3—H3	118.7
O3—Pb1—O1	78.6 (3)	C3—C4—C5	120.0 (11)
O2—Pb1—O1	52.0 (2)	C3—C4—C7	119.9 (12)
O3—Pb1—O2 ⁱ	75.3 (2)	C5—C4—C7	120.0 (11)
O2—Pb1—O2 ⁱ	64.3 (3)	C6—C5—C4	118.5 (11)
O1—Pb1—O2 ⁱ	114.8 (2)	C6—C5—H5	120.7
O3—Pb1—O4	50.8 (2)	C4—C5—H5	120.7
O2—Pb1—O4	117.2 (3)	C5—C6—C1	122.9 (11)
O1—Pb1—O4	82.3 (3)	C5—C6—H6	118.6
O2 ⁱ —Pb1—O4	119.7 (3)	C1—C6—H6	118.6
O3—Pb1—O1 ⁱⁱ	119.9 (2)	N3—C7—C4	177.7 (16)
O2—Pb1—O1 ⁱⁱ	108.4 (2)	C8—O1—Pb1	93.8 (6)
O1—Pb1—O1 ⁱⁱ	63.9 (3)	C8—O1—Pb1 ⁱⁱ	134.3 (6)
O2 ⁱ —Pb1—O1 ⁱⁱ	162.4 (3)	Pb1—O1—Pb1 ⁱⁱ	116.1 (3)
O4—Pb1—O1 ⁱⁱ	77.9 (3)	C8—O2—Pb1	94.3 (6)
O3—Pb1—N1	147.4 (2)	C8—O2—Pb1 ⁱ	147.4 (7)
O2—Pb1—N1	70.6 (3)	Pb1—O2—Pb1 ⁱ	115.7 (3)
O1—Pb1—N1	83.9 (3)	O1—C8—O2	119.8 (9)
O2 ⁱ —Pb1—N1	87.6 (3)	O1—C8—C9	120.6 (9)
O4—Pb1—N1	152.5 (3)	O2—C8—C9	119.6 (9)
O1 ⁱⁱ —Pb1—N1	74.7 (2)	C8—C9—H9A	109.5
C1—N1—Pb1	107.9 (6)	C8—C9—H9B	109.5
C1—N1—H1A	108 (9)	H9A—C9—H9B	109.5
Pb1—N1—H1A	109 (9)	C8—C9—H9C	109.5
C1—N1—H1B	106 (9)	H9A—C9—H9C	109.5
Pb1—N1—H1B	119 (9)	H9B—C9—H9C	109.5
H1A—N1—H1B	107 (5)	C10—O3—Pb1	98.9 (7)
C2—N2—H2A	128 (10)	C10—O4—Pb1	88.8 (7)
C2—N2—H2B	123 (10)	O4—C10—O3	121.5 (11)
H2A—N2—H2B	106 (5)	O4—C10—C11	119.8 (11)
N1—C1—C6	120.8 (10)	O3—C10—C11	118.6 (11)
N1—C1—C2	120.7 (10)	C10—C11—H11A	109.5
C6—C1—C2	118.1 (10)	C10—C11—H11B	109.5
C3—C2—N2	122.3 (11)	H11A—C11—H11B	109.5
C3—C2—C1	117.7 (10)	C10—C11—H11C	109.5
N2—C2—C1	120.0 (10)	H11A—C11—H11C	109.5

C2—C3—C4	122.7 (11)	H11B—C11—H11C	109.5
C2—C3—H3	118.7		
Pb1—N1—C1—C6	91.4 (11)	C2—C1—C6—C5	-3.7 (18)
Pb1—N1—C1—C2	-81.2 (10)	Pb1—O1—C8—O2	-3.0 (10)
N1—C1—C2—C3	177.1 (10)	Pb1 ⁱⁱ —O1—C8—O2	-135.8 (8)
C6—C1—C2—C3	4.2 (16)	Pb1—O1—C8—C9	176.5 (10)
N1—C1—C2—N2	-4.3 (17)	Pb1 ⁱⁱ —O1—C8—C9	43.6 (15)
C6—C1—C2—N2	-177.1 (12)	Pb1—O2—C8—O1	3.0 (10)
N2—C2—C3—C4	177.4 (13)	Pb1 ⁱ —O2—C8—O1	-154.9 (9)
C1—C2—C3—C4	-4.0 (18)	Pb1—O2—C8—C9	-176.4 (10)
C2—C3—C4—C5	3.0 (19)	Pb1 ⁱ —O2—C8—C9	26 (2)
C2—C3—C4—C7	-175.9 (12)	Pb1—O4—C10—O3	2.1 (11)
C3—C4—C5—C6	-2.2 (19)	Pb1—O4—C10—C11	-178.9 (11)
C7—C4—C5—C6	176.7 (12)	Pb1—O3—C10—O4	-2.3 (13)
C4—C5—C6—C1	2.7 (19)	Pb1—O3—C10—C11	178.7 (10)
N1—C1—C6—C5	-176.6 (11)		

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

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—H1A...O4 ⁱⁱ	0.88 (2)	2.46 (4)	3.310 (14)	164 (12)
N1—H1B...O3 ⁱ	0.87 (2)	2.40 (8)	3.139 (12)	143 (11)
N2—H2A...O3 ⁱ	0.88 (2)	2.25 (9)	3.044 (14)	150 (16)
N2—H2B...N3 ⁱⁱⁱ	0.88 (2)	2.62 (11)	3.355 (18)	142 (14)

Symmetry codes: (i) $-x+2, -y+1, -z+1$; (ii) $-x+1, -y+1, -z+1$; (iii) $-x+1, -y+2, -z+2$.