

# Poly[( $\mu_5$ -2,2'-bipyridine-5,5'-dicarboxylato)lead(II)]

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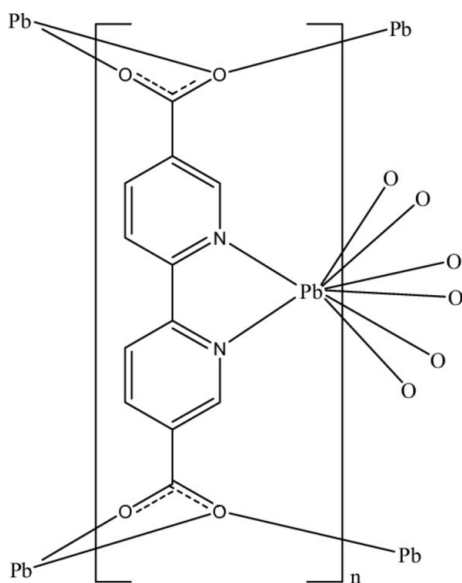
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Key indicators: single-crystal X-ray study;  $T = 100$  K; mean  $\sigma(\text{C}-\text{C}) = 0.009$  Å;  $R$  factor = 0.019;  $wR$  factor = 0.055; data-to-parameter ratio = 17.1.

In the title polymeric compound,  $[\text{Pb}(\text{C}_{12}\text{H}_6\text{N}_2\text{O}_4)]_n$ , the  $\text{Pb}^{\text{II}}$  cation, located on a mirror plane, is  $N,N'$ -chelated by a 2,2'-bipyridine-5,5'-dicarboxylate (bpdc) anion and is further coordinated by six O atoms from four carboxyl groups of bpdc anions in an irregular  $\text{N}_2\text{O}_6$  geometry. The carboxylate groups bridge the  $\text{Pb}^{\text{II}}$  cations, forming a three-dimensional polymeric structure. The carboxylate group is twisted away from the attached pyridine ring by  $11.4$  (3)°.

## Related literature

For background to niacin, see: Krishnamachari (1974) and to  $N,N$ -diethylnicotinamide, see: Bigoli *et al.* (1972). For related structures, see: Greenaway *et al.* (1984); Hökelek & Necefoğlu (1996); Hökelek *et al.* (2009*a,b,c,d*, 2010*a,b*, 2011).



## Experimental

### Crystal data

$[\text{Pb}(\text{C}_{12}\text{H}_6\text{N}_2\text{O}_4)]$   
 $M_r = 449.39$   
 Orthorhombic,  $Pmn2_1$   
 $a = 13.6224$  (3) Å  
 $b = 4.1923$  (2) Å  
 $c = 10.2180$  (3) Å  
 $V = 583.54$  (3) Å<sup>3</sup>  
 $Z = 2$   
 Mo  $K\alpha$  radiation  
 $\mu = 14.47$  mm<sup>-1</sup>  
 $T = 100$  K  
 $0.32 \times 0.18 \times 0.10$  mm

### Data collection

Bruker Kappa APEXII CCD area-detector diffractometer  
 Absorption correction: multi-scan (SADABS; Bruker, 2005)  
 $T_{\text{min}} = 0.055$ ,  $T_{\text{max}} = 0.235$   
 9729 measured reflections  
 1542 independent reflections  
 1511 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.036$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.019$   
 $wR(F^2) = 0.055$   
 $S = 1.24$   
 1542 reflections  
 90 parameters  
 1 restraint  
 H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 1.53$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.75$  e Å<sup>-3</sup>  
 Absolute structure: Flack (1983),  
 728 Friedel pairs  
 Flack parameter: 0.497 (14)

**Table 1**

Selected bond lengths (Å).

|                     |           |                      |           |
|---------------------|-----------|----------------------|-----------|
| Pb1—O1 <sup>i</sup> | 2.819 (5) | Pb1—O2 <sup>ii</sup> | 2.860 (5) |
| Pb1—O2 <sup>i</sup> | 2.383 (5) | Pb1—N1               | 2.669 (5) |

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

Data collection: APEX2 (Bruker, 2007); cell refinement: SAINT (Bruker, 2007); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997); software used to prepare material for publication: WinGX publication routines (Farrugia, 1999) and PLATON (Spek, 2009).

