

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

Bis(μ -2-methylquinolin-8-olato)- κ^3 N,O:O; κ^3 O:N,O-bis[(acetato- κ^2 O,O')]lead(II)

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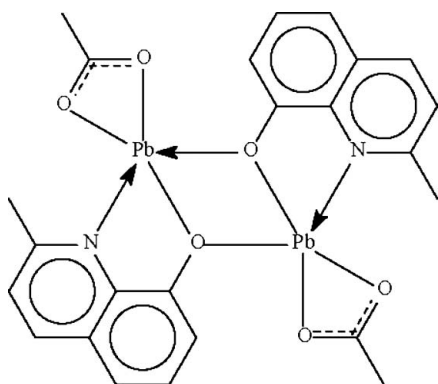
Received 27 January 2009; accepted 29 January 2009

Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.014$ Å; R factor = 0.046; wR factor = 0.115; data-to-parameter ratio = 15.4.

Both independent Pb^{II} atoms in the title compound, $[\text{Pb}_2(\text{C}_{10}\text{H}_8\text{NO})_2(\text{C}_2\text{H}_3\text{O}_2)_2]$, are chelated by acetate and substituted quinolin-8-olate anions; the O atoms of the quinolin-8-olates also bridge to confer a five-coordinate status to each metal center. The geometry approximates a distorted Ψ -*fac* octahedron in which one of the sites is occupied by a stereochemically active lone pair.

Related literature

The structural chemistry of lead(II) 8-hydroxyquinolinates has been reviewed, including bis(μ -acetato)diacetatotetrakis(μ -quinolin-8-olato)tetralead dihydrate (Shahverdizadeh *et al.*, 2008).



Experimental

Crystal data

$[\text{Pb}_2(\text{C}_{10}\text{H}_8\text{NO})_2(\text{C}_2\text{H}_3\text{O}_2)_2]$
 $M_r = 848.82$
 Orthorhombic, *Pbca*
 $a = 13.7421$ (2) Å
 $b = 18.0682$ (3) Å
 $c = 18.6113$ (4) Å

$V = 4621.1$ (1) Å³
 $Z = 8$
 Mo $K\alpha$ radiation
 $\mu = 14.60$ mm⁻¹
 $T = 100$ (2) K
 $0.20 \times 0.10 \times 0.08$ mm

Data collection

Bruker SMART APEX diffractometer
 Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
 $T_{\text{min}} = 0.158$, $T_{\text{max}} = 0.388$
 (expected range = 0.127–0.311)

33016 measured reflections
 4063 independent reflections
 3133 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.090$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.046$
 $wR(F^2) = 0.115$
 $S = 1.45$
 4063 reflections
 264 parameters

192 restraints
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 4.37$ e Å⁻³
 $\Delta\rho_{\text{min}} = -2.70$ e Å⁻³

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *pubCIF* (Westrip, 2009).

We thank Shahid Beheshti University and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2364).

References

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supplementary materials

Acta Cryst. (2009). E65, m260 [doi:10.1107/S1600536809003560]

Bis(μ -2-methylquinolin-8-olato)- $\kappa^3N,O:O;\kappa^3O:N,O$ -bis[(acetato- κ^2O,O')]lead(II)]

G. Mohammadnezhad Sh., M. M. Amini and S. W. Ng

Comment

(type here to add)

Experimental

Lead acetate (0.38 g, 1 mmol) and 2-methyl-8-hydroxyquinoline (0.32 g, 2 mmol) were loaded into a convection tube; the tube was filled with dry methanol and kept at 333 K. Crystals were collected from the side arm after 1 day.

Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.93 to 0.98 Å) and were included in the refinement in the riding model approximation, with $U(H)$ set to 1.2 to 1.5 $U(C)$.

The quinolinyl ring was refined as a rigid group with C—C = 1.39 Å. The crystal diffracted strongly owing to two extremely heavy metal atoms. However, their presence introduced severe absorption problems that could not be corrected analytically as the crystal did not have regular faces. Although a sphere of reflections was measured, multi-scan treatment only marginally improved the quality. The final difference Fourier map had large peaks/deep holes near the lead atoms. The anisotropic displacement factors of the carbon, nitrogen and oxygen atoms had to be restrained to be nearly isotropic.

