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# $\mu$-4,4′-Bipyridine-$\kappa^2N$:$N'$-bis[bis(2-chlorobenzoato-$\kappa^2O,O'$)lead(II)]

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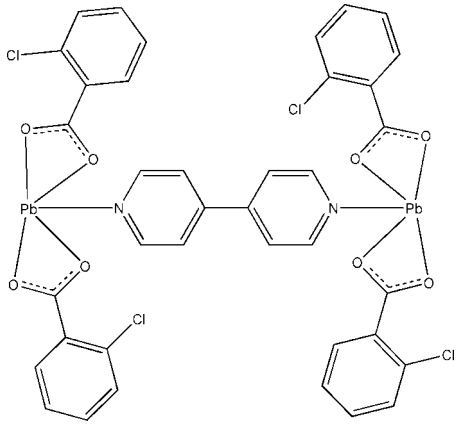
Key indicators: single-crystal X-ray study; $T$ = 296 K; mean $\sigma$(C–C) = 0.010 Å; $R$ factor = 0.026; $wR$ factor = 0.081; data-to-parameter ratio = 13.9.

In the title dinuclear complex, $[Pb_2(C_7H_4ClO_2)_4(C_{10}H_8N_2)]$, the $Pb^{II}$ atom is five-coordinated by four carboxylate O atoms from two 2-chlorobenzoate ligands and one N atom from a bridging 4,4′-bipyridine (4,4′-bpy) ligand, displaying a hemi-directed coordination. The 4,4′-bpy ligand has an inversion center at the mid-point of the central C—C bond. The empty side of the metal ion is capped by two carboxylate O atoms from a neighboring molecule, with weak Pb···O contacts [Pb···O = 3.069 (2) and 3.071 (3) Å]. The crystal structure is stabilized by C—H···O hydrogen bonds and $\pi$–$\pi$ stacking interactions between the benzene and pyridine rings [centroid–centroid distance = 3.749 (3) Å].

## Related literature

For general background to 2-chlorobenzoate complexes, see: Gomez & Corbella (2009); Motokawa *et al.* (2010). For general background to 4,4′-bipyridine complexes, see: Biradha *et al.* (2006). For hemi- and holo-directed geometries of lead(II) complexes, see: Shimoni-Livny *et al.* (1998).




## Experimental

### Crystal data

$[Pb_2(C_7H_4ClO_2)_4(C_{10}H_8N_2)]$
$M_r$ = 1192.79
Orthorhombic, *Pbca*
$a$ = 21.9370 (13) Å
$b$ = 7.4569 (5) Å
$c$ = 23.2379 (14) Å
$V$ = 3801.3 (4) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 9.18 mm$^{-1}$
$T$ = 296 K
0.30 × 0.28 × 0.22 mm

### Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.092, $T_{max}$ = 0.145
15375 measured reflections
3398 independent reflections
2712 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.032

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.081
$S$ = 1.09
3398 reflections
244 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.84 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.53 e Å$^{-3}$

**Table 1**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C15—H15···O1$^{i}$ | 0.93 | 2.40 | 3.239 (7) | 150 |
| C16—H16···O2$^{ii}$ | 0.93 | 2.55 | 3.361 (6) | 146 |
| C19—H19···O4 | 0.93 | 2.39 | 3.026 (5) | 125 |

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

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: *SHELXTL* (Sheldrick, 2008) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXTL*.

The authors acknowledge Lanzhou Jiaotong University for supporting this work.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HY2436).

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# supporting information

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## *μ*-4,4′-Bipyridine-$\kappa^2N$:*N*′-bis[bis(2-chlorobenzoato-$\kappa^2O$,*O*′)lead(II)]

**Tian-Jun Feng**

### S1. Comment

In the structural investigation of 2-chlorobenzoate complexes, it has been found that 2-chlorobenzoic acid (2-Hcbz) functions as a multidentate ligand with versatile binding and coordination modes (Gomez & Corbella, 2009; Motokawa *et al.*, 2010). As is well known, 4,4′-bipyridine (4,4′-bpy) ligand can act in bidentate bridging or monodentate terminal modes (Biradha *et al.*, 2006). In this paper, we report the crystal structure of the title compound, a new Pb(II) complex obtained by the reaction of 2-Hcbz, 4,4′-bpy and lead(II) acetate in an alkaline aqueous solution.