The authors are indebted to Anadolu University and the Medicinal Plants and Medicine Research Centre of Anadolu University, Eskişehir, Turkey, for the use of X-ray diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5606).

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## supporting information

*Acta Cryst.* (2012). E68, m1196–m1197 [doi:10.1107/S1600536812035647]

**Poly[( $\mu_5$ -2,2'-bipyridine-5,5'-dicarboxylato)lead(II)]****Mustafa Sertçelik, Nagihan Çaylak Delibaş, Sabri Çevik, Hacali Necefoğlu and Tuncer Hökelek****S1. Comment**

As a part of our ongoing investigation on transition metal complexes of nicotinamide (NA), one form of niacin (Krishnamachari, 1974), and/or the nicotinic acid derivative *N,N*-diethylnicotinamide (DENA), an important respiratory stimulant (Bigoli *et al.*, 1972) the title compound was synthesized and its crystal structure is reported herein. In fact, in the synthesis we aimed to obtain a mixed complex of lead with nicotinic acid and DENA. But, the nicotinic acid molecules have been interacted to form 2-2'-bipyridine-5-5'-dicarboxylic (bpdc) acid in the hydrothermal synthesis media.

In the crystal structure of the polymeric title compound, (I), the Pb<sup>II</sup> ion is chelated by the O atoms of the carboxylate groups and the nitrogen atoms from 2-2'-bipyridine-5,5'-dicarboxylato (bpdc) ligands (Fig. 1); the symmetry related Pb<sup>II</sup> ions are bridged through the O atoms of the carboxyl groups and the nitrogen atoms of the bpdc ligands to form a 3-D polymeric structure (Fig. 2), in which the Pb<sup>II</sup> ion is in an irregular eight-coordination geometry (Fig. 1).

The Pb–O bond lengths are ranged from 2.383 (5) and 2.860 (5) Å (Table 1) and the Pb atom is displaced out of the least-square plane of the carboxylate group (O1/C6/O2) by -1.5813 (1) Å. The Pb1...Pb1b distance [symmetry code: (b) x, 1 + y, z] is 4.1923 (3) Å (Fig. 1). In (I), the O1–Pb1–O2 and N1–Pb1–N1<sup>i</sup> [symmetry code: (i) -x, y, z] angles are 50.4 (2) and 52.7 (3) °, respectively.

The corresponding O–M–O (where M is a metal) angles are 51.10 (15)° and 51.95 (16)° in {[Pb(PEB)<sub>2</sub>(NA)].H<sub>2</sub>O}<sub>n</sub> (Hökelek *et al.*, 2011), 51.09 (6)° and 51.71 (5)° in [Pb(PMB)<sub>2</sub>(NA)]<sub>n</sub> (Hökelek *et al.*, 2010a), 55.96 (4)° and 53.78 (4)° in [Cd<sub>2</sub>(DMAB)<sub>4</sub>(NA)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>] (Hökelek *et al.*, 2010b), 52.91 (4)° and 53.96 (4)° in [Cd(FB)<sub>2</sub>(INA)<sub>2</sub>(H<sub>2</sub>O)].H<sub>2</sub>O (Hökelek *et al.*, 2009a), 60.70 (4)° in [Co(DMAB)<sub>2</sub>(INA)(H<sub>2</sub>O)<sub>2</sub>] (Hökelek *et al.*, 2009b), 58.45 (9)° in [Mn(DMAB)<sub>2</sub>(INA)(H<sub>2</sub>O)<sub>2</sub>] (Hökelek *et al.*, 2009c), 60.03 (6)° in [Zn(MAB)<sub>2</sub>(INA)<sub>2</sub>].H<sub>2</sub>O (Hökelek *et al.*, 2009d), 58.3 (3)° in [Zn<sub>2</sub>(DENA)<sub>2</sub>(HB)<sub>4</sub>].2H<sub>2</sub>O (Hökelek & Necefoğlu, 1996) [where NA, INA, DENA, HB, FB, MAB, PMB, PEB and DMAB are nicotinamide, isonicotinamide, *N,N*-diethylnicotinamide, 4-hydroxybenzoate, 4-formylbenzoate, 4-methylaminobenzoate, 4-methylbenzoate, 4-ethylbenzoate and 4-dimethylaminobenzoate, respectively] and 55.2 (1)° in [Cu(Asp)<sub>2</sub>(py)<sub>2</sub>] (where Asp is acetylsalicylate and py is pyridine) (Greenaway *et al.*, 1984).