Figures

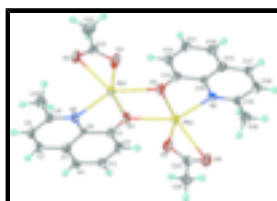


Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of $Pb_2(C_2H_3O_2)_2(C_{10}H_8NO)_2$ at the 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius.

Bis(μ -2-methylquinolin-8-olato)- $\kappa^3N,O:O;\kappa^3O:N,O$ - bis[(acetato- κ^2O,O')]lead(II)]

Crystal data

$[Pb_2(C_{10}H_8NO)_2(C_2H_3O_2)_2]$

$M_r = 848.82$

Orthorhombic, *Pbca*

Hall symbol: -P 2ac 2ab

$a = 13.7421$ (2) Å

$F_{000} = 3136$

$D_x = 2.440$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 6225 reflections

$\theta = 2.2$ – 28.3°

supplementary materials

$b = 18.0682 (3) \text{ \AA}$
 $c = 18.6113 (4) \text{ \AA}$
 $V = 4621.1 (1) \text{ \AA}^3$
 $Z = 8$

$\mu = 14.60 \text{ mm}^{-1}$
 $T = 100 \text{ K}$
Block, yellow
 $0.20 \times 0.10 \times 0.08 \text{ mm}$

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Monochromator: graphite
 $T = 100 \text{ K}$
 ω scans
Absorption correction: Multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.158, T_{\max} = 0.388$
33016 measured reflections

4063 independent reflections
3133 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.090$
 $\theta_{\max} = 25.0^\circ$
 $\theta_{\min} = 2.2^\circ$
 $h = -16 \rightarrow 16$
 $k = -21 \rightarrow 21$
 $l = -22 \rightarrow 21$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.046$
 $wR(F^2) = 0.115$
 $S = 1.45$
4063 reflections
264 parameters
192 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
 $w = 1/[\sigma^2(F_o^2) + (0.0388P)^2 + 1P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 4.37 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -2.70 \text{ e \AA}^{-3}$
Extinction correction: none

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

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|-------------|-------------|-------------|----------------------------------|
| Pb1 | 0.28159 (3) | 0.37522 (2) | 0.52835 (2) | 0.01649 (15) |
| Pb2 | 0.56385 (3) | 0.38486 (2) | 0.48211 (2) | 0.01580 (14) |
| O1 | 0.3933 (6) | 0.4289 (4) | 0.4554 (4) | 0.0190 (18) |
| O2 | 0.4542 (5) | 0.3401 (4) | 0.5634 (4) | 0.0193 (17) |
| O3 | 0.3217 (6) | 0.4747 (4) | 0.6014 (5) | 0.029 (2) |
| O4 | 0.1631 (6) | 0.4725 (4) | 0.5938 (5) | 0.027 (2) |
| O5 | 0.5071 (6) | 0.2851 (4) | 0.4186 (4) | 0.027 (2) |
| O6 | 0.6648 (6) | 0.2738 (4) | 0.3994 (5) | 0.030 (2) |
| C1 | 0.3673 (3) | 0.4758 (3) | 0.4007 (3) | 0.017 (2) |
| C2 | 0.4354 (3) | 0.5134 (3) | 0.3593 (3) | 0.021 (3) |
| H2 | 0.5029 | 0.5076 | 0.3689 | 0.026* |
| C3 | 0.4048 (3) | 0.5595 (3) | 0.3040 (3) | 0.020 (3) |

