

Acta Crystallographica Section E Structure Reports Online

ISSN 1600-5368

## Tetrakis(6-methyl-2,2'-bipyridine)- $1\kappa^2 N, N'; 2\kappa^2 N, N'; 3\kappa^2 N, N'; 4\kappa^2 N, N'$ tetra- $\mu$ -nitrato-1: $2\kappa^2 O:O'; 2:3\kappa^3 O:O', O'';$ - $2:3\kappa^3 O, O':O''; 3:4\kappa^2 O:O'$ -tetranitrato- $1\kappa^4 O, O'; 4\kappa^2 O, O'$ -tetralead(II)

### Roya Ahmadi,<sup>a</sup> Khadijeh Kalateh,<sup>a</sup> Robabeh Alizadeh,<sup>b</sup>\* Zeinab Khoshtarkib<sup>a</sup> and Vahid Amani<sup>a</sup>

<sup>a</sup>Islamic Azad University, Shahr-e-Rey Branch, Tehran, Iran, and <sup>b</sup>Damghan University of Basic Sciences, School of Chemistry, Damghan, Iran Correspondence e-mail: robabeh\_alizadeh@yahoo.com

Received 31 July 2009; accepted 28 August 2009

Key indicators: single-crystal X-ray study; T = 298 K; mean  $\sigma$ (C–C) = 0.016 Å; R factor = 0.049; wR factor = 0.137; data-to-parameter ratio = 19.0.

In the tetranuclear centrosymmetric title compound,  $[Pb_4(NO_3)_8(C_{11}H_{10}N_2)_4]$ , irregular  $PbN_2O_5$  and  $PbN_2O_4$ coordination polyhedra occur. The heptacoordinated lead(II) ion is bonded to two bidentate and one monodentate nitrate ion and one bidentate 6-methyl-2,2'-bipyridine (mbpy) ligand. The six-coordinate lead(II) ion is bonded to one bidentate and two monodentate nitrate anions and one mbpy ligand. In the crystal, bridging nitrate anions lead to infinite chains propagating in [111]. A number of  $C-H\cdots O$  hydrogen bonds may stabilize the structure.

### **Related literature**

For different metal complexes of 6-methyl-2,2'-bipyridine, see: Ahmadi, Kalateh *et al.* (2008); Ahmadi, Ebadi *et al.* (2008); Newkome *et al.* (1982); Onggo *et al.* (1990, 2005).



### Experimental

| Crystal data                        |                                 |
|-------------------------------------|---------------------------------|
| $[Pb_4(NO_3)_8(C_{11}H_{10}N_2)_4]$ | c = 12.642 (3) Å                |
| $M_r = 1002.84$                     | $\alpha = 109.25 \ (3)^{\circ}$ |
| Triclinic, $P\overline{1}$          | $\beta = 95.43 \ (3)^{\circ}$   |
| a = 11.093 (2) Å                    | $\gamma = 105.62 \ (3)^{\circ}$ |
| b = 11.266 (2) Å                    | V = 1407.0 (7) Å <sup>3</sup>   |

 $0.40 \times 0.30 \times 0.25 \text{ mm}$ 

RED32; Stoe & Cie, 2005)

 $T_{\min} = 0.021, T_{\max} = 0.052$ 

16752 measured reflections

7561 independent reflections

6277 reflections with  $I > 2\sigma(I)$ 

H-atom parameters constrained

T = 298 K

 $R_{\rm int} = 0.089$ 

397 parameters

 $\Delta \rho_{\text{max}} = 2.18 \text{ e } \text{\AA}^{-3}$  $\Delta \rho_{\text{min}} = -2.37 \text{ e } \text{\AA}^{-3}$ 

Z = 2Mo  $K\alpha$  radiation  $\mu = 12.03 \text{ mm}^{-1}$ 

### Data collection

Bruker SMART CCD

diffractometer Absorption correction: numerical shape of crystal determined optically (X-SHAPE and X-

### Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.049$  $wR(F^2) = 0.137$ S = 1.107561 reflections

### Table 1

Selected bond lengths (Å).

| Pb1-O1 | 2.566 (8) | Pb2-O9               | 2.624 (7)  |
|--------|-----------|----------------------|------------|
| Pb1-O2 | 2.609 (8) | Pb2-O10              | 2.763 (11) |
| Pb1-O4 | 2.851 (8) | Pb2-O11              | 2.629 (8)  |
| Pb1-O5 | 2.675 (9) | Pb2-N6               | 2.470 (7)  |
| Pb1-O8 | 2.693 (8) | Pb2-N7               | 2.422 (8)  |
| Pb1-N1 | 2.618 (8) | Pb2-O12 <sup>i</sup> | 2.910 (9)  |
| Pb1-N2 | 2.528 (8) |                      |            |

Symmetry code: (i) -x + 1, -y, -z.

### Table 2

Hydrogen-bond geometry (Å,  $^{\circ}$ ).

| $D - H \cdots A$             | D-H              | $H \cdot \cdot \cdot A$ | $D \cdots A$   | $D - \mathbf{H} \cdots A$ |
|------------------------------|------------------|-------------------------|----------------|---------------------------|
| C1−H1A···O4 <sup>ii</sup>    | 0.96             | 2.55                    | 3.378 (14)     | 144                       |
| C3−H3···O10 <sup>iii</sup>   | 0.93             | 2.50                    | 3.368 (17)     | 155                       |
| $C10-H10\cdots O6^{iv}$      | 0.93             | 2.47                    | 3.349 (17)     | 157                       |
| C12−H12A···O10               | 0.96             | 2.48                    | 3.299 (15)     | 143                       |
| $C12 - H12B \cdots O7$       | 0.96             | 2.49                    | 3.412 (17)     | 160                       |
| $C12 - H12C \cdots O8^{iii}$ | 0.96             | 2.59                    | 3.516 (14)     | 162                       |
| C19−H19· · · O9 <sup>v</sup> | 0.93             | 2.45                    | 3.253 (13)     | 145                       |
| $C21 - H21 \cdots O1^{iv}$   | 0.93             | 2.42                    | 3.212 (15)     | 143                       |
| $C22-H22\cdots O12^{i}$      | 0.93             | 2.53                    | 3.268 (15)     | 137                       |
| Symmetry codes:              | (i) $-r + 1 - r$ | -v - z (ii)             | -r + 2 - v + 1 | $-7 \pm 1$ (iii)          |

Symmetry codes: (1) -x + 1, -y, -z; (11) -x + 2, -y + 1, -z + 1; (11) -x + 1, -y + 1, -z + 1; (iv) -x + 2, -y, -z + 1; (v) -x + 1, -y, -z + 1.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

We are grateful to Damghan University of Basic Sciences and the Islamic Azad University, Shahr-e-Rey Branch, for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5028).

### References

Ahmadi, R., Ebadi, A., Kalateh, K., Norouzi, A. & Amani, V. (2008). Acta Cryst. E64, m1407.

Ahmadi, R., Kalateh, K., Ebadi, A., Amani, V. & Khavasi, H. R. (2008). Acta Cryst. E64, m1266.