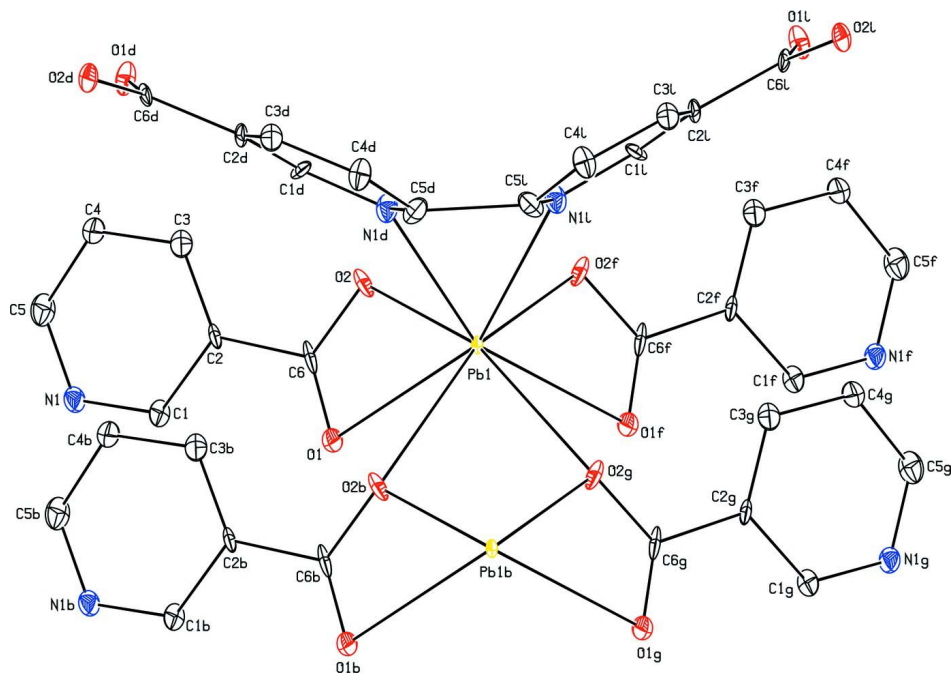
The dihedral angle between the planar carboxylate group and the adjacent pyridine ring A (N1/C1–C5) is 11.44 (31)°.

**S2. Experimental**

The title compound was obtained after leaving a mixture of Pb(NO<sub>3</sub>)<sub>2</sub> (0.33 g, 1 mmol), nicotinic acid, (NA), (0.24 g, 2 mmol), diethylnicotinamide, (DENA), (0.35 g, 2 mmol) and distilled water (5 ml) in a Teflon-lined autoclave at 433 K for 41 h.