| | | | | |
|------|-------------|--------------|------------|-----------|
| H3 | 0.4514 | 0.5852 | 0.2757 | 0.024* |
| C4 | 0.3061 (4) | 0.5680 (3) | 0.2900 (3) | 0.019 (3) |
| H4 | 0.2852 | 0.5996 | 0.2522 | 0.023* |
| C5 | 0.2380 (3) | 0.53044 (19) | 0.3314 (2) | 0.021 (3) |
| C6 | 0.2686 (3) | 0.48434 (18) | 0.3868 (2) | 0.015 (2) |
| N1 | 0.2004 (3) | 0.4467 (2) | 0.4282 (2) | 0.016 (2) |
| C9 | 0.1017 (3) | 0.4552 (3) | 0.4142 (3) | 0.017 (2) |
| C8 | 0.0711 (3) | 0.5013 (3) | 0.3589 (3) | 0.022 (3) |
| H8 | 0.0036 | 0.5072 | 0.3494 | 0.026* |
| C7 | 0.1392 (3) | 0.5389 (3) | 0.3175 (3) | 0.019 (3) |
| H7 | 0.1183 | 0.5705 | 0.2797 | 0.023* |
| C10 | 0.0363 (9) | 0.4109 (6) | 0.4591 (7) | 0.025 (3) |
| H10A | 0.0535 | 0.3585 | 0.4547 | 0.038* |
| H10B | -0.0310 | 0.4183 | 0.4432 | 0.038* |
| H10C | 0.0427 | 0.4264 | 0.5093 | 0.038* |
| C11 | 0.4812 (3) | 0.2885 (3) | 0.6133 (3) | 0.017 (2) |
| C12 | 0.4148 (3) | 0.2545 (3) | 0.6589 (3) | 0.024 (3) |
| H12 | 0.3475 | 0.2659 | 0.6554 | 0.028* |
| C13 | 0.4469 (3) | 0.2036 (3) | 0.7097 (3) | 0.018 (3) |
| H13 | 0.4016 | 0.1803 | 0.7409 | 0.022* |
| C14 | 0.5454 (4) | 0.1868 (3) | 0.7148 (3) | 0.022 (3) |
| H14 | 0.5673 | 0.1521 | 0.7496 | 0.026* |
| C15 | 0.6118 (3) | 0.22089 (19) | 0.6692 (2) | 0.018 (3) |
| C16 | 0.5797 (3) | 0.27173 (19) | 0.6184 (2) | 0.013 (2) |
| N2 | 0.6460 (3) | 0.3058 (2) | 0.5728 (2) | 0.015 (2) |
| C19 | 0.7445 (3) | 0.2890 (3) | 0.5779 (3) | 0.017 (3) |
| C18 | 0.7766 (3) | 0.2382 (3) | 0.6287 (3) | 0.021 (3) |
| H18 | 0.8439 | 0.2267 | 0.6322 | 0.026* |
| C17 | 0.7102 (3) | 0.2041 (3) | 0.6743 (3) | 0.017 (2) |
| H17 | 0.7322 | 0.1694 | 0.7090 | 0.021* |
| C20 | 0.8077 (9) | 0.3291 (6) | 0.5282 (7) | 0.024 (3) |
| H20A | 0.8022 | 0.3824 | 0.5372 | 0.036* |
| H20B | 0.7880 | 0.3184 | 0.4787 | 0.036* |
| H20C | 0.8752 | 0.3134 | 0.5355 | 0.036* |
| C21 | 0.2377 (9) | 0.5005 (6) | 0.6175 (6) | 0.019 (3) |
| C22 | 0.2349 (10) | 0.5681 (6) | 0.6657 (7) | 0.031 (3) |
| H22A | 0.2560 | 0.6116 | 0.6385 | 0.047* |
| H22B | 0.2785 | 0.5605 | 0.7067 | 0.047* |
| H22C | 0.1683 | 0.5757 | 0.6831 | 0.047* |
| C23 | 0.5803 (8) | 0.2518 (6) | 0.3911 (6) | 0.017 (3) |
| C24 | 0.5582 (9) | 0.1846 (6) | 0.3472 (6) | 0.023 (3) |
| H24A | 0.5722 | 0.1948 | 0.2965 | 0.035* |
| H24B | 0.4894 | 0.1717 | 0.3526 | 0.035* |
| H24C | 0.5987 | 0.1432 | 0.3637 | 0.035* |

Atomic displacement parameters (\AA^2)