As depicted in Fig. 1, the $Pb^{II}$ atom is coordinated by four O atoms from two 2-cbz ligands and one N atom from a *μ*-4,4′-bpy ligand. The coordination environment of the $Pb^{II}$ atom is hemidirected. The empty sapce around the metal ion is filled by the stereochemically active $6S^2$ electron pair (Shimoni-Livny *et al.*, 1998) and two Pb···O contacts [Pb1···O1$^{i}$ = 3.069 (2), Pb1···O3$^{i}$ = 3.071 (3) Å. Symmetry code: (i) 1/2-*x*, 1/2+*y*, *z*]. The *μ*-4,4′-bpy ligand, having an inversion center at the mid-point of the central C—C bond, bridges two Pb atoms, with a Pb1···Pb1$^{ii}$ distance of 12.073 (3) Å [symmetry code: (ii) -*x*, 3-*y*, -*z*]. The interactions of the structural components are governed by Pb···O contacts, C—H···O hydrogen bonds (Table 1) and *π*–*π* stacking interactions. The centroid–centroid distance between the benzene ring of a 2-cbz ligand and the pyridine ring of a 4,4′-bpy at (*x*, -1+*y*, *z*) is 3.749 (3) Å (Fig. 2).

### S2. Experimental

A mixture of lead acetate (1 mmol, 0.325 g), 2-Hcbz (1 mmol, 0.156 g), 4,4′-bpy (1 mmol, 0.156 g), NaOH (1.5 mmol, 0.06 g) and $H_2O$ (12 ml) was placed in a 23 ml Teflon-lined reactor, which was heated at 433 K for three days and then cooled to room temperature at a rate of 10 K h$^{-1}$. The colorless crystals obtained were washed with water and dried in air.

### S3. Refinement

H atoms were placed at calculated positions and treated as riding atoms, with C—H = 0.93 Å and with $U_{iso}$(H) = 1.2$U_{eq}$(C). The highest peak in final difference map is located 0.85 Å from Pb1 and the deepest hole is located 0.40 Å from Cl2.

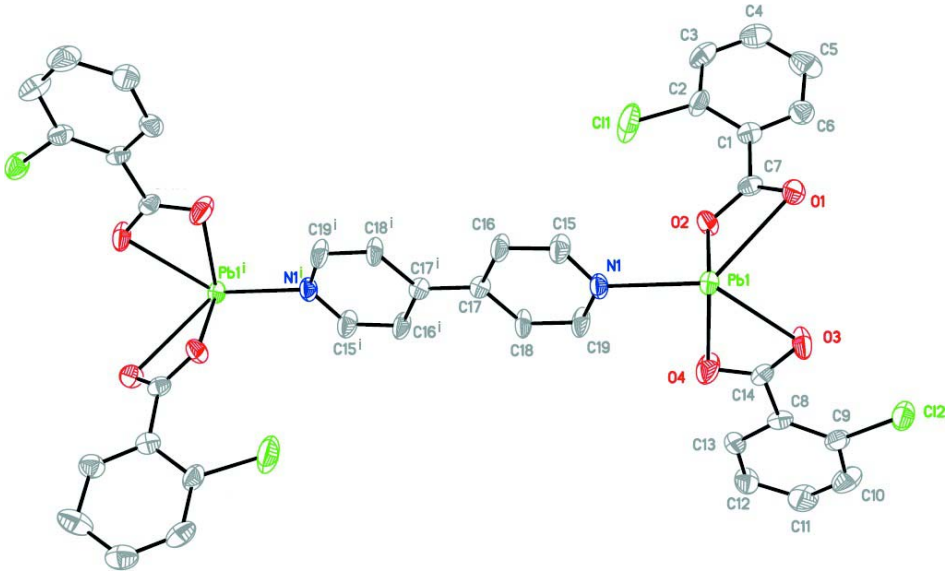


**Figure 1**

The molecular structure of the title compound, showing 30% probability displacement ellipsoids. H atoms were omitted for clarity. [Symmetry code: (i) -x, 3-y, -z.]

