

# Di- $\mu$ -aqua-bis( $\mu$ -pyridazine-4-carboxylato- $\kappa^2 N:N'$ )bis[triaqua-(pyridazine-4-carboxylato- $\kappa^2 O,O'$ )-lead(II)] dihydrate

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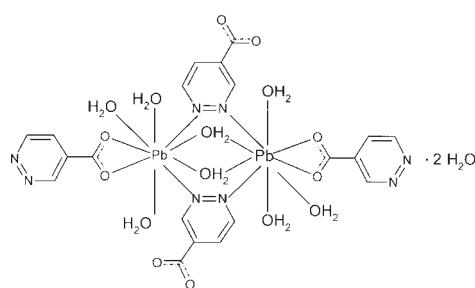
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Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(C-C) = 0.008$  Å;  $R$  factor = 0.045;  $wR$  factor = 0.122; data-to-parameter ratio = 17.8.

The structure of the title compound,  $[Pb_2(C_5H_3N_2O_2)_4 \cdot (H_2O)_6] \cdot 2H_2O$ , is composed of dimeric molecules in which two symmetry-related  $Pb^{2+}$  ions are bridged by a pair of two pyridazine-4-carboxylate ligand molecules *via* both heterocyclic N atoms and two water O atoms. Each  $Pb^{2+}$  ion is also coordinated by two carboxylate O atoms and three water O atoms, leading to a highly irregular coordination polyhedron around  $Pb^{2+}$ . The dimers are interconnected by hydrogen bonds between coordinated and uncoordinated water molecules and the carboxylate O atoms. O—H···N interactions are also present.

## Related literature

For the crystal structure of pyridazine-4-carboxylic acid hydrochloride, see: Starosta & Leciejewicz (2008). Centrosymmetric dimeric molecules were reported in the structure of a calcium(II) complex with pyridazine-3-dicarboxylate and water ligands (Starosta & Leciejewicz, 2007) and an uranyl complex with the same ligands (Leciejewicz & Starosta, 2009). Each dimer shows a different bridging mode.



## Experimental

### Crystal data

$[Pb_2(C_5H_3N_2O_2)_4(H_2O)_6] \cdot 2H_2O$	$\gamma = 102.85 (3)^\circ$
$M_r = 1086.92$	$V = 790.4 (3) \text{ \AA}^3$
Triclinic, $P\bar{1}$	$Z = 1$
$a = 7.0762 (14) \text{ \AA}$	Mo $K\alpha$ radiation
$b = 9.2967 (19) \text{ \AA}$	$\mu = 10.73 \text{ mm}^{-1}$
$c = 12.830 (3) \text{ \AA}$	$T = 293 \text{ K}$
$\alpha = 92.05 (3)^\circ$	$0.35 \times 0.18 \times 0.03 \text{ mm}$
$\beta = 105.13 (3)^\circ$	

### Data collection

Kuma KM-4 four-circle diffractometer	4512 independent reflections
Absorption correction: analytical ( <i>CrysAlis RED</i> ; Oxford Diffraction, 2008)	3958 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.254$ , $T_{\max} = 0.762$	$R_{\text{int}} = 0.016$
4862 measured reflections	3 standard reflections every 200 reflections
	intensity decay: 1.2%

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.045$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.122$	$\Delta\rho_{\max} = 5.63 \text{ e \AA}^{-3}$
$S = 1.06$	$\Delta\rho_{\min} = -3.77 \text{ e \AA}^{-3}$
4512 reflections	15 restraints
253 parameters	

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O4—H41···O12 <sup>i</sup>	0.86 (2)	1.93 (2)	2.736 (6)	154 (4)
O4—H42···O12 <sup>ii</sup>	0.87 (2)	1.92 (4)	2.754 (6)	161 (11)
O1—H11···O5	0.86 (2)	2.08 (3)	2.943 (9)	174 (10)
O1—H12···O11 <sup>iii</sup>	0.86 (2)	2.09 (6)	2.849 (7)	146 (9)
O5—H51···N22 <sup>iv</sup>	0.86 (2)	2.23 (5)	3.013 (8)	151 (9)
O5—H52···O21 <sup>v</sup>	0.87 (2)	2.12 (6)	2.897 (8)	149 (10)
O3—H31···O12 <sup>vi</sup>	0.86 (2)	2.09 (7)	2.819 (7)	142 (10)
O3—H32···N21 <sup>iv</sup>	0.86 (2)	2.01 (6)	2.794 (7)	151 (10)
O2—H21···O5 <sup>vii</sup>	0.86 (2)	2.13 (5)	2.906 (8)	150 (9)
O2—H22···O11 <sup>ii</sup>	0.86 (2)	2.07 (4)	2.891 (7)	159 (9)