U^{11}

U^{22}

U^{33}

U^{12}

U^{13}

U^{23}

supplementary materials

| | | | | | | |
|-----|------------|------------|------------|---------------|--------------|--------------|
| Pb1 | 0.0121 (3) | 0.0160 (2) | 0.0214 (3) | -0.00051 (15) | 0.00086 (17) | 0.00142 (16) |
| Pb2 | 0.0108 (2) | 0.0164 (2) | 0.0201 (3) | 0.00012 (15) | 0.00039 (17) | 0.00064 (17) |
| O1 | 0.011 (4) | 0.024 (4) | 0.023 (4) | 0.004 (3) | 0.002 (3) | 0.008 (3) |
| O2 | 0.013 (4) | 0.021 (4) | 0.023 (4) | 0.003 (3) | 0.002 (3) | 0.004 (3) |
| O3 | 0.013 (5) | 0.027 (4) | 0.047 (5) | -0.002 (3) | 0.000 (4) | -0.013 (4) |
| O4 | 0.018 (5) | 0.031 (4) | 0.033 (5) | 0.003 (3) | -0.001 (4) | -0.008 (3) |
| O5 | 0.018 (4) | 0.027 (4) | 0.034 (5) | -0.005 (3) | -0.001 (4) | -0.012 (3) |
| O6 | 0.020 (5) | 0.034 (4) | 0.036 (5) | 0.001 (4) | 0.000 (4) | -0.004 (4) |
| C1 | 0.013 (6) | 0.018 (5) | 0.019 (6) | -0.004 (4) | 0.004 (4) | -0.001 (4) |
| C2 | 0.018 (6) | 0.018 (5) | 0.028 (6) | -0.003 (4) | 0.001 (5) | 0.002 (4) |
| C3 | 0.018 (6) | 0.022 (5) | 0.020 (6) | -0.005 (4) | 0.002 (5) | -0.001 (4) |
| C4 | 0.020 (6) | 0.018 (5) | 0.020 (6) | -0.002 (4) | -0.006 (5) | -0.001 (4) |
| C5 | 0.022 (6) | 0.014 (5) | 0.025 (6) | 0.003 (4) | 0.000 (5) | -0.002 (4) |
| C6 | 0.006 (5) | 0.018 (5) | 0.020 (6) | 0.003 (4) | 0.005 (4) | -0.002 (4) |
| N1 | 0.018 (5) | 0.012 (4) | 0.018 (5) | 0.010 (3) | -0.003 (4) | 0.002 (3) |
| C9 | 0.011 (5) | 0.017 (5) | 0.024 (6) | 0.001 (4) | 0.001 (4) | -0.007 (4) |
| C8 | 0.014 (6) | 0.024 (5) | 0.027 (6) | 0.003 (4) | -0.005 (5) | 0.001 (4) |
| C7 | 0.016 (6) | 0.018 (5) | 0.023 (6) | -0.001 (4) | -0.004 (5) | 0.004 (4) |
| C10 | 0.020 (6) | 0.027 (5) | 0.029 (6) | -0.002 (5) | 0.000 (5) | -0.002 (5) |
| C11 | 0.014 (6) | 0.024 (5) | 0.014 (5) | 0.003 (4) | 0.001 (4) | 0.000 (4) |
| C12 | 0.020 (6) | 0.027 (5) | 0.025 (6) | -0.001 (4) | 0.002 (5) | -0.002 (5) |
| C13 | 0.016 (6) | 0.019 (5) | 0.020 (6) | -0.007 (4) | 0.003 (5) | 0.000 (4) |
| C14 | 0.026 (6) | 0.023 (5) | 0.016 (6) | -0.004 (5) | 0.002 (5) | 0.001 (4) |
| C15 | 0.020 (6) | 0.014 (5) | 0.021 (6) | 0.002 (4) | -0.001 (5) | -0.005 (4) |
| C16 | 0.012 (6) | 0.016 (5) | 0.012 (5) | -0.003 (4) | -0.001 (4) | -0.005 (4) |
| N2 | 0.020 (5) | 0.013 (4) | 0.013 (5) | 0.005 (4) | -0.001 (4) | 0.001 (3) |
| C19 | 0.013 (6) | 0.019 (5) | 0.018 (6) | -0.001 (4) | 0.002 (4) | 0.000 (4) |
| C18 | 0.016 (6) | 0.025 (5) | 0.023 (6) | 0.005 (4) | -0.002 (5) | 0.000 (4) |
| C17 | 0.017 (6) | 0.016 (5) | 0.019 (6) | 0.003 (4) | -0.002 (5) | 0.004 (4) |
| C20 | 0.020 (6) | 0.021 (5) | 0.031 (6) | 0.002 (4) | -0.003 (5) | -0.001 (4) |
| C21 | 0.017 (6) | 0.017 (5) | 0.022 (6) | -0.002 (4) | -0.001 (5) | 0.006 (4) |
| C22 | 0.036 (7) | 0.022 (5) | 0.036 (7) | -0.002 (5) | 0.005 (6) | -0.003 (5) |
| C23 | 0.015 (6) | 0.018 (5) | 0.019 (5) | -0.003 (4) | 0.000 (5) | 0.003 (4) |
| C24 | 0.023 (6) | 0.023 (5) | 0.023 (6) | -0.001 (5) | -0.001 (5) | 0.000 (4) |