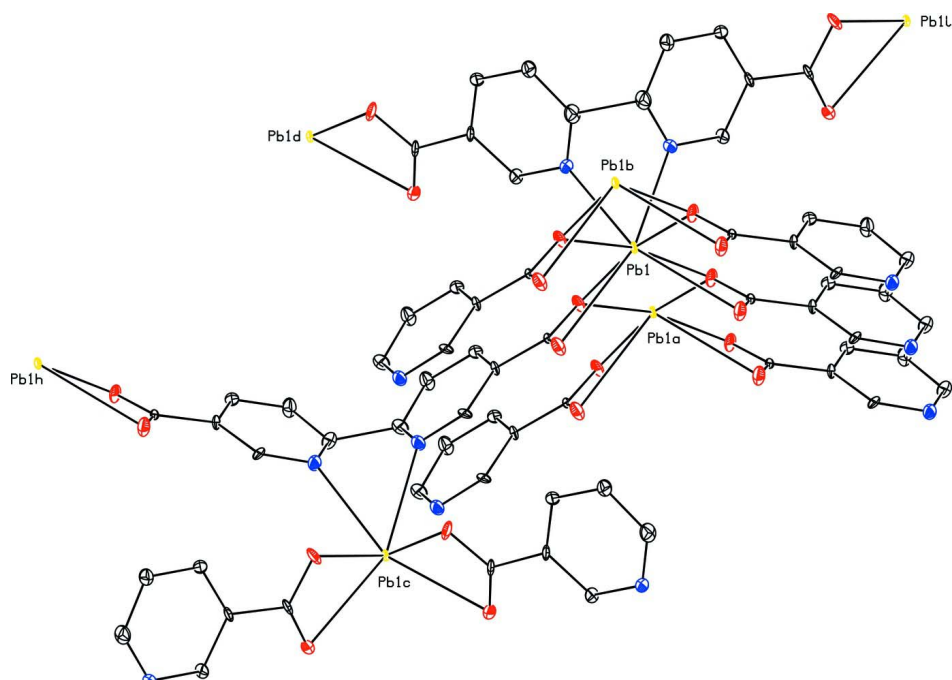
### S3. Refinement

The C-bound H-atoms were positioned geometrically with C—H = 0.95 Å, for aromatic H-atoms, and constrained to ride on their parent atoms, with  $U_{\text{iso}}(\text{H}) = 1.2 \times U_{\text{eq}}(\text{C})$ . The highest residual electron density was found 0.89 Å from Pb1 and the deepest hole 0.93 Å from Pb1.



**Figure 1**

The molecular structure of the title compound with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Atoms are generated by the symmetry operators: (b)  $x, 1 + y, z$ , (d)  $1/2 - x, 1 - y, 1/2 + z$ , (f)  $-x, y, z$ , (g)  $-x, 1 + y, z$ , (l)  $-1/2 + x, 1 - y, 1/2 + z$ .


**Figure 2**

The polymeric structure. Pb atoms are generated by the symmetry operators: (a)  $x, -1 + y, z$ , (b)  $x, 1 + y, z$ , (c)  $1/2 - x, 1 - y, -1/2 + z$ , (d)  $1/2 - x, 1 - y, 1/2 + z$ , (h)  $1 - x, y, z$ , (l)  $-1/2 + x, 1 - y, 1/2 + z$ .

**Poly[( $\mu_5$ -2,2'-bipyridine-5,5'-dicarboxylato)lead(II)]**
*Crystal data*

[Pb(C<sub>12</sub>H<sub>6</sub>N<sub>2</sub>O<sub>4</sub>)]

$M_r = 449.39$

Orthorhombic,  $Pmn2_1$

Hall symbol: P 2ac -2

$a = 13.6224 (3) \text{ \AA}$

$b = 4.1923 (2) \text{ \AA}$

$c = 10.2180 (3) \text{ \AA}$

$V = 583.54 (3) \text{ \AA}^3$

$Z = 2$

$F(000) = 412$

$D_x = 2.558 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation,  $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 8915 reflections

$\theta = 2.5\text{--}28.5^\circ$

$\mu = 14.47 \text{ mm}^{-1}$

$T = 100 \text{ K}$

Prism, colorless

$0.32 \times 0.18 \times 0.10 \text{ mm}$

*Data collection*

Bruker Kappa APEXII CCD area-detector  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2005)

$T_{\min} = 0.055$ ,  $T_{\max} = 0.235$

9729 measured reflections

1542 independent reflections

1511 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.036$

$\theta_{\max} = 28.5^\circ$ ,  $\theta_{\min} = 2.5^\circ$

$h = -18 \rightarrow 17$

$k = -5 \rightarrow 5$

$l = -13 \rightarrow 13$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.019$

$wR(F^2) = 0.055$

$S = 1.24$

1542 reflections

90 parameters  
 1 restraint  
 Primary atom site location: structure-invariant  
 direct methods  
 Secondary atom site location: difference Fourier  
 map  
 Hydrogen site location: inferred from  
 neighbouring sites  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0212P)^2 + 3.443P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 1.53 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.75 \text{ e } \text{\AA}^{-3}$   
 Extinction correction: *SHELXL*,  
 $F_c^* = kFc[1 + 0.001x Fc^2 \lambda^3 / \sin(2\theta)]^{-1/4}$   
 Extinction coefficient: 0.0097 (6)  
 Absolute structure: Flack (1983), 728 Friedel  
 pairs  
 Absolute structure parameter: 0.497 (14)