- Bruker (1998). SMART, SAINT and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.
- Farrugia, L. J. (1997). J. Appl. Cryst. 30, 565.
- Farrugia, L. J. (1999). J. Appl. Cryst. 32, 837–838.
- Newkome, G. R., Fronczek, F. R., Gupta, V. K., Puckett, W. E., Pantaleo, D. C. & Kiefer, G. E. (1982). J. Am. Chem. Soc. 104, 1782–1783.
- Onggo, D., Hook, J. M., Rae, A. D. & Goodwin, H. A. (1990). *Inorg. Chim.* Acta, **173**, 19–30.
- Onggo, D., Scudder, M. L., Craig, D. C. & Goodwin, H. A. (2005). J. Mol. Struct. 738, 129-136.
- Sheldrick, G. M. (2008). Acta Cryst. A64, 112-122.
- Stoe & Cie (2005). X-RED32 and X-SHAPE. Stoe & Cie, Darmstadt, Germany.

# supporting information

Acta Cryst. (2009). E65, m1169-m1170 [doi:10.1107/S160053680903459X]

## Tetrakis(6-methyl-2,2'-bipyridine)- $1\kappa^2 N, N'; 2\kappa^2 N, N'; 3\kappa^2 N, N'; 4\kappa^2 N, N'$ -tetra- $\mu$ nitrato- $1:2\kappa^2 O:O'; 2:3\kappa^3 O:O', O''; 2:3\kappa^3 O, O':O''; 3:4\kappa^2 O:O'$ tetranitrato- $1\kappa^4 O, O'; 4\kappa^2 O, O'$ -tetralead(II)

## Roya Ahmadi, Khadijeh Kalateh, Robabeh Alizadeh, Zeinab Khoshtarkib and Vahid Amani

## S1. Comment

6-Methyl-2,2'-bipyridine (6-mbipy) is a good bidentate ligand, and numerous complexes with 6-mbipy have been prepared, such as that of zinc (Ahmadi, Kalateh *et al.*, 2008), mercury (Ahmadi, Ebadi *et al.*, 2008), palladium (Newkome *et al.*, 1982), ruthenium (Onggo, Scudder *et al.*, 2005) and iron (Onggo, Hook *et al.*, 1990). We report herein the synthesis and crystal structure of the title compound (I).

The asymmetric unit of this tetrameric compound, (I), (Fig. 1), contains two different hepta and hexa-coordinated  $Pb^{II}$  centers that the metallic centers site in distorted environment by five oxygen atoms from three nitrate and two nitrogen atom from one 6-methyl-2,2'-bipyridine and distorted octahedral environment by four oxygen atoms from three nitrate and two nitrogen atom from one 6-methyl-2,2'-bipyridine respectively. The Pb—O and Pb—N bond lengths and angles are collected in Table 1.

In the crystal structure, intermolecular C—H···O hydrogen bonds (Table 2) may stabilize the structure.

## **S2.** Experimental

6-Methyl-2,2'-bipyridine (0.14 g, 0.79 mmol, 0.13 ml) in 5 ml me thanol was added to a solution of  $Pb(NO_3)_2$  (0.26 g, 0.79 mmol) in methanol (10 ml) and the resulting colorless solution was stirred at 313 K for 30 min. This solution was left to evaporate slowly at room temperature. After one week, colorless blocks of (I) were isolated (yield 0.29 g, 73.2%).

## S3. Refinement

All H atoms were positioned geometrically, with C—H = 0.93Å and refined as riding with  $U_{iso}$ (H)=1.2 $U_{eq}$ .



## Figure 1

The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level. [Symmetry codes: (*a*) -*x*,-y,-z]

Tetrakis(6-methyl-2,2'-bipyridine)- $1\kappa^2 N, N'$ ;  $2\kappa^2 N, N'$ ;  $3\kappa^2 N, N'$ ;  $4\kappa^2 N, N'$ - tetra- $\mu$ -nitrato-1: $2\kappa^2 O:O'$ ; 2: $3\kappa^3 O:O',O''$ ; 2: $3\kappa^3 O,O':O''$ ; 3: $4\kappa^2 O:O'$ -tetranitrato-  $1\kappa^4 O,O'$ ;  $4\kappa^2 O,O'$ -tetralead(II)

| $[Pb_4(NO_3)_8(C_{11}H_{10}N_2)_4]$             | Z = 2   |
|---|---|
| $M_r = 1002.84$                                 | F(000) = 936  |
| Triclinic, $P\overline{1}$                      | $D_{\rm x} = 2.367 {\rm Mg} {\rm m}^{-3}$                                 |
| Hall symbol: -P 1                               | Mo <i>K</i> $\alpha$ radiation, $\lambda = 0.71073$ Å                     |
| a = 11.093 (2) Å                                | Cell parameters from 1311 reflections                                     |
| b = 11.266(2) Å                                 | $\theta = 1.7 - 29.2^{\circ}$   |
| c = 12.642 (3) Å                                | $\mu = 12.03 \text{ mm}^{-1}$   |
| $\alpha = 109.25 \ (3)^{\circ}$                 | T = 298  K  |
| $\beta = 95.43 (3)^{\circ}$                     | Block, colorless  |
| $\gamma = 105.62(3)^{\circ}$                    | $0.40 \times 0.30 \times 0.25 \text{ mm}$                                 |
| V = 1407.0 (7) Å <sup>3</sup>                   |   |
| Data collection                                 |   |
| Bruker SMART CCD                                | 16752 measured reflections  |
| diffractometer                                  | 7561 independent reflections  |
| Radiation source: fine-focus sealed tube        | 6277 reflections with $I > 2\sigma(I)$                                    |
| Graphite monochromator                          | $R_{\rm int} = 0.089$   |
| ωscans  | $\theta_{\text{max}} = 29.2^{\circ}, \ \theta_{\text{min}} = 1.7^{\circ}$ |
| Absorption correction: numerical                | $h = -15 \rightarrow 15$  |
| shape of crystal determined optically (program? | $k = -15 \rightarrow 14$  |
| reference?)                                     | $l = -17 \rightarrow 17$  |
| $T_{\min} = 0.021, \ T_{\max} = 0.052$          |   |

Refinement

| Refinement on $F^2$                             | Secondary atom site location: difference Fourier        |
|---|---|
| Least-squares matrix: full                      | map   |
| $R[F^2 > 2\sigma(F^2)] = 0.049$                 | Hydrogen site location: inferred from                   |
| $wR(F^2) = 0.137$                               | neighbouring sites                                      |
| S = 1.10  | H-atom parameters constrained                           |
| 7561 reflections                                | $w = 1/[\sigma^2(F_o^2) + (0.0636P)^2 + 3.7094P]$       |
| 397 parameters                                  | where $P = (F_o^2 + 2F_c^2)/3$                          |
| 0 restraints                                    | $(\Delta/\sigma)_{\rm max} = 0.011$                     |
| Primary atom site location: structure-invariant | $\Delta \rho_{\rm max} = 2.18 \text{ e} \text{ Å}^{-3}$ |
| direct methods                                  | $\Delta \rho_{\min} = -2.37 \text{ e} \text{ Å}^{-3}$   |
|   |   |