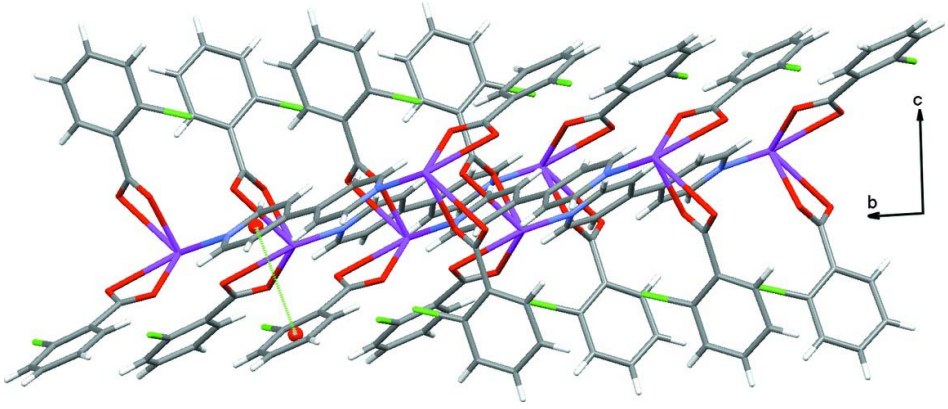


**Figure 2**

A packing view of the title compound. $\pi$–$\pi$ interactions are shown as dashed lines.

### *μ*-4,4′-Bipyridine-$\kappa^2N$:*N*′-bis[bis(2-chlorobenzoato-$\kappa^2O$,*O*′)lead(II)]

*Crystal data*

$[Pb_2(C_7H_4ClO_2)_4(C_{10}H_8N_2)]$
$M_r$ = 1192.79
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
*a* = 21.9370 (13) Å
*b* = 7.4569 (5) Å
*c* = 23.2379 (14) Å
*V* = 3801.3 (4) Å$^3$
*Z* = 4
*F*(000) = 2248
$D_x$ = 2.084 Mg m$^{-3}$
Mo *Kα* radiation, $\lambda$ = 0.71073 Å
Cell parameters from 5300 reflections
$\theta$ = 1.3–28.0°
$\mu$ = 9.18 mm$^{-1}$
*T* = 296 K
Block, colorless
0.30 × 0.28 × 0.22 mm

*Data collection*

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.092, $T_{max}$ = 0.145
15375 measured reflections
3398 independent reflections
2712 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.032
$\theta_{max}$ = 25.2°, $\theta_{min}$ = 1.8°
$h$ = −25→26
$k$ = −8→8
$l$ = −27→19

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.081
$S$ = 1.09
3398 reflections
244 parameters
0 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0403P)^2 + 6.2959P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.84 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.53 e Å$^{-3}$

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.1794 (3) | 0.7076 (9) | −0.1258 (3) | 0.0427 (15) |
| C2 | 0.1528 (3) | 0.8251 (10) | −0.1646 (3) | 0.0552 (18) |
| C3 | 0.1478 (4) | 0.7786 (12) | −0.2219 (3) | 0.068 (2) |
| H3 | 0.1289 | 0.8564 | −0.2476 | 0.082* |
| C4 | 0.1705 (4) | 0.6187 (13) | −0.2411 (3) | 0.070 (2) |
| H4 | 0.1692 | 0.5922 | −0.2802 | 0.084* |
| C5 | 0.1949 (4) | 0.4974 (16) | −0.2036 (4) | 0.082 (3) |
| H5 | 0.2071 | 0.3840 | −0.2157 | 0.098* |
| C6 | 0.2009 (3) | 0.5513 (11) | −0.1462 (3) | 0.0555 (19) |
| H6 | 0.2208 | 0.4748 | −0.1208 | 0.067* |
| C7 | 0.1912 (3) | 0.7545 (9) | −0.0634 (3) | 0.0405 (14) |
| C8 | 0.0956 (3) | 0.5487 (8) | 0.1402 (2) | 0.0389 (14) |
| C9 | 0.1121 (3) | 0.3828 (10) | 0.1618 (3) | 0.0517 (17) |
| C10 | 0.0715 (4) | 0.2756 (12) | 0.1901 (3) | 0.075 (2) |
| H10 | 0.0851 | 0.1680 | 0.2058 | 0.090* |
| C11 | 0.0125 (4) | 0.3208 (13) | 0.1958 (4) | 0.080 (3) |
| H11 | −0.0153 | 0.2422 | 0.2124 | 0.096* |
| C12 | −0.0058 (3) | 0.4903 (11) | 0.1761 (3) | 0.060 (2) |
| H12 | −0.0458 | 0.5282 | 0.1815 | 0.071* |
| C13 | 0.0351 (3) | 0.5999 (10) | 0.1490 (3) | 0.0498 (17) |
| H13 | 0.0222 | 0.7116 | 0.1360 | 0.060* |
| C14 | 0.1369 (3) | 0.6803 (9) | 0.1091 (2) | 0.0421 (15) |
| C15 | 0.1235 (3) | 1.3106 (9) | −0.0034 (3) | 0.059 (2) |
| H15 | 0.1630 | 1.3251 | −0.0172 | 0.071* |
| C16 | 0.0814 (3) | 1.4422 (9) | −0.0149 (4) | 0.060 (2) |