*Special details*

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

**Refinement.** Refinement of  $F^2$  against ALL reflections. The weighted R-factor wR and goodness of fit S are based on  $F^2$ , conventional R-factors R are based on F, with F set to zero for negative  $F^2$ . The threshold expression of  $F^2 > 2\sigma(F^2)$  is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on  $F^2$  are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

|     | x          | y           | z          | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|------------|-------------|------------|----------------------------------|
| Pb1 | 0.0000     | 0.81379 (5) | 0.6243     | 0.00544 (10)                     |
| O1  | 0.3476 (4) | 0.2412 (13) | 0.9391 (5) | 0.0145 (9)                       |
| O2  | 0.3886 (3) | 0.6151 (12) | 1.0873 (4) | 0.0136 (9)                       |
| N1  | 0.0870 (4) | 0.7262 (14) | 0.8556 (5) | 0.0134 (11)                      |
| C1  | 0.1730 (4) | 0.5765 (15) | 0.8787 (5) | 0.0099 (11)                      |
| H1  | 0.1929     | 0.4075      | 0.8227     | 0.012*                           |
| C2  | 0.2338 (4) | 0.6648 (14) | 0.9835 (6) | 0.0091 (11)                      |
| C3  | 0.2044 (5) | 0.9052 (17) | 1.0680 (6) | 0.0138 (11)                      |
| H3  | 0.2446     | 0.9677      | 1.1395     | 0.017*                           |
| C4  | 0.1157 (5) | 1.0506 (17) | 1.0457 (6) | 0.0149 (12)                      |
| H4  | 0.0926     | 1.2117      | 1.1034     | 0.018*                           |
| C5  | 0.0596 (5) | 0.9597 (17) | 0.9373 (6) | 0.0160 (12)                      |
| C6  | 0.3296 (4) | 0.4896 (16) | 1.0016 (5) | 0.0127 (12)                      |

*Atomic displacement parameters ( $\text{\AA}^2$ )*

|     | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$     | $U^{13}$     | $U^{23}$     |
|-----|--------------|--------------|--------------|--------------|--------------|--------------|
| Pb1 | 0.00259 (13) | 0.00704 (13) | 0.00668 (12) | 0.000        | 0.000        | 0.00030 (19) |
| O1  | 0.009 (2)    | 0.022 (2)    | 0.013 (2)    | 0.0027 (19)  | -0.0030 (18) | -0.0058 (18) |
| O2  | 0.007 (2)    | 0.018 (2)    | 0.015 (2)    | -0.0035 (17) | -0.0056 (13) | -0.0002 (15) |
| N1  | 0.009 (3)    | 0.022 (3)    | 0.009 (2)    | 0.000 (2)    | -0.001 (2)   | -0.002 (2)   |
| C1  | 0.009 (3)    | 0.013 (3)    | 0.008 (2)    | 0.004 (2)    | -0.0023 (19) | 0.004 (2)    |
| C2  | 0.003 (3)    | 0.014 (3)    | 0.011 (3)    | -0.001 (2)   | -0.002 (2)   | 0.0008 (19)  |
| C3  | 0.010 (3)    | 0.025 (3)    | 0.006 (2)    | 0.001 (3)    | 0.000 (2)    | 0.000 (3)    |
| C4  | 0.010 (3)    | 0.018 (3)    | 0.017 (3)    | 0.003 (2)    | -0.001 (2)   | -0.004 (2)   |

|    |           |           |           |            |              |           |
|----|-----------|-----------|-----------|------------|--------------|-----------|
| C5 | 0.012 (3) | 0.020 (3) | 0.016 (3) | 0.005 (2)  | -0.002 (2)   | 0.004 (2) |
| C6 | 0.003 (2) | 0.029 (4) | 0.006 (2) | -0.004 (2) | -0.0017 (18) | 0.006 (2) |