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

Data collection: *KM-4 Software* (Kuma, 1996); cell refinement: *KM-4 Software*; data reduction: *DATAPROC* (Kuma, 2001); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BV2128).

## References

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# supporting information

*Acta Cryst.* (2009). E65, m1291 [https://doi.org/10.1107/S1600536809039658]

## Di- $\mu$ -aqua-bis( $\mu$ -pyridazine-4-carboxylato- $\kappa^2N:N'$ )bis[triaqua(pyridazine-4-carboxylato- $\kappa^2O,O'$ )lead(II)] dihydrate

Wojciech Starosta and Janusz Leciejewicz

### S1. Comment

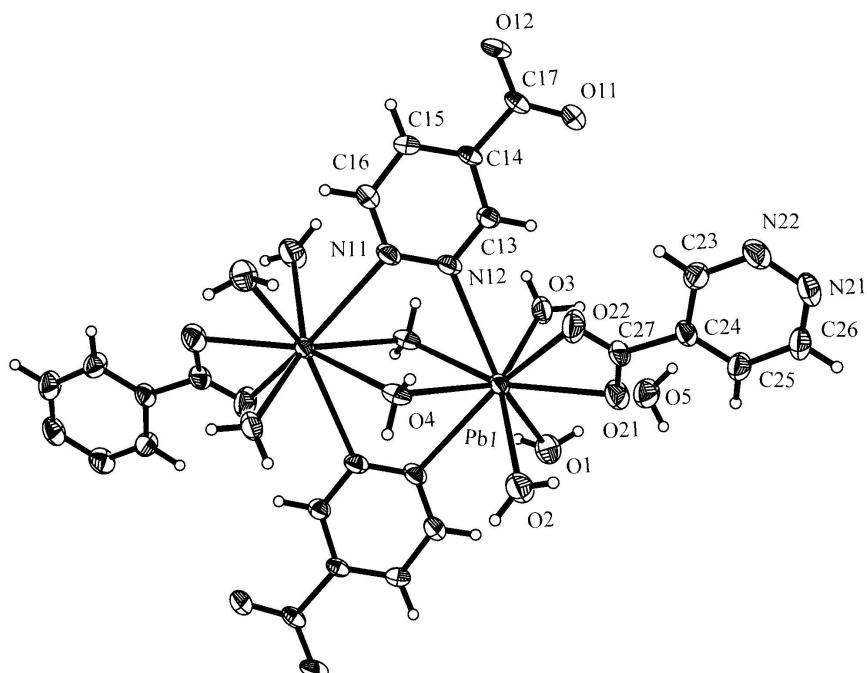
The structure of the title compound (I) is built of dimeric molecules (Fig.1) in which two symmetry related, nine-coordinate  $Pb^{2+}$  ions are chelated by two pairs of pyridazine-4-carboxylate anions *via* their both hetero-ring atoms, each pair showing different chelating mode: one uses both its hetero-ring N atoms to bridge the metal ions, its deprotonated carboxylate O atoms are left inactive in coordination, the other coordinates the  $Pb^{2+}$  ions only *via* carboxylate groups which act as bidentate. In addition, the  $Pb^{2+}$  ions are bridged by a pair of water molecules. Three coordinated water O atoms complete the coordination environment of  $Pb^{2+}$  ions. The coordination geometry around a metal ion is highly irregular. Pyridazine rings are planar with r.m.s. 0.0081 (2) $\text{\AA}$  (ligand 1) and 0.0030 (2) $\text{\AA}$  (ligand 2). The coordinated (C27/O21/O22) and non-coordinated (C17/O11/O12) carboxylic groups make dihedral angles of 5.6 (1) $^\circ$  and 11.9 (1) $^\circ$  with their respective pyridazine rings. A packing diagram of (I) displayed in Fig.2 shows how the dimers are linked by a network of hydrogen bonds. Their relevant geometrical parameters are listed in Table 1.