| | | | | |
|---|---|---|---|---|
| H16 | 0.0930 | 1.5416 | −0.0364 | 0.072* |
| C17 | 0.0229 (2) | 1.4299 (7) | 0.0047 (3) | 0.0343 (13) |
| C18 | 0.0088 (3) | 1.2696 (9) | 0.0332 (3) | 0.0503 (18) |
| H18 | −0.0308 | 1.2478 | 0.0457 | 0.060* |
| C19 | 0.0541 (3) | 1.1442 (9) | 0.0427 (3) | 0.0547 (18) |
| H19 | 0.0443 | 1.0388 | 0.0620 | 0.066* |
| Cl1 | 0.12558 (15) | 1.0350 (3) | −0.14483 (12) | 0.1014 (9) |
| Cl2 | 0.18725 (7) | 0.3008 (3) | 0.15571 (9) | 0.0618 (5) |
| N1 | 0.1109 (2) | 1.1684 (7) | 0.0254 (2) | 0.0441 (12) |
| O1 | 0.23874 (19) | 0.7055 (6) | −0.04024 (17) | 0.0497 (12) |
| O2 | 0.15049 (19) | 0.8395 (6) | −0.03665 (17) | 0.0472 (11) |
| O3 | 0.1874 (2) | 0.6329 (7) | 0.0914 (3) | 0.0665 (16) |
| O4 | 0.1161 (2) | 0.8292 (7) | 0.1003 (3) | 0.0747 (16) |
| Pb1 | 0.197071 (9) | 0.95216 (3) | 0.044368 (10) | 0.03659 (10) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.035 (3) | 0.051 (4) | 0.042 (3) | −0.010 (3) | −0.002 (3) | 0.002 (3) |
| C2 | 0.068 (4) | 0.047 (4) | 0.051 (4) | 0.012 (3) | −0.016 (3) | 0.012 (3) |
| C3 | 0.077 (5) | 0.076 (6) | 0.051 (4) | 0.001 (4) | −0.019 (4) | 0.024 (4) |
| C4 | 0.075 (5) | 0.086 (6) | 0.049 (4) | −0.017 (5) | −0.003 (4) | −0.001 (5) |
| C5 | 0.081 (6) | 0.112 (8) | 0.053 (5) | 0.003 (5) | −0.001 (4) | −0.029 (5) |
| C6 | 0.057 (4) | 0.062 (5) | 0.048 (4) | 0.002 (4) | −0.002 (3) | −0.002 (4) |
| C7 | 0.041 (3) | 0.044 (4) | 0.037 (3) | −0.013 (3) | 0.001 (3) | 0.006 (3) |
| C8 | 0.042 (3) | 0.044 (3) | 0.031 (3) | −0.019 (3) | −0.001 (3) | 0.001 (3) |
| C9 | 0.054 (4) | 0.063 (4) | 0.038 (3) | 0.001 (4) | 0.001 (3) | 0.010 (3) |
| C10 | 0.075 (5) | 0.081 (6) | 0.069 (5) | −0.015 (5) | 0.003 (4) | 0.032 (5) |
| C11 | 0.061 (5) | 0.096 (7) | 0.083 (6) | −0.007 (5) | 0.024 (4) | 0.024 (5) |
| C12 | 0.040 (4) | 0.062 (5) | 0.077 (5) | 0.000 (3) | 0.014 (4) | 0.008 (4) |
| C13 | 0.045 (3) | 0.057 (4) | 0.048 (4) | 0.012 (3) | 0.006 (3) | 0.001 (3) |
| C14 | 0.049 (4) | 0.044 (4) | 0.034 (3) | −0.001 (3) | −0.007 (3) | 0.008 (3) |
| C15 | 0.040 (4) | 0.047 (4) | 0.090 (6) | 0.009 (3) | 0.011 (4) | 0.021 (4) |
| C16 | 0.041 (4) | 0.049 (4) | 0.089 (5) | 0.020 (3) | 0.018 (4) | 0.031 (4) |
| C17 | 0.029 (3) | 0.031 (3) | 0.043 (3) | −0.010 (2) | −0.004 (2) | 0.000 (3) |
| C18 | 0.031 (3) | 0.031 (3) | 0.089 (5) | 0.008 (3) | 0.008 (3) | 0.014 (3) |
| C19 | 0.045 (4) | 0.026 (3) | 0.093 (5) | −0.001 (3) | 0.008 (3) | 0.018 (4) |
| Cl1 | 0.149 (2) | 0.0533 (13) | 0.1017 (18) | 0.0321 (15) | −0.0395 (17) | 0.0097 (13) |
| Cl2 | 0.0521 (10) | 0.0578 (11) | 0.0754 (12) | 0.0092 (8) | 0.0001 (8) | 0.0201 (10) |
| N1 | 0.042 (3) | 0.039 (3) | 0.052 (3) | 0.015 (2) | −0.003 (2) | 0.001 (3) |
| O1 | 0.040 (2) | 0.064 (3) | 0.045 (2) | 0.013 (2) | −0.0075 (19) | 0.002 (2) |
| O2 | 0.041 (2) | 0.051 (3) | 0.049 (3) | 0.013 (2) | −0.0015 (19) | −0.011 (2) |
| O3 | 0.047 (3) | 0.035 (3) | 0.117 (5) | 0.010 (2) | 0.032 (3) | 0.021 (3) |
| O4 | 0.061 (3) | 0.057 (3) | 0.106 (4) | 0.008 (3) | 0.024 (3) | 0.040 (3) |
| Pb1 | 0.03377 (14) | 0.03153 (15) | 0.04447 (15) | 0.00209 (9) | −0.00447 (9) | 0.00210 (10) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—C6 | 1.344 (10) | C11—H11 | 0.9300 |
| C1—C2 | 1.386 (9) | C12—C13 | 1.367 (10) |
| C1—C7 | 1.512 (8) | C12—H12 | 0.9300 |
| C2—C3 | 1.381 (10) | C13—H13 | 0.9300 |
| C2—Cl1 | 1.737 (8) | C14—O4 | 1.217 (8) |
| C3—C4 | 1.366 (12) | C14—O3 | 1.234 (7) |
| C3—H3 | 0.9300 | C15—N1 | 1.284 (8) |
| C4—C5 | 1.365 (13) | C15—C16 | 1.373 (9) |
| C4—H4 | 0.9300 | C15—H15 | 0.9300 |
| C5—C6 | 1.400 (11) | C16—C17 | 1.365 (8) |
| C5—H5 | 0.9300 | C16—H16 | 0.9300 |
| C6—H6 | 0.9300 | C17—C18 | 1.402 (8) |
| C7—O1 | 1.230 (7) | C17—C17$^{i}$ | 1.467 (11) |
| C7—O2 | 1.259 (7) | C18—C19 | 1.383 (9) |
| C8—C9 | 1.383 (9) | C18—H18 | 0.9300 |
| C8—C13 | 1.396 (8) | C19—N1 | 1.322 (8) |
| C8—C14 | 1.518 (8) | C19—H19 | 0.9300 |
| C9—C10 | 1.364 (10) | N1—Pb1 | 2.523 (5) |
| C9—Cl2 | 1.764 (7) | O2—Pb1 | 2.301 (4) |
| C10—C11 | 1.344 (11) | O3—Pb1 | 2.629 (5) |
| C10—H10 | 0.9300 | O4—Pb1 | 2.384 (5) |
| C11—C12 | 1.403 (12) | | |