## 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.

**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  $F^2 > \sigma(F^2)$  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  $(A^2)$ 

|      | x           | у           | Ζ           | $U_{ m iso}$ */ $U_{ m eq}$ |
|------|-------------|-------------|-------------|-----------------------------|
| C1   | 0.9335 (12) | 0.7297 (9)  | 0.8235 (9)  | 0.062 (3)                   |
| H1A  | 0.9208      | 0.6913      | 0.7416      | 0.074*                      |
| H1B  | 1.0234      | 0.7629      | 0.8558      | 0.074*                      |
| H1C  | 0.8983      | 0.8013      | 0.8446      | 0.074*                      |
| C2   | 0.8691 (9)  | 0.6277 (9)  | 0.8670 (7)  | 0.0478 (18)                 |
| C3   | 0.7749 (12) | 0.6393 (12) | 0.9294 (9)  | 0.066 (3)                   |
| H3   | 0.7515      | 0.7159      | 0.9485      | 0.079*                      |
| C4   | 0.7160 (12) | 0.5403 (12) | 0.9634 (10) | 0.067 (3)                   |
| H4   | 0.6503      | 0.5476      | 1.0032      | 0.080*                      |
| C5   | 0.7530 (10) | 0.4297 (10) | 0.9393 (8)  | 0.053 (2)                   |
| Н5   | 0.7142      | 0.3619      | 0.9636      | 0.063*                      |
| C6   | 0.8491 (8)  | 0.4204 (8)  | 0.8785 (7)  | 0.0412 (15)                 |
| C7   | 0.8970 (8)  | 0.3056 (8)  | 0.8500 (7)  | 0.0424 (16)                 |
| C8   | 0.8691 (9)  | 0.2158 (9)  | 0.9059 (9)  | 0.052 (2)                   |
| H8   | 0.8187      | 0.2264      | 0.9609      | 0.063*                      |
| C9   | 0.9175 (12) | 0.1117 (9)  | 0.8781 (10) | 0.062 (3)                   |
| H9   | 0.8987      | 0.0501      | 0.9132      | 0.074*                      |
| C10  | 0.9918 (11) | 0.0992 (10) | 0.8002 (10) | 0.060 (2)                   |
| H10  | 1.0288      | 0.0319      | 0.7834      | 0.072*                      |
| C11  | 1.0127 (10) | 0.1894 (9)  | 0.7449 (9)  | 0.053 (2)                   |
| H11  | 1.0577      | 0.1762      | 0.6859      | 0.063*                      |
| C12  | 0.3946 (11) | 0.3441 (9)  | 0.3475 (10) | 0.062 (3)                   |
| H12A | 0.3814      | 0.3192      | 0.2659      | 0.075*                      |
| H12B | 0.4834      | 0.3913      | 0.3805      | 0.075*                      |
| H12C | 0.3446      | 0.4000      | 0.3778      | 0.075*                      |

| C13 | 0.3544 (9)  | 0.2208 (9)   | 0.3761 (8)  | 0.0505 (19)  |
|-----|-------------|--------------|-------------|--------------|
| C14 | 0.2605 (10) | 0.2030 (13)  | 0.4403 (10) | 0.068 (3)    |
| H14 | 0.2193      | 0.2660       | 0.4646      | 0.081*       |
| C15 | 0.2295 (11) | 0.0929 (14)  | 0.4671 (10) | 0.075 (3)    |
| H15 | 0.1664      | 0.0798       | 0.5094      | 0.091*       |
| C16 | 0.2915 (9)  | 0.0006 (12)  | 0.4318 (9)  | 0.062 (3)    |
| H16 | 0.2723      | -0.0745      | 0.4507      | 0.074*       |
| C17 | 0.3841 (7)  | 0.0227 (8)   | 0.3667 (7)  | 0.0425 (16)  |
| C18 | 0.4550 (8)  | -0.0716 (8)  | 0.3243 (7)  | 0.0442 (17)  |
| C19 | 0.4256 (11) | -0.1938 (10) | 0.3387 (9)  | 0.063 (3)    |
| H19 | 0.3604      | -0.2185      | 0.3759      | 0.076*       |
| C20 | 0.4965 (13) | -0.2769 (10) | 0.2960 (11) | 0.072 (3)    |
| H20 | 0.4774      | -0.3589      | 0.3036      | 0.087*       |
| C21 | 0.5926 (12) | -0.2405 (10) | 0.2438 (11) | 0.067 (3)    |
| H21 | 0.6405      | -0.2959      | 0.2160      | 0.081*       |
| C22 | 0.6182 (10) | -0.1186 (10) | 0.2325 (10) | 0.059 (2)    |
| H22 | 0.6836      | -0.0931      | 0.1956      | 0.071*       |
| N1  | 0.9053 (7)  | 0.5176 (7)   | 0.8420 (6)  | 0.0411 (13)  |
| N2  | 0.9713 (8)  | 0.2921 (7)   | 0.7728 (6)  | 0.0477 (16)  |
| N3  | 1.2895 (7)  | 0.5378 (9)   | 0.7956 (8)  | 0.057 (2)    |
| N4  | 0.9322 (8)  | 0.2233 (8)   | 0.4204 (7)  | 0.0519 (17)  |
| N5  | 0.7285 (7)  | 0.3733 (8)   | 0.4898 (6)  | 0.0469 (15)  |
| N6  | 0.4148 (6)  | 0.1316 (7)   | 0.3391 (6)  | 0.0410 (13)  |
| N7  | 0.5523 (7)  | -0.0368 (7)  | 0.2727 (6)  | 0.0443 (14)  |
| N8  | 0.3510 (8)  | 0.0199 (8)   | 0.0272 (7)  | 0.0505 (16)  |
| 01  | 1.2315 (7)  | 0.4180 (7)   | 0.7307 (8)  | 0.071 (2)    |
| O2  | 1.2252 (7)  | 0.6164 (7)   | 0.8206 (7)  | 0.0591 (16)  |
| O3  | 1.4048 (7)  | 0.5766 (10)  | 0.8320 (9)  | 0.083 (3)    |
| O4  | 0.9642 (9)  | 0.3404 (8)   | 0.4264 (7)  | 0.068 (2)    |
| 05  | 0.9608 (9)  | 0.2066 (8)   | 0.5144 (8)  | 0.076 (2)    |
| O6  | 0.8789 (12) | 0.1308 (10)  | 0.3342 (9)  | 0.110 (4)    |
| 07  | 0.7188 (10) | 0.4369 (9)   | 0.4276 (8)  | 0.082 (3)    |
| 08  | 0.7761 (7)  | 0.4279 (7)   | 0.5941 (6)  | 0.0578 (16)  |
| O9  | 0.6873 (7)  | 0.2468 (7)   | 0.4469 (6)  | 0.0612 (17)  |
| O10 | 0.3682 (10) | 0.1365 (8)   | 0.0852 (8)  | 0.077 (3)    |
| 011 | 0.4108 (7)  | -0.0399 (7)  | 0.0670 (7)  | 0.0614 (17)  |
| O12 | 0.2747 (8)  | -0.0373 (8)  | -0.0665 (7) | 0.070 (2)    |
| Pb1 | 1.02005 (3) | 0.45241 (3)  | 0.67017 (2) | 0.04043 (9)  |
| Pb2 | 0.58525 (3) | 0.16179 (3)  | 0.22876 (3) | 0.04202 (10) |