### S2. Experimental

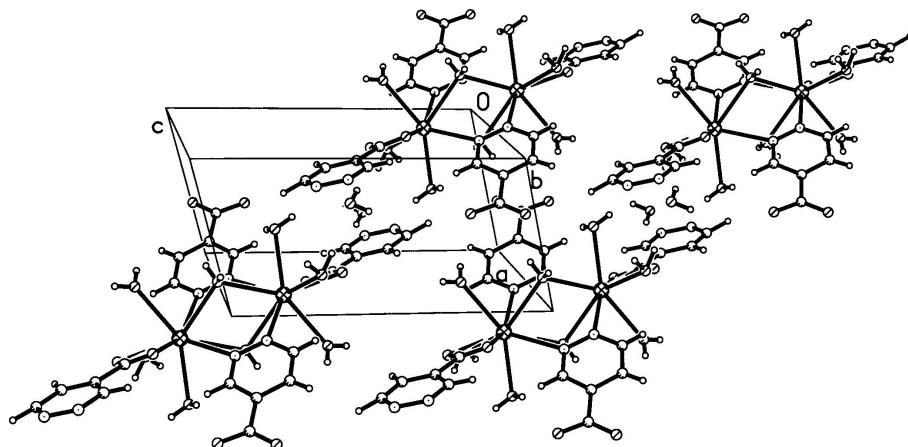
2 Mmol of pyridazine-4-carboxylic acid were dissolved in 100 ml of hot water and boiled for two hours with small excess of  $Pb(OH)_2$ . After cooling to room temperature the mixture was filtered and left to crystallize. Few days later colorless single crystals were found in the mother liquid. They were separated, washed with cold ethanol and dried in air.

### S3. Refinement

H atoms attached to pyridazine-ring C atoms were positioned geometrically and refined with a riding model using AFIX43 instruction. The positions of water H atoms were initially located from Fourier maps and refined isotropically with restraints on O—H distance (0.86  $\text{\AA}$ ) and H—O—H angle.

**Figure 1**

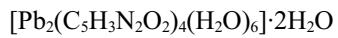
A dimer of (1) with atom labelling scheme and 50% probability displacement ellipsoids.

**Figure 2**

Packing diagram of the structure.

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*Crystal data*



$M_r = 1086.92$

Triclinic,  $P\bar{1}$

$a = 7.0762 (14) \text{ \AA}$

$b = 9.2967 (19) \text{ \AA}$

$c = 12.830 (3) \text{ \AA}$

$\alpha = 92.05 (3)^\circ$

$\beta = 105.13 (3)^\circ$

$\gamma = 102.85 (3)^\circ$

$V = 790.4 (3) \text{ \AA}^3$

$Z = 1$

$F(000) = 516$

$D_x = 2.283 \text{ Mg m}^{-3}$   
 Mo  $K\alpha$  radiation,  $\lambda = 0.71073 \text{ \AA}$   
 Cell parameters from 25 reflections  
 $\theta = 6\text{--}15^\circ$

$\mu = 10.73 \text{ mm}^{-1}$   
 $T = 293 \text{ K}$   
 Plate, colourless  
 $0.35 \times 0.18 \times 0.03 \text{ mm}$

#### Data collection

Kuma KM-4 four-circle diffractometer  
 Radiation source: fine-focus sealed tube  
 Graphite monochromator  
 Profile data from  $\omega/2\theta$  scans  
 Absorption correction: analytical (*CrysAlis RED*; Oxford Diffraction, 2008)  
 $T_{\min} = 0.254$ ,  $T_{\max} = 0.762$   
 4862 measured reflections

4512 independent reflections  
 3958 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.016$   
 $\theta_{\max} = 30.2^\circ$ ,  $\theta_{\min} = 1.7^\circ$   
 $h = 0 \rightarrow 9$   
 $k = -13 \rightarrow 12$   
 $l = -18 \rightarrow 16$   
 3 standard reflections every 200 reflections  
 intensity decay: 1.2%