| | | | |
|---|---|---|---|
| C6—C1—C2 | 117.8 (6) | C11—C12—H12 | 120.0 |
| C6—C1—C7 | 118.6 (6) | C12—C13—C8 | 121.8 (6) |
| C2—C1—C7 | 123.3 (6) | C12—C13—H13 | 119.1 |
| C3—C2—C1 | 120.2 (7) | C8—C13—H13 | 119.1 |
| C3—C2—Cl1 | 117.0 (5) | O4—C14—O3 | 122.8 (6) |
| C1—C2—Cl1 | 122.9 (5) | O4—C14—C8 | 116.5 (6) |
| C4—C3—C2 | 120.3 (7) | O3—C14—C8 | 120.6 (6) |
| C4—C3—H3 | 119.9 | N1—C15—C16 | 123.1 (6) |
| C2—C3—H3 | 119.9 | N1—C15—H15 | 118.4 |
| C3—C4—C5 | 120.9 (8) | C16—C15—H15 | 118.4 |
| C3—C4—H4 | 119.5 | C17—C16—C15 | 121.3 (6) |
| C5—C4—H4 | 119.5 | C17—C16—H16 | 119.3 |
| C4—C5—C6 | 117.0 (9) | C15—C16—H16 | 119.3 |
| C4—C5—H5 | 121.5 | C16—C17—C18 | 115.0 (5) |
| C6—C5—H5 | 121.5 | C16—C17—C17$^{i}$ | 123.2 (6) |
| C1—C6—C5 | 123.6 (8) | C18—C17—C17$^{i}$ | 121.7 (6) |
| C1—C6—H6 | 118.2 | C19—C18—C17 | 119.5 (5) |
| C5—C6—H6 | 118.2 | C19—C18—H18 | 120.2 |
| O1—C7—O2 | 122.3 (6) | C17—C18—H18 | 120.2 |
| O1—C7—C1 | 119.7 (6) | N1—C19—C18 | 122.5 (6) |
| O2—C7—C1 | 117.9 (5) | N1—C19—H19 | 118.8 |
| C9—C8—C13 | 116.1 (6) | C18—C19—H19 | 118.8 |
| C9—C8—C14 | 126.5 (6) | C15—N1—C19 | 118.3 (6) |