Atomic displacement parameters  $(Å^2)$ 

|    | $U^{11}$  | $U^{22}$  | $U^{33}$  | $U^{12}$  | $U^{13}$  | $U^{23}$  |
|----|-----------|-----------|-----------|-----------|-----------|-----------|
| C1 | 0.087 (7) | 0.044 (5) | 0.063 (6) | 0.030 (5) | 0.012 (5) | 0.024 (4) |
| C2 | 0.057 (5) | 0.046 (4) | 0.046 (4) | 0.030 (4) | 0.008 (4) | 0.014 (3) |
| C3 | 0.086 (8) | 0.074 (7) | 0.052 (5) | 0.049 (6) | 0.018 (5) | 0.021 (5) |
| C4 | 0.076 (7) | 0.081 (7) | 0.065 (6) | 0.048 (6) | 0.041 (5) | 0.028 (5) |
| C5 | 0.057 (5) | 0.064 (5) | 0.048 (4) | 0.022 (4) | 0.017 (4) | 0.029 (4) |

# supporting information

| C6  | 0.045 (4)    | 0.048 (4)    | 0.038 (3)    | 0.021 (3)    | 0.009 (3)    | 0.020 (3)    |
|-----|--------------|--------------|--------------|--------------|--------------|--------------|
| C7  | 0.047 (4)    | 0.040 (4)    | 0.042 (4)    | 0.012 (3)    | 0.011 (3)    | 0.019 (3)    |
| C8  | 0.053 (5)    | 0.050 (5)    | 0.067 (5)    | 0.016 (4)    | 0.021 (4)    | 0.036 (4)    |
| C9  | 0.079 (7)    | 0.043 (4)    | 0.073 (6)    | 0.019 (5)    | 0.020 (5)    | 0.032 (5)    |
| C10 | 0.070 (6)    | 0.043 (4)    | 0.075 (6)    | 0.026 (4)    | 0.017 (5)    | 0.026 (4)    |
| C11 | 0.059 (5)    | 0.048 (5)    | 0.061 (5)    | 0.028 (4)    | 0.020 (4)    | 0.022 (4)    |
| C12 | 0.068 (6)    | 0.045 (5)    | 0.078 (7)    | 0.035 (4)    | 0.015 (5)    | 0.013 (4)    |
| C13 | 0.048 (5)    | 0.050 (5)    | 0.049 (4)    | 0.019 (4)    | 0.008 (4)    | 0.010 (4)    |
| C14 | 0.052 (5)    | 0.086 (8)    | 0.067 (6)    | 0.031 (5)    | 0.027 (5)    | 0.019 (6)    |
| C15 | 0.056 (6)    | 0.112 (10)   | 0.064 (6)    | 0.025 (6)    | 0.036 (5)    | 0.035 (6)    |
| C16 | 0.044 (5)    | 0.080 (7)    | 0.064 (6)    | 0.009 (5)    | 0.018 (4)    | 0.038 (5)    |
| C17 | 0.034 (3)    | 0.048 (4)    | 0.044 (4)    | 0.007 (3)    | 0.008 (3)    | 0.021 (3)    |
| C18 | 0.040 (4)    | 0.039 (4)    | 0.048 (4)    | 0.003 (3)    | 0.004 (3)    | 0.019 (3)    |
| C19 | 0.070 (6)    | 0.053 (5)    | 0.066 (6)    | 0.002 (5)    | 0.007 (5)    | 0.037 (5)    |
| C20 | 0.093 (8)    | 0.044 (5)    | 0.082 (7)    | 0.019 (5)    | -0.008 (6)   | 0.036 (5)    |
| C21 | 0.083 (8)    | 0.050 (5)    | 0.080 (7)    | 0.039 (5)    | 0.005 (6)    | 0.027 (5)    |
| C22 | 0.053 (5)    | 0.052 (5)    | 0.075 (6)    | 0.026 (4)    | 0.006 (5)    | 0.022 (5)    |
| N1  | 0.046 (4)    | 0.040 (3)    | 0.043 (3)    | 0.022 (3)    | 0.010 (3)    | 0.016 (3)    |
| N2  | 0.060 (4)    | 0.043 (4)    | 0.045 (4)    | 0.020 (3)    | 0.015 (3)    | 0.018 (3)    |
| N3  | 0.038 (4)    | 0.064 (5)    | 0.081 (6)    | 0.012 (3)    | 0.004 (4)    | 0.047 (5)    |
| N4  | 0.057 (4)    | 0.054 (4)    | 0.055 (4)    | 0.019 (3)    | 0.021 (3)    | 0.017 (3)    |
| N5  | 0.038 (3)    | 0.056 (4)    | 0.049 (4)    | 0.020 (3)    | 0.007 (3)    | 0.019 (3)    |
| N6  | 0.035 (3)    | 0.041 (3)    | 0.045 (3)    | 0.012 (3)    | 0.006 (3)    | 0.014 (3)    |
| N7  | 0.052 (4)    | 0.036 (3)    | 0.047 (4)    | 0.017 (3)    | 0.011 (3)    | 0.016 (3)    |
| N8  | 0.051 (4)    | 0.053 (4)    | 0.050 (4)    | 0.018 (3)    | 0.015 (3)    | 0.020 (3)    |
| 01  | 0.054 (4)    | 0.053 (4)    | 0.118 (7)    | 0.021 (3)    | 0.016 (4)    | 0.045 (4)    |
| O2  | 0.054 (4)    | 0.053 (4)    | 0.076 (5)    | 0.024 (3)    | 0.017 (3)    | 0.025 (3)    |
| O3  | 0.043 (4)    | 0.096 (6)    | 0.111 (7)    | 0.012 (4)    | 0.003 (4)    | 0.053 (6)    |
| O4  | 0.088 (6)    | 0.058 (4)    | 0.062 (4)    | 0.015 (4)    | 0.019 (4)    | 0.032 (3)    |
| O5  | 0.086 (6)    | 0.066 (5)    | 0.091 (6)    | 0.027 (4)    | 0.019 (5)    | 0.047 (4)    |
| 06  | 0.121 (9)    | 0.070 (6)    | 0.086 (7)    | 0.011 (6)    | 0.025 (6)    | -0.023 (5)   |
| O7  | 0.104 (7)    | 0.089 (6)    | 0.081 (6)    | 0.051 (5)    | 0.020 (5)    | 0.051 (5)    |
| 08  | 0.052 (4)    | 0.065 (4)    | 0.046 (3)    | 0.019 (3)    | 0.011 (3)    | 0.007 (3)    |
| 09  | 0.056 (4)    | 0.063 (4)    | 0.058 (4)    | 0.011 (3)    | 0.009 (3)    | 0.022 (3)    |
| O10 | 0.089 (7)    | 0.059 (5)    | 0.078 (5)    | 0.040 (5)    | -0.004 (5)   | 0.023 (4)    |
| O11 | 0.057 (4)    | 0.062 (4)    | 0.075 (5)    | 0.025 (3)    | 0.009 (3)    | 0.033 (4)    |
| O12 | 0.062 (4)    | 0.078 (5)    | 0.059 (4)    | 0.015 (4)    | -0.002 (3)   | 0.021 (4)    |
| Pb1 | 0.04429 (17) | 0.04041 (16) | 0.04192 (15) | 0.01671 (12) | 0.01061 (11) | 0.01886 (12) |
| Pb2 | 0.04585 (17) | 0.03412 (15) | 0.04792 (17) | 0.01031 (11) | 0.01553 (12) | 0.01783 (12) |
|     |              |              |              |              |              |              |