Geometric parameters (Å, °)

| | | | |
|--------|-----------|----------|--------|
| Pb1—O1 | 2.267 (8) | C7—H7 | 0.9500 |
| Pb1—O3 | 2.321 (8) | C10—H10A | 0.9800 |
| Pb1—N1 | 2.527 (4) | C10—H10B | 0.9800 |
| Pb1—O2 | 2.541 (7) | C10—H10C | 0.9800 |
| Pb1—O4 | 2.688 (8) | C11—C12 | 1.3900 |
| Pb2—O2 | 2.283 (7) | C11—C16 | 1.3900 |
| Pb2—O5 | 2.292 (7) | C12—C13 | 1.3900 |
| Pb2—N2 | 2.482 (4) | C12—H12 | 0.9500 |
| Pb2—O1 | 2.525 (7) | C13—C14 | 1.3900 |
| Pb2—O4 | 6.095 (8) | C13—H13 | 0.9500 |
| O1—C1 | 1.372 (8) | C14—C15 | 1.3900 |
| O2—C11 | 1.366 (8) | C14—H14 | 0.9500 |

| | | | |
|------------|-------------|---------------|-------------|
| O3—C21 | 1.280 (14) | C15—C16 | 1.3900 |
| O4—C21 | 1.226 (14) | C15—C17 | 1.3900 |
| O5—C23 | 1.279 (13) | C16—N2 | 1.3900 |
| O6—C23 | 1.237 (13) | N2—C19 | 1.3900 |
| C1—C2 | 1.3900 | C19—C18 | 1.3900 |
| C1—C6 | 1.3900 | C19—C20 | 1.460 (12) |
| C2—C3 | 1.3900 | C18—C17 | 1.3900 |
| C2—H2 | 0.9500 | C18—H18 | 0.9500 |
| C3—C4 | 1.3900 | C17—H17 | 0.9500 |
| C3—H3 | 0.9500 | C20—H20A | 0.9800 |
| C4—C5 | 1.3900 | C20—H20B | 0.9800 |
| C4—H4 | 0.9500 | C20—H20C | 0.9800 |
| C5—C6 | 1.3900 | C21—C22 | 1.515 (16) |
| C5—C7 | 1.3900 | C22—H22A | 0.9800 |
| C6—N1 | 1.3900 | C22—H22B | 0.9800 |
| N1—C9 | 1.3900 | C22—H22C | 0.9800 |
| C9—C8 | 1.3900 | C23—C24 | 1.494 (15) |
| C9—C10 | 1.465 (12) | C24—H24A | 0.9800 |
| C8—C7 | 1.3900 | C24—H24B | 0.9800 |
| C8—H8 | 0.9500 | C24—H24C | 0.9800 |
| O1—Pb1—O3 | 81.9 (3) | C9—C10—H10A | 109.5 |
| O1—Pb1—N1 | 68.8 (2) | C9—C10—H10B | 109.5 |
| O3—Pb1—N1 | 98.1 (2) | H10A—C10—H10B | 109.5 |
| O1—Pb1—O2 | 68.2 (2) | C9—C10—H10C | 109.5 |
| O3—Pb1—O2 | 79.7 (3) | H10A—C10—H10C | 109.5 |
| N1—Pb1—O2 | 136.80 (19) | H10B—C10—H10C | 109.5 |
| O1—Pb1—O4 | 113.7 (2) | O2—C11—C12 | 122.6 (4) |
| O3—Pb1—O4 | 51.1 (3) | O2—C11—C16 | 117.4 (4) |
| N1—Pb1—O4 | 74.5 (2) | C12—C11—C16 | 120.0 |
| O2—Pb1—O4 | 127.8 (2) | C11—C12—C13 | 120.0 |
| O2—Pb2—O5 | 80.7 (3) | C11—C12—H12 | 120.0 |
| O2—Pb2—N2 | 69.3 (2) | C13—C12—H12 | 120.0 |
| O5—Pb2—N2 | 93.0 (2) | C14—C13—C12 | 120.0 |
| O2—Pb2—O1 | 68.3 (2) | C14—C13—H13 | 120.0 |
| O5—Pb2—O1 | 80.3 (3) | C12—C13—H13 | 120.0 |
| N2—Pb2—O1 | 137.5 (2) | C13—C14—C15 | 120.0 |
| O2—Pb2—O4 | 43.12 (19) | C13—C14—H14 | 120.0 |
| O5—Pb2—O4 | 94.2 (2) | C15—C14—H14 | 120.0 |
| N2—Pb2—O4 | 109.19 (13) | C14—C15—C16 | 120.0 |
| O1—Pb2—O4 | 31.4 (2) | C14—C15—C17 | 120.0 |
| C1—O1—Pb1 | 122.2 (5) | C16—C15—C17 | 120.0 |
| C1—O1—Pb2 | 125.6 (5) | N2—C16—C15 | 120.0 |
| Pb1—O1—Pb2 | 112.1 (3) | N2—C16—C11 | 120.0 |
| C11—O2—Pb2 | 120.9 (5) | C15—C16—C11 | 120.0 |
| C11—O2—Pb1 | 126.7 (5) | C16—N2—C19 | 120.0 |
| Pb2—O2—Pb1 | 111.0 (3) | C16—N2—Pb2 | 111.83 (17) |
| C21—O3—Pb1 | 101.8 (7) | C19—N2—Pb2 | 127.96 (17) |
| C21—O4—Pb1 | 85.8 (7) | N2—C19—C18 | 120.0 |
| C21—O4—Pb2 | 58.3 (7) | N2—C19—C20 | 115.3 (6) |