*Geometric parameters (Å, °)*

|  |             |                           |            |
|--|-------------|---------------------------|------------|
| Pb1—O1 <sup>i</sup>                    | 2.819 (5)   | C1—C2                     | 1.403 (8)  |
| Pb1—O2 <sup>i</sup>                    | 2.383 (5)   | C1—H1                     | 0.9500     |
| Pb1—O2 <sup>ii</sup>                   | 2.383 (5)   | C2—C3                     | 1.386 (9)  |
| Pb1—O2 <sup>iii</sup>                  | 2.860 (5)   | C2—C6                     | 1.509 (8)  |
| Pb1—N1                                 | 2.669 (5)   | C3—C4                     | 1.373 (9)  |
| Pb1—N1 <sup>iv</sup>                   | 2.669 (5)   | C3—H3                     | 0.9500     |
| O1—C6                                  | 1.245 (8)   | C4—C5                     | 1.398 (9)  |
| O2—Pb1 <sup>v</sup>                    | 2.383 (5)   | C4—H4                     | 0.9500     |
| N1—C1                                  | 1.350 (8)   | C5—C5 <sup>iv</sup>       | 1.623 (13) |
| N1—C5                                  | 1.340 (9)   | C6—O2                     | 1.300 (7)  |
| O2 <sup>ii</sup> —Pb1—O2 <sup>i</sup>  | 79.1 (2)    | C3—C2—C1                  | 119.8 (6)  |
| O2 <sup>i</sup> —Pb1—N1 <sup>iv</sup>  | 108.61 (16) | C3—C2—C6                  | 121.8 (5)  |
| O2 <sup>ii</sup> —Pb1—N1               | 108.61 (17) | C2—C3—H3                  | 120.9      |
| O2 <sup>i</sup> —Pb1—N1                | 75.73 (16)  | C4—C3—C2                  | 118.3 (6)  |
| O2 <sup>ii</sup> —Pb1—N1 <sup>iv</sup> | 75.73 (16)  | C4—C3—H3                  | 120.9      |
| N1—Pb1—N1 <sup>iv</sup>                | 52.7 (3)    | C3—C4—C5                  | 119.4 (6)  |
| C6—O2—Pb1 <sup>v</sup>                 | 101.2 (4)   | C3—C4—H4                  | 120.3      |
| C1—N1—Pb1                              | 127.2 (4)   | C5—C4—H4                  | 120.3      |
| C5—N1—Pb1                              | 109.1 (4)   | N1—C5—C4                  | 122.7 (6)  |
| C5—N1—C1                               | 118.2 (6)   | N1—C5—C5 <sup>iv</sup>    | 106.2 (4)  |
| N1—C1—C2                               | 121.5 (6)   | C4—C5—C5 <sup>iv</sup>    | 123.1 (4)  |
| N1—C1—H1                               | 119.2       | O1—C6—O2                  | 124.2 (6)  |
| C2—C1—H1                               | 119.2       | O1—C6—C2                  | 120.9 (5)  |
| C1—C2—C6                               | 118.4 (5)   | O2—C6—C2                  | 114.8 (6)  |
| O2 <sup>i</sup> —Pb1—N1—C1             | 19.7 (5)    | N1—C1—C2—C6               | -179.0 (5) |
| O2 <sup>ii</sup> —Pb1—N1—C1            | 92.8 (5)    | C1—C2—C3—C4               | -0.1 (10)  |
| O2 <sup>i</sup> —Pb1—N1—C5             | 172.7 (5)   | C6—C2—C3—C4               | -179.3 (6) |
| O2 <sup>ii</sup> —Pb1—N1—C5            | -114.2 (5)  | C1—C2—C6—O1               | -11.3 (9)  |
| N1 <sup>iv</sup> —Pb1—N1—C1            | 147.1 (5)   | C1—C2—C6—O2               | 169.9 (5)  |
| N1 <sup>iv</sup> —Pb1—N1—C5            | -59.9 (5)   | C3—C2—C6—O1               | 167.9 (6)  |
| Pb1—N1—C1—C2                           | 149.5 (5)   | C3—C2—C6—O2               | -10.9 (8)  |
| C5—N1—C1—C2                            | -1.3 (9)    | C2—C3—C4—C5               | -1.9 (10)  |
| Pb1—N1—C5—C4                           | -156.5 (5)  | C3—C4—C5—N1               | 2.5 (11)   |
| Pb1—N1—C5—C5 <sup>iv</sup>             | 53.8 (3)    | C3—C4—C5—C5 <sup>iv</sup> | 147.0 (5)  |
| C1—N1—C5—C4                            | -0.8 (10)   | O1—C6—O2—Pb1 <sup>v</sup> | -14.7 (7)  |
| C1—N1—C5—C5 <sup>iv</sup>              | -150.5 (5)  | C2—C6—O2—Pb1 <sup>v</sup> | 164.0 (4)  |
| N1—C1—C2—C3                            | 1.8 (9)     |                           |            |

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