#### Refinement

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.045$   
 $wR(F^2) = 0.122$   
 $S = 1.06$   
 4512 reflections  
 253 parameters  
 15 restraints  
 Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map  
 Hydrogen site location: inferred from neighbouring sites  
 H atoms treated by a mixture of independent and constrained refinement  
 $w = 1/[\sigma^2(F_o^2) + (0.1029P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.002$   
 $\Delta\rho_{\max} = 5.63 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -3.77 \text{ e \AA}^{-3}$

#### 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 ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
Pb1	0.11555 (3)	0.048890 (18)	0.675973 (14)	0.02300 (9)
O11	0.4666 (8)	0.6974 (5)	0.6607 (4)	0.0360 (10)
O4	-0.2317 (7)	0.0086 (5)	0.5150 (4)	0.0309 (9)
O21	0.2153 (8)	0.1496 (5)	0.8788 (4)	0.0387 (11)
O1	0.3206 (8)	-0.1283 (6)	0.7946 (4)	0.0395 (11)
H11	0.430 (8)	-0.075 (11)	0.838 (5)	0.047*
H12	0.347 (11)	-0.155 (10)	0.736 (4)	0.047*
N12	0.1246 (8)	0.2794 (5)	0.5304 (4)	0.0265 (9)
N11	0.0425 (8)	0.2525 (5)	0.4237 (4)	0.0280 (10)
O22	0.0922 (10)	0.3005 (6)	0.7692 (4)	0.0452 (13)

O12	0.4408 (7)	0.7751 (4)	0.4972 (4)	0.0331 (9)
C17	0.4044 (8)	0.6811 (6)	0.5601 (5)	0.0247 (10)
C14	0.2716 (7)	0.5309 (5)	0.5091 (4)	0.0208 (9)
C13	0.2351 (9)	0.4143 (6)	0.5710 (5)	0.0249 (10)
H13	0.2916	0.4317	0.6458	0.030*
C15	0.1807 (9)	0.5029 (6)	0.3995 (5)	0.0273 (11)
H15	0.1940	0.5769	0.3532	0.033*
C16	0.0682 (10)	0.3600 (7)	0.3606 (5)	0.0309 (12)
H16	0.0079	0.3389	0.2863	0.037*
C24	0.2220 (9)	0.3860 (6)	0.9548 (5)	0.0273 (11)
C27	0.1684 (10)	0.2701 (6)	0.8588 (5)	0.0297 (11)
O5	0.7080 (10)	0.0331 (7)	0.9439 (4)	0.0471 (13)
H51	0.753 (16)	0.123 (4)	0.975 (6)	0.057*
H52	0.682 (16)	-0.024 (7)	0.993 (5)	0.057*
N21	0.3150 (10)	0.6062 (7)	1.1197 (5)	0.0385 (13)
N22	0.2595 (11)	0.6394 (6)	1.0184 (6)	0.0391 (13)
C25	0.2804 (11)	0.3556 (7)	1.0582 (5)	0.0342 (13)
H23	0.2907	0.2606	1.0748	0.041*
C23	0.2131 (11)	0.5332 (7)	0.9376 (5)	0.0325 (12)
H25	0.1731	0.5571	0.8667	0.039*
C26	0.3247 (13)	0.4707 (9)	1.1396 (6)	0.0396 (15)
H26	0.3631	0.4503	1.2115	0.048*
O3	0.4919 (9)	0.1749 (7)	0.7070 (4)	0.0449 (12)
O2	-0.1694 (9)	-0.0786 (7)	0.7638 (5)	0.0490 (14)
H41	-0.277 (8)	0.082 (6)	0.533 (5)	0.07 (3)*
H42	-0.316 (12)	-0.072 (6)	0.521 (10)	0.07 (3)*
H31	0.529 (18)	0.231 (10)	0.661 (6)	0.07 (4)*
H32	0.557 (16)	0.215 (11)	0.771 (3)	0.08 (4)*
H21	-0.172 (17)	-0.018 (9)	0.815 (6)	0.05 (3)*
H22	-0.259 (10)	-0.160 (6)	0.740 (7)	0.04 (2)*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Pb1	0.02442 (13)	0.01967 (12)	0.02294 (13)	0.00112 (8)	0.00730 (9)	-0.00286 (7)
O11	0.040 (2)	0.026 (2)	0.032 (2)	-0.0024 (18)	0.0027 (19)	-0.0031 (17)
O4	0.029 (2)	0.0177 (18)	0.044 (3)	0.0019 (15)	0.0105 (19)	0.0037 (16)
O21	0.049 (3)	0.029 (2)	0.035 (2)	0.010 (2)	0.008 (2)	-0.0061 (18)
O1	0.041 (3)	0.039 (3)	0.039 (3)	0.012 (2)	0.010 (2)	-0.003 (2)
N12	0.031 (2)	0.0161 (19)	0.032 (2)	0.0016 (17)	0.012 (2)	0.0006 (17)
N11	0.032 (2)	0.0169 (19)	0.032 (2)	0.0005 (17)	0.009 (2)	-0.0032 (17)
O22	0.069 (4)	0.039 (3)	0.025 (2)	0.016 (3)	0.007 (2)	-0.0038 (19)
O12	0.036 (2)	0.0166 (17)	0.046 (3)	-0.0025 (16)	0.016 (2)	0.0023 (16)
C17	0.022 (2)	0.015 (2)	0.036 (3)	0.0028 (17)	0.009 (2)	-0.0044 (18)
C14	0.019 (2)	0.0133 (19)	0.031 (3)	0.0033 (16)	0.0100 (19)	-0.0011 (17)
C13	0.025 (2)	0.020 (2)	0.028 (3)	0.0035 (19)	0.006 (2)	0.0020 (19)
C15	0.030 (3)	0.019 (2)	0.033 (3)	0.003 (2)	0.010 (2)	0.006 (2)
C16	0.032 (3)	0.024 (3)	0.030 (3)	0.000 (2)	0.003 (2)	-0.002 (2)