## Geometric parameters (Å, °)

| Pb1—O1 | 2.566 (8) | C5—C6   | 1.379 (14) |  |
|--------|-----------|---------|------------|--|
| Pb1—O2 | 2.609 (8) | C6—C7   | 1.478 (13) |  |
| Pb1—O4 | 2.851 (8) | C7—C8   | 1.401 (14) |  |
| Pb1—O5 | 2.675 (9) | C8—C9   | 1.375 (16) |  |
| Pb1—O8 | 2.693 (8) | C9—C10  | 1.339 (18) |  |
| Pb1—N1 | 2.618 (8) | C10-C11 | 1.396 (16) |  |
|        |           |         |            |  |

| DI 1 NO               | 2 529 (9)              | C12 C12   | 1 512 (15)             |
|-----------------------|------------------------|---|------------------------|
| Pb1—N2                | 2.528 (8)              |   | 1.512 (15)             |
| Pb2—09                | 2.624 (7)              | C13—C14   | 1.392 (16)             |
| Pb2—O10               | 2.763 (11)             | C14—C15   | 1.36 (2)               |
| Pb2—O11               | 2.629 (8)              | C15—C16   | 1.375 (18)             |
| Pb2—N6                | 2.470 (7)              | C16—C17   | 1.397 (14)             |
| Pb2—N7                | 2.422 (8)              | C17—C18   | 1.480 (13)             |
| $Pb2-O12^{i}$         | 2.910 (9)              | C18—C19   | 1.403 (15)             |
| 01—N3                 | 1269(13)               | C19-C20   | 1 389 (18)             |
| $O_2 N_3$             | 1.267(13)              | $C_{20}$ $C_{21}$                                     | 1.309(10)<br>1.348(10) |
| $O_2 = N_3$           | 1.201(12)              | $C_{20} = C_{21}$                                     | 1.346(19)<br>1.284(17) |
|                       | 1.222 (12)             |   | 1.384 (17)             |
| 04—N4                 | 1.245 (13)             | CI—HIA  | 0.9600                 |
| O5—N4                 | 1.287 (13)             | C1—H1B  | 0.9600                 |
| O6—N4                 | 1.192 (14)             | C1—H1C  | 0.9600                 |
| O7—N5                 | 1.241 (13)             | С3—Н3   | 0.9300                 |
| O8—N5                 | 1.247 (10)             | C4—H4   | 0.9300                 |
| O9—N5                 | 1.275 (12)             | С5—Н5   | 0.9300                 |
| O10—N8                | 1.226 (13)             | С8—Н8   | 0.9300                 |
| 011—N8                | 1 244 (12)             | С9—Н9   | 0.9300                 |
| 012 N8                | 1.244(12)              | C10 H10   | 0.9300                 |
| N1_C2                 | 1.244(12)<br>1.254(12) |   | 0.9300                 |
| NI-C2                 | 1.334(13)              |   | 0.9300                 |
|                       | 1.349 (12)             | CI2—HI2A  | 0.9600                 |
| N2C/                  | 1.333 (12)             | С12—Н12В  | 0.9600                 |
| N2—C11                | 1.312 (14)             | C12—H12C  | 0.9600                 |
| N6—C13                | 1.338 (13)             | C14—H14   | 0.9300                 |
| N6—C17                | 1.350 (12)             | C15—H15   | 0.9300                 |
| N7—C18                | 1.348 (12)             | C16—H16   | 0.9300                 |
| N7—C22                | 1.329 (14)             | C19—H19   | 0.9300                 |
| C1—C2                 | 1 477 (15)             | С20—Н20   | 0.9300                 |
| $C^2 - C^3$           | 1 376 (16)             | C21—H21   | 0.9300                 |
| $C_2 C_3$             | 1.370(10)<br>1.354(10) | $\begin{array}{c} C21 & H21 \\ C22 & H22 \end{array}$ | 0.9300                 |
| $C_{3}$               | 1.334(19)              | 022—1122  | 0.9300                 |
| C4—C3                 | 1.303 (18)             |   |                        |
|                       |                        |   |                        |
| O1—Pb1—O2             | 49.6 (3)               | N1  | 119.6 (10)             |
| O1—Pb1—O4             | 105.4 (3)              | C1—C2—C3  | 123.3 (10)             |
| O1—Pb1—O5             | 84.7 (3)               | C2—C3—C4  | 120.6 (12)             |
| O1—Pb1—O8             | 166.8 (3)              | C3—C4—C5  | 120.0 (12)             |
| O1—Pb1—N1             | 111.9 (3)              | C4—C5—C6  | 118.8 (11)             |
| O1—Pb1—N2             | 73.1 (3)               | N1—C6—C5  | 121.1 (9)              |
| O2—Pb1—O4             | 133.0 (3)              | N1—C6—C7  | 116.2 (8)              |
| 02 - Pb1 - 05         | 1341(3)                | $C_{5}-C_{6}-C_{7}$                                   | 122.7(9)               |
| 02 - Pb1 - 08         | 1419(2)                | $N^{2}-C^{7}-C^{6}$                                   | 1184(8)                |
| $O_2$ Pb1 N1          | (2)                    | $N_2 = C_7 = C_0$                                     | 120.7(0)               |
| $O_2$ $P_1$ $N_2$     | 03.7(2)                | $N_2 - C_7 - C_8$                                     | 120.7(9)               |
| $02 - r_{01} - n_{2}$ | 74.7 (J)               | $C - C - C \delta$                                    | 120.8(8)               |
| 04—Pb1—05             | 45.5 (3)               | $C/-C\delta-C9$                                       | 118.9 (10)             |
| O4—Pb1—O8             | 71.0 (3)               | C8—C9—C10   | 119.8 (10)             |
| O4—Pb1—N1             | 140.2 (3)              | C9—C10—C11  | 118.6 (11)             |
| O4—Pb1—N2             | 116.8 (3)              | N2—C11—C10  | 122.6 (10)             |
| O5—Pb1—O8             | 84.0 (3)               | N6-C13-C12  | 117.1 (9)              |