supplementary materials

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|---------------|-------------|---------------|------------|
| C23—O5—Pb2 | 108.0 (7) | C18—C19—C20 | 124.7 (6) |
| O1—C1—C2 | 122.5 (4) | C17—C18—C19 | 120.0 |
| O1—C1—C6 | 117.5 (4) | C17—C18—H18 | 120.0 |
| C2—C1—C6 | 120.0 | C19—C18—H18 | 120.0 |
| C3—C2—C1 | 120.0 | C18—C17—C15 | 120.0 |
| C3—C2—H2 | 120.0 | C18—C17—H17 | 120.0 |
| C1—C2—H2 | 120.0 | C15—C17—H17 | 120.0 |
| C2—C3—C4 | 120.0 | C19—C20—H20A | 109.5 |
| C2—C3—H3 | 120.0 | C19—C20—H20B | 109.5 |
| C4—C3—H3 | 120.0 | H20A—C20—H20B | 109.5 |
| C5—C4—C3 | 120.0 | C19—C20—H20C | 109.5 |
| C5—C4—H4 | 120.0 | H20A—C20—H20C | 109.5 |
| C3—C4—H4 | 120.0 | H20B—C20—H20C | 109.5 |
| C6—C5—C4 | 120.0 | O4—C21—O3 | 121.3 (10) |
| C6—C5—C7 | 120.0 | O4—C21—C22 | 121.6 (11) |
| C4—C5—C7 | 120.0 | O3—C21—C22 | 117.1 (11) |
| N1—C6—C5 | 120.0 | C21—C22—H22A | 109.5 |
| N1—C6—C1 | 120.0 | C21—C22—H22B | 109.5 |
| C5—C6—C1 | 120.0 | H22A—C22—H22B | 109.5 |
| C6—N1—C9 | 120.0 | C21—C22—H22C | 109.5 |
| C6—N1—Pb1 | 111.19 (17) | H22A—C22—H22C | 109.5 |
| C9—N1—Pb1 | 128.68 (17) | H22B—C22—H22C | 109.5 |
| C8—C9—N1 | 120.0 | O6—C23—O5 | 122.4 (10) |
| C8—C9—C10 | 124.4 (6) | O6—C23—C24 | 121.3 (11) |
| N1—C9—C10 | 115.6 (6) | O5—C23—C24 | 116.2 (10) |
| C9—C8—C7 | 120.0 | C23—C24—H24A | 109.5 |
| C9—C8—H8 | 120.0 | C23—C24—H24B | 109.5 |
| C7—C8—H8 | 120.0 | H24A—C24—H24B | 109.5 |
| C8—C7—C5 | 120.0 | C23—C24—H24C | 109.5 |
| C8—C7—H7 | 120.0 | H24A—C24—H24C | 109.5 |
| C5—C7—H7 | 120.0 | H24B—C24—H24C | 109.5 |
| O3—Pb1—O1—C1 | -96.4 (6) | C2—C1—C6—N1 | 180.0 |
| N1—Pb1—O1—C1 | 5.6 (5) | O1—C1—C6—C5 | -179.2 (6) |
| O2—Pb1—O1—C1 | -178.6 (7) | C2—C1—C6—C5 | 0.0 |
| O4—Pb1—O1—C1 | -55.5 (7) | C5—C6—N1—C9 | 0.0 |
| O3—Pb1—O1—Pb2 | 87.6 (3) | C1—C6—N1—C9 | 180.0 |
| N1—Pb1—O1—Pb2 | -170.4 (4) | C5—C6—N1—Pb1 | -176.3 (3) |
| O2—Pb1—O1—Pb2 | 5.5 (3) | C1—C6—N1—Pb1 | 3.7 (3) |
| O4—Pb1—O1—Pb2 | 128.5 (3) | O1—Pb1—N1—C6 | -4.6 (2) |
| O2—Pb2—O1—C1 | 178.1 (7) | O3—Pb1—N1—C6 | 73.3 (3) |
| O5—Pb2—O1—C1 | -98.1 (6) | O2—Pb1—N1—C6 | -10.3 (4) |
| N2—Pb2—O1—C1 | 178.1 (5) | O4—Pb1—N1—C6 | 119.1 (3) |
| O4—Pb2—O1—C1 | 146.9 (8) | O1—Pb1—N1—C9 | 179.5 (3) |
| O2—Pb2—O1—Pb1 | -6.1 (3) | O3—Pb1—N1—C9 | -102.5 (3) |
| O5—Pb2—O1—Pb1 | 77.7 (3) | O2—Pb1—N1—C9 | 173.9 (3) |
| N2—Pb2—O1—Pb1 | -6.1 (5) | O4—Pb1—N1—C9 | -56.8 (3) |
| O4—Pb2—O1—Pb1 | -37.3 (2) | C6—N1—C9—C8 | 0.0 |
| O5—Pb2—O2—C11 | 89.3 (6) | Pb1—N1—C9—C8 | 175.5 (3) |
| N2—Pb2—O2—C11 | -7.5 (5) | C6—N1—C9—C10 | 177.7 (6) |