C24	0.032 (3)	0.023 (2)	0.026 (3)	0.005 (2)	0.007 (2)	-0.0055 (19)
C27	0.038 (3)	0.024 (2)	0.027 (3)	0.004 (2)	0.011 (2)	-0.006 (2)
O5	0.064 (4)	0.042 (3)	0.039 (3)	0.016 (3)	0.017 (3)	0.001 (2)
N21	0.040 (3)	0.037 (3)	0.034 (3)	0.004 (2)	0.009 (2)	-0.013 (2)
N22	0.048 (3)	0.025 (2)	0.042 (3)	0.009 (2)	0.010 (3)	-0.005 (2)
C25	0.048 (4)	0.031 (3)	0.025 (3)	0.013 (3)	0.012 (3)	0.001 (2)
C23	0.044 (3)	0.028 (3)	0.027 (3)	0.013 (2)	0.009 (2)	0.003 (2)
C26	0.052 (4)	0.042 (4)	0.024 (3)	0.010 (3)	0.009 (3)	-0.006 (2)
O3	0.043 (3)	0.051 (3)	0.025 (2)	-0.016 (2)	0.008 (2)	-0.007 (2)
O2	0.045 (3)	0.053 (3)	0.042 (3)	-0.012 (2)	0.023 (2)	-0.011 (2)