| O5—Pb1—N1                | 124.2 (3)  | N6-C13-C14    | 121.8 (10) |
|--------------------------|------------|---------------|------------|
| O5—Pb1—N2                | 72.2 (3)   | C12—C13—C14   | 121.1 (10) |
| O8—Pb1—N1                | 69.5 (2)   | C13—C14—C15   | 119.5 (12) |
| O8—Pb1—N2                | 96.9 (3)   | C14—C15—C16   | 120.0 (12) |
| N1—Pb1—N2                | 63.9 (3)   | C15—C16—C17   | 118.2 (12) |
| O9—Pb2—O10               | 141.1 (3)  | N6—C17—C16    | 122.1 (9)  |
| O9—Pb2—O11               | 141.2 (3)  | N6-C17-C18    | 115.9 (7)  |
| O9—Pb2—N6                | 70.3 (2)   | C16—C17—C18   | 122.0 (9)  |
| O9—Pb2—N7                | 75.0 (2)   | N7—C18—C17    | 118.5 (8)  |
| O9—Pb2—O12 <sup>i</sup>  | 117.1 (2)  | N7—C18—C19    | 119.6 (9)  |
| O10—Pb2—O11              | 46.0 (3)   | C17—C18—C19   | 121.9 (9)  |
| O10—Pb2—N6               | 75.8 (3)   | C18—C19—C20   | 118.3 (11) |
| O10—Pb2—N7               | 109.1 (3)  | C19—C20—C21   | 121.0 (11) |
| O10—Pb2—O12 <sup>i</sup> | 101.8 (3)  | C20—C21—C22   | 118.3 (12) |
| O11—Pb2—N6               | 81.6 (2)   | N7—C22—C21    | 122.0 (11) |
| O11—Pb2—N7               | 69.7 (3)   | C2—C1—H1A     | 110.00     |
| O11—Pb2—O12 <sup>i</sup> | 74.6 (3)   | C2—C1—H1B     | 110.00     |
| N6—Pb2—N7                | 67.5 (3)   | C2—C1—H1C     | 110.00     |
| O12 <sup>i</sup> —Pb2—N6 | 147.3 (3)  | H1A—C1—H1B    | 109.00     |
| O12 <sup>i</sup> —Pb2—N7 | 83.3 (3)   | H1A—C1—H1C    | 109.00     |
| Pb1—O1—N3                | 96.4 (6)   | H1B—C1—H1C    | 109.00     |
| Pb1—O2—N3                | 94.6 (6)   | С2—С3—Н3      | 120.00     |
| Pb1—O4—N4                | 94.8 (6)   | С4—С3—Н3      | 120.00     |
| Pb1—O4—Pb1 <sup>ii</sup> | 110.9 (3)  | C3—C4—H4      | 120.00     |
| Pb1 <sup>ii</sup> —O4—N4 | 153.4 (7)  | C5—C4—H4      | 120.00     |
| Pb1—O5—N4                | 102.2 (6)  | C4—C5—H5      | 121.00     |
| Pb1—O8—N5                | 120.0 (6)  | С6—С5—Н5      | 121.00     |
| Pb2—O9—N5                | 110.7 (5)  | С7—С8—Н8      | 121.00     |
| Pb2—O10—N8               | 94.1 (7)   | С9—С8—Н8      | 121.00     |
| Pb2—O11—N8               | 100.3 (6)  | С8—С9—Н9      | 120.00     |
| Pb2 <sup>i</sup> —O12—N8 | 106.0 (7)  | С10—С9—Н9     | 120.00     |
| Pb1—N1—C2                | 119.9 (6)  | С9—С10—Н10    | 121.00     |
| Pb1—N1—C6                | 116.9 (6)  | C11—C10—H10   | 121.00     |
| C2—N1—C6                 | 119.9 (8)  | N2-C11-H11    | 119.00     |
| Pb1—N2—C7                | 120.8 (6)  | C10-C11-H11   | 119.00     |
| Pb1—N2—C11               | 119.8 (7)  | C13—C12—H12A  | 109.00     |
| C7—N2—C11                | 119.2 (9)  | C13—C12—H12B  | 109.00     |
| O1—N3—O2                 | 118.2 (8)  | C13—C12—H12C  | 110.00     |
| O1—N3—O3                 | 121.0 (10) | H12A—C12—H12B | 109.00     |
| O2—N3—O3                 | 120.7 (10) | H12A—C12—H12C | 109.00     |
| O4—N4—O5                 | 115.7 (9)  | H12B—C12—H12C | 109.00     |
| O4—N4—O6                 | 123.4 (10) | C13—C14—H14   | 120.00     |
| O5—N4—O6                 | 120.9 (10) | C15—C14—H14   | 120.00     |
| O7—N5—O8                 | 122.7 (9)  | C14—C15—H15   | 120.00     |
| O7—N5—O9                 | 119.5 (8)  | C16—C15—H15   | 120.00     |
| O8—N5—O9                 | 117.8 (8)  | C15—C16—H16   | 121.00     |
| Pb2—N6—C13               | 122.8 (6)  | C17—C16—H16   | 121.00     |
| Pb2—N6—C17               | 118.7 (5)  | C18—C19—H19   | 121.00     |