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| O1—Pb2—O2—C11 | 172.5 (6) | Pb1—N1—C9—C10 | -6.8 (6) |
| O4—Pb2—O2—C11 | -164.3 (7) | N1—C9—C8—C7 | 0.0 |
| O5—Pb2—O2—Pb1 | -77.7 (3) | C10—C9—C8—C7 | -177.4 (7) |
| N2—Pb2—O2—Pb1 | -174.6 (3) | C9—C8—C7—C5 | 0.0 |
| O1—Pb2—O2—Pb1 | 5.4 (2) | C6—C5—C7—C8 | 0.0 |
| O4—Pb2—O2—Pb1 | 28.68 (16) | C4—C5—C7—C8 | 180.0 |
| O1—Pb1—O2—C11 | -172.1 (7) | Pb2—O2—C11—C12 | -173.4 (2) |
| O3—Pb1—O2—C11 | 102.6 (6) | Pb1—O2—C11—C12 | -8.6 (7) |
| N1—Pb1—O2—C11 | -166.5 (5) | Pb2—O2—C11—C16 | 8.0 (7) |
| O4—Pb1—O2—C11 | 84.2 (6) | Pb1—O2—C11—C16 | 172.8 (3) |
| O1—Pb1—O2—Pb2 | -6.0 (3) | O2—C11—C12—C13 | -178.6 (6) |
| O3—Pb1—O2—Pb2 | -91.3 (3) | C16—C11—C12—C13 | 0.0 |
| N1—Pb1—O2—Pb2 | -0.4 (5) | C11—C12—C13—C14 | 0.0 |
| O4—Pb1—O2—Pb2 | -109.7 (3) | C12—C13—C14—C15 | 0.0 |
| O1—Pb1—O3—C21 | 130.1 (7) | C13—C14—C15—C16 | 0.0 |
| N1—Pb1—O3—C21 | 63.0 (7) | C13—C14—C15—C17 | 180.0 |
| O2—Pb1—O3—C21 | -160.8 (7) | C14—C15—C16—N2 | 180.0 |
| O4—Pb1—O3—C21 | 0.5 (6) | C17—C15—C16—N2 | 0.0 |
| O1—Pb1—O4—C21 | -57.0 (7) | C14—C15—C16—C11 | 0.0 |
| O3—Pb1—O4—C21 | -0.5 (7) | C17—C15—C16—C11 | 180.0 |
| N1—Pb1—O4—C21 | -114.9 (7) | O2—C11—C16—N2 | -1.4 (6) |
| O2—Pb1—O4—C21 | 23.0 (8) | C12—C11—C16—N2 | 180.0 |
| O1—Pb1—O4—Pb2 | -37.8 (2) | O2—C11—C16—C15 | 178.6 (6) |
| O3—Pb1—O4—Pb2 | 18.6 (4) | C12—C11—C16—C15 | 0.0 |
| N1—Pb1—O4—Pb2 | -95.7 (2) | C15—C16—N2—C19 | 0.0 |
| O2—Pb1—O4—Pb2 | 42.2 (2) | C11—C16—N2—C19 | 180.0 |
| O2—Pb2—O4—C21 | 97.5 (8) | C15—C16—N2—Pb2 | 175.1 (3) |