| | | | |
|---|---|---|---|
| C13—C8—C14 | 117.3 (6) | C15—N1—Pb1 | 117.3 (4) |
| C10—C9—C8 | 121.9 (7) | C19—N1—Pb1 | 124.4 (4) |
| C10—C9—Cl2 | 116.4 (6) | C7—O2—Pb1 | 105.9 (4) |
| C8—C9—Cl2 | 121.7 (5) | C14—O3—Pb1 | 87.2 (4) |
| C11—C10—C9 | 121.9 (8) | C14—O4—Pb1 | 99.4 (4) |
| C11—C10—H10 | 119.0 | O2—Pb1—O4 | 88.57 (19) |
| C9—C10—H10 | 119.0 | O2—Pb1—N1 | 75.99 (16) |
| C10—C11—C12 | 118.0 (8) | O4—Pb1—N1 | 77.48 (18) |
| C10—C11—H11 | 121.0 | O2—Pb1—O3 | 88.51 (18) |
| C12—C11—H11 | 121.0 | O4—Pb1—O3 | 50.55 (15) |
| C13—C12—C11 | 120.1 (7) | N1—Pb1—O3 | 126.24 (15) |
| C13—C12—H12 | 120.0 | | |

Symmetry code: (i) $-x$, $-y+3$, $-z$.

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C15—H15···O1$^{ii}$ | 0.93 | 2.40 | 3.239 (7) | 150 |
| C16—H16···O2$^{iii}$ | 0.93 | 2.55 | 3.361 (6) | 146 |
| C19—H19···O4 | 0.93 | 2.39 | 3.026 (5) | 125 |

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