| C13—N6—C17   | 118.4 (8)             | С20—С19—Н19  | 121.00     |
|--|-----------------------|--|------------|
| Pb2—N7—C18   | 118.9 (6)             | С19—С20—Н20  | 119.00     |
| Pb2—N7—C22   | 119.8 (7)             | C21—C20—H20  | 119.00     |
| C18—N7—C22   | 120.7 (9)             | C20—C21—H21  | 121.00     |
| O10—N8—O11   | 117.3 (9)             | C22—C21—H21  | 121.00     |
| O10-N8-O12   | 121.4 (10)            | N7—C22—H22   | 119.00     |
| 011-N8-012   | 121.3 (9)             | C21—C22—H22  | 119.00     |
| N1—C2—C1   | 117.2 (9)             |  |            |
|  |                       |  |            |
| O2—Pb1—O1—N3   | -6.2 (6)              | O11—Pb2—N7—C22   | 87.4 (8)   |
| O4—Pb1—O1—N3   | 127.9 (6)             | N6—Pb2—N7—C22  | 176.5 (8)  |
| O5—Pb1—O1—N3   | 168.9 (7)             | O12 <sup>i</sup> —Pb2—N7—C22   | 11.4 (8)   |
| N1—Pb1—O1—N3   | -66.3 (7)             | $O12^{i}$ —Pb2—N6—C13  | -155.8 (6) |
| N2—Pb1—O1—N3   | -118.2(7)             | 09—Pb2—N6—C17  | -83.2 (6)  |
| $O4^{ii}$ —Pb1—O1—N3   | 54.0 (7)              | O10—Pb2—N6—C17   | 116.3 (6)  |
| 01—Pb1— $02$ —N3   | 6.2 (6)               | 011—Pb2—N6—C17   | 69.7 (6)   |
| 04—Pb1— $02$ —N3   | -65.2(7)              | N7—Pb2—N6—C17  | -1.8(6)    |
| 05-Pb1-02-N3   | -0.8(8)               | $O12^{i}$ Pb2 N6 C17   | 26.5 (8)   |
| 08—Pb1— $02$ —N3   | 176.8 (5)             | O9-Pb2-N7-C18  | 80.0 (6)   |
| N1—Pb1— $O2$ — $N3$  | 132 1 (6)             | 010 Pb2 N7 C18   | -594(7)    |
| $N_2$ —Pb1— $\Omega_2$ — $N_3$   | 69 1 (6)              | $011 - Pb^2 - N7 - C18$  | -83.6(6)   |
| $\Omega_{4i}^{ii}$ Pb1 $\Omega_{2}^{ii}$ N3  | -122.8(6)             | $N6_{Pb}2_{N7}C18$   | 5 5 (6)    |
| 01 - Pb1 - 04 - N4   | 73 9 (7)              | $O12^{i}$ Pb2 N7 C18   | -1596(6)   |
| $\Omega^2$ _Pb1_ $\Omega^4$ _N4  | 122 3 (6)             | $09_{Pb2}$ N6_C13  | 945(7)     |
| 05-Pb1-04-N4   | 77(6)                 | 010 Pb2 N6 C13   | -66.0(7)   |
| 08—Pb1— $04$ —N4   | -929(7)               | 011 - Pb2 - N6 - C13   | -1126(7)   |
| $N1$ _Pb1_04_N4  | -85.2(8)              | $N7_Pb2_N6_C13$  | 175.9(7)   |
| N2 - Pb1 - O4 - N4   | -4.7(8)               | 09 - Pb2 - N7 - C22  | -1090(8)   |
| $\Omega 4^{ii}$ Pb1 $\Omega 4$ N4  | -1732(7)              | $0^{-1}0^{-$ | 111.6 (8)  |
| $\begin{array}{cccccccccccccccccccccccccccccccccccc$   | -113.0(3)             | Ph1 O1 N3 O3   | -1681(10)  |
| $O_2 Pb1 O_4 Pb1^{ii}$   | -64.5(5)              | Pb1 01 N3 02   | 100.1(10)  |
| 02-101-04-101  | -1791(6)              | Pb1 = 02 = N3 = 01   | -10.8(10)  |
| $O_{3}$ $D_{1}$ $O_{4}$ $D_{1}$ $D_{1}$  | 80.3 (3)              | Pb1 = 02 = N3 = 03   | 168.4(10)  |
| N1 Pb1 $O4$ Pb1 <sup>ii</sup>  | 80.3 (3)              | $Pb1^{ii} O4 N4 O5$  | -1784(10)  |
| N2 Pb1 $O4$ Pb1 <sup>ii</sup>  | 1685(3)               | $\frac{101 - 04 - 14 - 05}{101 - 04}$  | 178.4(13)  |
| $\Lambda^{4i}$ <b>Pb1</b> $\Omega^{4}$ <b>Pb1</b>  | 100.3(3)              | Pb1  O4  N4  O5  | -12.8(11)  |
| $O_{1}^{11} = O_{1}^{11} = O_{$ | -0.0(3)               | $\frac{101-04-104-05}{2}$  | 12.8(10)   |
| O1 - F01 - O4 - F01  | -97.2(4)<br>-128.7(4) | P01 - 04 - N4 - 00   | 2(2)       |
| $O_2 = FUI = O_4 = FUI$  | -136.7(4)             | P01 - 05 - N4 - 04   | 15.9(11)   |
| $O4^{-1}$ PD1 <sup>-1</sup> O4 PD1   | 0.0(3)                | P01 - 03 - N4 - 00   | -100.4(10) |
| $O_3^{\text{m}}$ PD1 <sup>m</sup> $O_4$ PD1  | -0.7(3)               | PUI = 08 = N5 = 07   | 81.2(9)    |
| $V8^{"}$ PDI" $V4$ PDI   | /5.2 (3)              | Pb1 - 08 - N5 - 07   | -99.6 (10) |
| $NI^{*} - PbI^{*} - 04 - PbI$  | 138.0(3)              | Pb2  | 1//.8 (6)  |
| $O1^{\mu}$ PD1 <sup><math>\mu</math></sup> O4 N4   | 6/.4(18)              | Pb2 = 0.0 NS = 0.11  | -1.4(11)   |
| $O_2^{\prime\prime}$ PD1 $^{\prime\prime}$ O4 N4   | 20.0(17)              | PD2  | 14.9 (10)  |
| $U4^{\mu}$ $Pb1^{\mu}$ $U4^{\mu}$ $N4$   | 164.7 (18)            | Pb2-010-N8-012   | -166.7 (9) |
| $O3^{\mu}$ $Pb1^{\mu}$ $O4$ $N4$   | 164.0 (17)            | Pb2-011-N8-010   | -15.9(10)  |
| $O8^{\circ}$ $Pb1^{\circ}$ $O4$ $N4$   | -120.2 (17)           | Pb2—O11—N8—O12   | 165.7 (8)  |
| $N1^{n}$ —Pb1 <sup>n</sup> —O4—N4  | -56.7 (18)            | Pb2 <sup>1</sup> —O12—N8—O10   | 129.4 (9)  |