*Geometric parameters ( $\text{\AA}$ ,  $^{\circ}$ )*

Pb1—O3	2.578 (6)	C13—H13	0.9300
Pb1—O21	2.593 (5)	C15—C16	1.386 (8)
Pb1—O2	2.638 (6)	C15—H15	0.9300
Pb1—O22	2.647 (5)	C16—H16	0.9300
Pb1—O1	2.688 (6)	C24—C25	1.343 (9)
Pb1—O4	2.707 (5)	C24—C23	1.406 (8)
O11—C17	1.242 (8)	C24—C27	1.517 (8)
O4—H41	0.86 (2)	O5—H51	0.86 (2)
O4—H42	0.87 (2)	O5—H52	0.87 (2)
O21—C27	1.254 (8)	N21—C26	1.308 (10)
O1—H11	0.86 (2)	N21—N22	1.324 (10)
O1—H12	0.86 (2)	N22—C23	1.328 (8)
N12—C13	1.327 (7)	C25—C26	1.390 (9)
N12—N11	1.331 (7)	C25—H23	0.9300
N11—C16	1.320 (8)	C23—H25	0.9300
O22—C27	1.208 (8)	C26—H26	0.9300
O12—C17	1.241 (7)	O3—H31	0.86 (2)
C17—C14	1.514 (7)	O3—H32	0.86 (2)
C14—C15	1.373 (8)	O2—H21	0.86 (2)
C14—C13	1.385 (7)	O2—H22	0.86 (2)
O3—Pb1—O21	79.05 (17)	N12—C13—H13	118.0
O3—Pb1—O2	146.72 (18)	C14—C13—H13	118.0
O21—Pb1—O2	70.88 (18)	C14—C15—C16	117.4 (5)
O3—Pb1—O22	85.5 (2)	C14—C15—H15	121.3
O21—Pb1—O22	49.16 (16)	C16—C15—H15	121.3
O2—Pb1—O22	85.5 (2)	N11—C16—C15	123.2 (6)
O3—Pb1—O1	74.2 (2)	N11—C16—H16	118.4
O21—Pb1—O1	71.28 (16)	C15—C16—H16	118.4
O2—Pb1—O1	82.8 (2)	C25—C24—C23	117.0 (5)
O22—Pb1—O1	119.77 (16)	C25—C24—C27	123.0 (6)
O3—Pb1—O4	137.65 (16)	C23—C24—C27	120.1 (6)
O21—Pb1—O4	132.46 (16)	O22—C27—O21	124.6 (6)
O2—Pb1—O4	75.25 (18)	O22—C27—C24	119.1 (6)
O22—Pb1—O4	96.49 (16)	O21—C27—C24	116.3 (6)

O1—Pb1—O4	135.76 (15)	H51—O5—H52	107 (3)
Pb1—O4—H41	100.9 (17)	C26—N21—N22	120.3 (6)
Pb1—O4—H42	110 (8)	N21—N22—C23	119.1 (6)
H41—O4—H42	107 (3)	C24—C25—C26	117.9 (6)
C27—O21—Pb1	93.8 (4)	C24—C25—H23	121.1
Pb1—O1—H11	110 (8)	C26—C25—H23	121.1
Pb1—O1—H12	87 (7)	N22—C23—C24	122.7 (6)
H11—O1—H12	109 (3)	N22—C23—H25	118.7
C13—N12—N11	118.9 (5)	C24—C23—H25	118.7
C16—N11—N12	119.9 (5)	N21—C26—C25	123.0 (7)
C27—O22—Pb1	92.3 (4)	N21—C26—H26	118.5
O12—C17—O11	126.7 (5)	C25—C26—H26	118.5
O12—C17—C14	116.7 (5)	Pb1—O3—H31	120 (8)
O11—C17—C14	116.6 (5)	Pb1—O3—H32	117 (8)
C15—C14—C13	116.5 (5)	H31—O3—H32	109 (4)
C15—C14—C17	122.0 (5)	Pb1—O2—H21	108 (7)
C13—C14—C17	121.5 (5)	Pb1—O2—H22	127 (6)
N12—C13—C14	124.0 (5)	H21—O2—H22	124 (10)

*Hydrogen-bond geometry (Å, °)*

D—H···A	D—H	H···A	D···A	D—H···A
O4—H41···O12 <sup>i</sup>	0.86 (2)	1.93 (2)	2.736 (6)	154 (4)
O4—H42···O12 <sup>ii</sup>	0.87 (2)	1.92 (4)	2.754 (6)	161 (11)
O1—H11···O5	0.86 (2)	2.08 (3)	2.943 (9)	174 (10)
O1—H12···O11 <sup>iii</sup>	0.86 (2)	2.09 (6)	2.849 (7)	146 (9)
O5—H51···N22 <sup>iv</sup>	0.86 (2)	2.23 (5)	3.013 (8)	151 (9)
O5—H52···O21 <sup>v</sup>	0.87 (2)	2.12 (6)	2.897 (8)	149 (10)
O3—H31···O12 <sup>vi</sup>	0.86 (2)	2.09 (7)	2.819 (7)	142 (10)
O3—H32···N21 <sup>iv</sup>	0.86 (2)	2.01 (6)	2.794 (7)	151 (10)
O2—H21···O5 <sup>vii</sup>	0.86 (2)	2.13 (5)	2.906 (8)	150 (9)
O2—H22···O11 <sup>ii</sup>	0.86 (2)	2.07 (4)	2.891 (7)	159 (9)

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