| O5—Pb2—O4—C21 | 169.2 (8) | C11—C16—N2—Pb2 | -4.9 (3) |
| N2—Pb2—O4—C21 | 74.5 (8) | O2—Pb2—N2—C16 | 6.2 (2) |
| O1—Pb2—O4—C21 | -127.3 (8) | O5—Pb2—N2—C16 | -72.7 (3) |
| O2—Pb2—O4—Pb1 | -59.9 (3) | O1—Pb2—N2—C16 | 6.2 (4) |
| O5—Pb2—O4—Pb1 | 11.8 (3) | O4—Pb2—N2—C16 | 22.8 (2) |
| N2—Pb2—O4—Pb1 | -82.9 (3) | O2—Pb2—N2—C19 | -179.2 (3) |
| O1—Pb2—O4—Pb1 | 75.4 (4) | O5—Pb2—N2—C19 | 101.9 (3) |
| O2—Pb2—O5—C23 | -135.3 (7) | O1—Pb2—N2—C19 | -179.2 (3) |
| N2—Pb2—O5—C23 | -66.8 (7) | O4—Pb2—N2—C19 | -162.6 (2) |
| O1—Pb2—O5—C23 | 155.4 (7) | C16—N2—C19—C18 | 0.0 |
| O4—Pb2—O5—C23 | -176.4 (7) | Pb2—N2—C19—C18 | -174.2 (3) |
| Pb1—O1—C1—C2 | 174.9 (3) | C16—N2—C19—C20 | -178.9 (6) |
| Pb2—O1—C1—C2 | -9.7 (7) | Pb2—N2—C19—C20 | 6.9 (6) |
| Pb1—O1—C1—C6 | -5.9 (7) | N2—C19—C18—C17 | 0.0 |
| Pb2—O1—C1—C6 | 169.5 (3) | C20—C19—C18—C17 | 178.8 (7) |
| O1—C1—C2—C3 | 179.2 (6) | C19—C18—C17—C15 | 0.0 |
| C6—C1—C2—C3 | 0.0 | C14—C15—C17—C18 | 180.0 |
| C1—C2—C3—C4 | 0.0 | C16—C15—C17—C18 | 0.0 |
| C2—C3—C4—C5 | 0.0 | Pb1—O4—C21—O3 | 0.9 (11) |
| C3—C4—C5—C6 | 0.0 | Pb2—O4—C21—O3 | -10.0 (8) |
| C3—C4—C5—C7 | 180.0 | Pb1—O4—C21—C22 | 179.5 (10) |
| C4—C5—C6—N1 | 180.0 | Pb2—O4—C21—C22 | 168.6 (13) |

supplementary materials

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| C7—C5—C6—N1 | 0.0 | Pb1—O3—C21—O4 | -1.0 (13) |
| C4—C5—C6—C1 | 0.0 | Pb1—O3—C21—C22 | -179.7 (8) |
| C7—C5—C6—C1 | 180.0 | Pb2—O5—C23—O6 | -0.7 (13) |
| O1—C1—C6—N1 | 0.8 (5) | Pb2—O5—C23—C24 | 180.0 (7) |

Fig. 1