| O1—Pb1—O5—N4   | -125.3 (7)            | Pb2 <sup>i</sup>   | -52.2 (11)            |
|--|-----------------------|--|-----------------------|
| O2—Pb1—O5—N4   | -120.0 (7)            | Pb1—N1—C2—C1   | -20.6 (11)            |
| O4—Pb1—O5—N4   | -7.6 (6)              | C6—N1—C2—C1  | -179.4 (8)            |
| O8—Pb1—O5—N4   | 61.6 (7)              | Pb1—N1—C2—C3   | 158.7 (7)             |
| N1—Pb1—O5—N4   | 121.8 (6)             | C6—N1—C2—C3  | -0.1 (13)             |
| N2—Pb1—O5—N4   | 160.8 (8)             | C2—N1—C6—C5  | 1.3 (13)              |
| O4 <sup>ii</sup> —Pb1—O5—N4  | -8.5 (8)              | Pb1—N1—C6—C7   | 22.2 (10)             |
| O2—Pb1—O8—N5   | 141.4 (7)             | C2—N1—C6—C7  | -178.4 (8)            |
| O4—Pb1—O8—N5   | 4.4 (7)               | Pb1—N1—C6—C5   | -158.2 (7)            |
| O5—Pb1—O8—N5   | -40.4 (7)             | C11—N2—C7—C6   | -178.7 (9)            |
| N1—Pb1—O8—N5   | -170.4(8)             | Pb1—N2—C7—C8   | 179.4 (7)             |
| N2—Pb1—O8—N5   | -111.6 (7)            | C7—N2—C11—C10  | -5.9 (16)             |
| O4 <sup>ii</sup> —Pb1—O8—N5  | 77.1 (7)              | Pb1—N2—C11—C10   | 178.3 (8)             |
| O8—Pb1—N1—C6   | 92.2 (6)              | Pb1—N2—C7—C6   | -3.1(11)              |
| N2—Pb1—N1—C6   | -16.8(6)              | C11—N2—C7—C8   | 3.7 (14)              |
| $O4^{ii}$ —Pb1—N1—C6   | 159.2 (6)             | Pb2—N6—C17—C16   | 178.1 (7)             |
| 01—Pb1—N2—C7   | 135.5 (7)             | $C_{13}$ N6 $-C_{17}$ $-C_{16}$  | 0.3 (13)              |
| $\Omega_{2}$ Pb1 $N_{2}$ C7  | 90.4 (7)              | Pb2 - N6 - C13 - C14   | -178.6(8)             |
| 04—Pb1—N2—C7   | -1254(7)              | Pb2 - N6 - C13 - C12   | 0.1(12)               |
| 05-Pb1-N2-C7   | -1347(7)              | C17 - N6 - C13 - C12   | 1777(8)               |
| 08—Pb1—N2—C7   | -533(7)               | Pb2 - N6 - C17 - C18   | -16(9)                |
| N1 - Pb1 - N2 - C7   | 99(6)                 | C17 - N6 - C13 - C14   | -0.9(14)              |
| $\Omega_1$ _Pb1_N2_C11   | -48.8(8)              | $C_{13}$ N6 $C_{17}$ $C_{18}$  | -1794(8)              |
| $\Omega^2$ _Pb1_N2_C11   | -93.9(8)              | Pb2 N7 C22 C21   | -1724(9)              |
| 04—Pb1—N2—C11  | 50 2 (8)              | Pb2 = N7 = C18 = C19   | 172.1(9)<br>172.8(7)  |
| 05-Pb1-N2-C11  | 40.9 (8)              | Pb2 = N7 = C18 = C17   | -86(10)               |
| 08—Pb1—N2—C11  | 122 4 (8)             | $C_{18} N_{7} C_{22} C_{21}$   | -1.5(16)              |
| $N1_Pb1_N2_C11$  | -1744(8)              | $C_{22} = N_{7} = C_{18} = C_{17}$   | -1795(9)              |
| $\Omega_5$ _Pb1_N1_C6  | 250(7)                | $C_{22} = N7 - C_{18} - C_{19}$  | 19(13)                |
| $05_{101}$ N1_C2   | -1345(7)              | $C_{22} = R_{1} = C_{10} = C_{10}$   | 1.7(13)               |
| $O_{3}$ $P_{b1}$ $N_{1}$ $C_{2}$   | -67.3(7)              | N1 C2 C3 C4  | -17(16)               |
| $N_2 = Pb_1 = N_1 = C_2$   | -1762(8)              | $C_2 - C_3 - C_4$  | 23(18)                |
| $\Omega 4^{ii}$ Pb1 N1 C2  | -0.2(7)               | $C_2 = C_3 = C_4 = C_3$  | -1.1(17)              |
| $O_1 = D_1 = N_1 = C_2$  | 0.2(7)                | $C_{4} = C_{5} = C_{6} = 0$  | -0.7(14)              |
| $O_1 = O_1 = O_1 = O_2$  | 120.8 (7)<br>85 1 (7) | $C_{4} = C_{5} = C_{6} = C_{7}$  | 1700(0)               |
| $O_2$ $D_1$ $O_1$ $O_2$ $O_3$ $O_4$ $D_5$ $O_1$ $O_2$ $O_3$ $O_4$ $O_5$ $O_2$ $O_3$ $O_4$ $O_5$ $O_2$ $O_3$ $O_4$ $O_5$  | -750(8)               | $C_{4} C_{5} C_{6} C_{7} C_{8}$  | 1/9.0(9)<br>164.3(0)  |
| $O_4 = 101 = 101 = 0.2$  | 75.0 (8)<br>84 5 (7)  | N1 = C6 = C7 = C8  | -13.3(12)             |
| $O_1 = P_1 = N_1 = C_0$  | -737(7)               | 11 - 60 - 67 - 112   | -15.3(12)             |
| $O_1 = O_1 $   | -1154(6)              | $C_{5} = C_{6} = C_{7} = C_{8}$  | 15.4(14)              |
| $O_2$ $P_0 P_1 O_1 O_1 O_1 O_1 O_1 O_1 O_1 O_1 O_1 O$  | -113.4(0)             | $C_{3} = C_{0} = C_{1} = N_{2}$  | -178.0(9)             |
| 010 - 102 - 012 - 108  | -25.8(7)              | $N_{2} = C_{7} = C_{8} = C_{9}$  | -1.2(15)              |
| $V_{11} = F_{02} = O_{12} = N_{03}$  | -23.8(7)              | $N_2 - C_7 - C_8 - C_9$  | -1.3(13)              |
| N7i  D52i  O12  N8   | -70.4(8)<br>-96.6(7)  | $C^{*} = C^{*} = C^{*$ | 1.2(17)               |
| $\frac{1}{10} - \frac{1}{100} - \frac{1}{$ | -165.0(7)             | $C_0 = C_1 $ | 5.2(10)               |
| $0_{7} - r_{02} - 0_{12} - 0_{13}$   | 103.9(0)<br>-74.0(7)  | $C_{7}$ $C_{10}$ $C_{11}$ $N_{2}$ $C_{12}$ $C_{14}$ $C_{15}$   | J.0(10)<br>-178 1(11) |
| 010 - F02 - 09 - N3  | -/4.9(/)              | 12 - 013 - 014 - 015   | -1/0.1(11)            |
| V11-F02-O9-N3  | -131.9(0)             | 10 - 013 - 014 - 015   | 0.3(10)               |
| N7 P P 2 O P 2   | -105.8(0)<br>17(9(7)  | C13 - C14 - C15 - C10  | 0.0(18)               |
| N/—Pb2—O9—N3   | -1/6.8 (/)            | U14-U15-U16-U17  | -1.2 (17)             |

| O12 <sup>i</sup> —Pb2—O9—N5  | 109.0 (6)  | C15—C16—C17—N6  | 0.8 (15)   |
|------------------------------|------------|-----------------|------------|
| O9—Pb2—O10—N8                | -130.6 (6) | C15—C16—C17—C18 | -179.6 (9) |
| O11—Pb2—O10—N8               | -8.6 (6)   | C16—C17—C18—C19 | 5.6 (14)   |
| N6—Pb2—O10—N8                | -100.6 (7) | N6-C17-C18-C19  | -174.7 (8) |
| N7—Pb2—O10—N8                | -41.0(7)   | C16—C17—C18—N7  | -173.0 (8) |
| O12 <sup>i</sup> —Pb2—O10—N8 | 45.9 (7)   | N6—C17—C18—N7   | 6.7 (11)   |
| O9—Pb2—O11—N8                | 130.3 (6)  | C17—C18—C19—C20 | 179.8 (10) |
| O10—Pb2—O11—N8               | 8.6 (6)    | N7-C18-C19-C20  | -1.7 (15)  |
| N6—Pb2—O11—N8                | 87.0 (6)   | C18—C19—C20—C21 | 1.1 (18)   |
| N7—Pb2—O11—N8                | 156.0 (7)  | C19—C20—C21—C22 | -0.7 (19)  |
| O12 <sup>i</sup> —Pb2—O11—N8 | -115.5 (6) | C20—C21—C22—N7  | 0.9 (19)   |
|                              |            |                 |            |

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

## Hydrogen-bond geometry (Å, °)

| <i>D</i> —H··· <i>A</i>               | D—H  | H···A | D···A      | D—H···A |
|---------------------------------------|------|-------|------------|---------|
| C1—H1A····O4 <sup>ii</sup>            | 0.96 | 2.55  | 3.378 (14) | 144     |
| C3—H3…O10 <sup>iii</sup>              | 0.93 | 2.50  | 3.368 (17) | 155     |
| C10—H10····O6 <sup>iv</sup>           | 0.93 | 2.47  | 3.349 (17) | 157     |
| C12—H12A····O10                       | 0.96 | 2.48  | 3.299 (15) | 143     |
| C12—H12B····O7                        | 0.96 | 2.49  | 3.412 (17) | 160     |
| C12—H12 <i>C</i> ···O8 <sup>iii</sup> | 0.96 | 2.59  | 3.516 (14) | 162     |
| С19—Н19…О9 <sup>v</sup>               | 0.93 | 2.45  | 3.253 (13) | 145     |
| C21—H21···O1 <sup>iv</sup>            | 0.93 | 2.42  | 3.212 (15) | 143     |
| C22—H22…O12 <sup>i</sup>              | 0.93 | 2.53  | 3.268 (15) | 137     |

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