

Bis(μ -*N*-nitroso-*N*-phenylhydroxylaminato)- κ^3 O, O' : O' ; κ^3 O':O, O' -bis[(*N*-nitroso-*N*-phenylhydroxylaminato)- κ^2 O, O']lead(II)]

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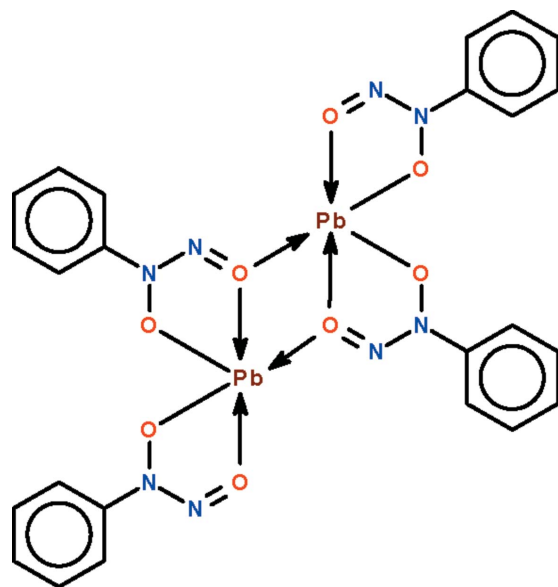
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.008$ Å; R factor = 0.026; wR factor = 0.071; data-to-parameter ratio = 16.1.

The four cupferronate ions in the dinuclear title compound, $[\text{Pb}_2(\text{C}_6\text{H}_5\text{N}_2\text{O}_2)_4]$, O,O' -chelate to the two Pb^{II} atoms; two of the four nitroso O atoms are also involved in bridging. The geometry of both five-coordinate Pb^{II} atoms is distorted Ψ -octahedral; if another two longer intermolecular $\text{Pb}\cdots\text{O}$ interactions [at 2.955 (1) and 3.099 (1) Å] are considered, the geometry is a distorted Ψ -square antiprism.

Related literature

For the spectroscopic assignment of the structure of the lead derivative, see: Bottei & Schneggenburger (1970). For the structure of the organic ligand, see: Hickmann *et al.* (1979).



Experimental

Crystal data

$[\text{Pb}_2(\text{C}_6\text{H}_5\text{N}_2\text{O}_2)_4]$
 $M_r = 962.86$
 Triclinic, $P\bar{1}$
 $a = 9.6149$ (5) Å
 $b = 11.5340$ (6) Å
 $c = 13.2724$ (7) Å
 $\alpha = 82.459$ (1)°
 $\beta = 79.280$ (1)°

$\gamma = 67.369$ (1)°
 $V = 1331.95$ (12) Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 12.69$ mm⁻¹
 $T = 100$ K
 $0.30 \times 0.15 \times 0.15$ mm

Data collection

Bruker SMART APEX
 diffractometer
 Absorption correction: multi-scan
 (*SADABS*; Sheldrick, 1996)
 $T_{\text{min}} = 0.115$, $T_{\text{max}} = 0.252$

16878 measured reflections
 6094 independent reflections
 5444 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.036$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.026$
 $wR(F^2) = 0.071$
 $S = 1.06$
 6094 reflections

379 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 1.74$ e Å⁻³
 $\Delta\rho_{\text{min}} = -2.24$ e Å⁻³

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); 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, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5480).

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supporting information

Acta Cryst. (2011). E67, m377 [doi:10.1107/S1600536811006775]

Bis(μ -*N*-nitroso-*N*-phenylhydroxylaminato)- κ^3 O, O' : O' ; κ^3 O':O, O' -bis[(*N*-nitroso-*N*-phenylhydroxylaminato)- κ^2 O, O']lead(II)]

Ezzatollah Najafi, Mostafa M. Amini and Seik Weng Ng

S1. Comment

The cupferronate ion is a common ion used for the complexation of metals; the crystal structure of the chelate has been reported (Hickmann *et al.*, 1979). The synthesis of the lead(II) derivative has been known for a long time (Bottei & Schneggenburger, 1970), and the compound was assumed to exist as a mononuclear compound. The compound is, in fact, a dinuclear compound (Scheme I). The four cupferronate ions in dinuclear $[\text{Pb}(\text{C}_6\text{H}_5\text{N}_2\text{O}_2)_2]_2$ O, O' -chelate to the lead(II) atom; two of the four nitroso O atoms are also involved in bridging (Fig. 1). The geometry of both five-coordinate lead atoms is Ψ -octahedral; if another longer intermolecular Pb \cdots O interactions (approx. 3.0 Å) are considered, the geometry is a Ψ -square-antiprism (Fig. 2).

S2. Experimental

Lead(II) nitrate (0.33 g, 1 mmol) dissolved in ethanol (20 ml) was added to the cupferron ligand (0.31 g, 2 mmol) dissolved in ethanol (20 ml). The mixture was stirred and then set aside for the growth of brown colored crystals.

S3. Refinement

Hydrogen atoms were placed in calculated positions (C–H 0.95 Å) and were included in the refinement in the riding model approximation, with $U(\text{H})$ set to $1.2U_{\text{eq}}(\text{C})$.

Omitted from the refinement were the following reflections owing to bad disagreement between the observed and calculated F^2 values: (0 0 1), (0 1 2), (1 0 1), (0 0 2), (11 4 7), (-9 - 11 5), (11 3 8), (11 5 6), (-4 - 9 10), (-9 -9 2) and (-4 7 0). The final difference Fourier map had a peak in the vicinity of Pb2 and a hole in the vicinity of the same atom.

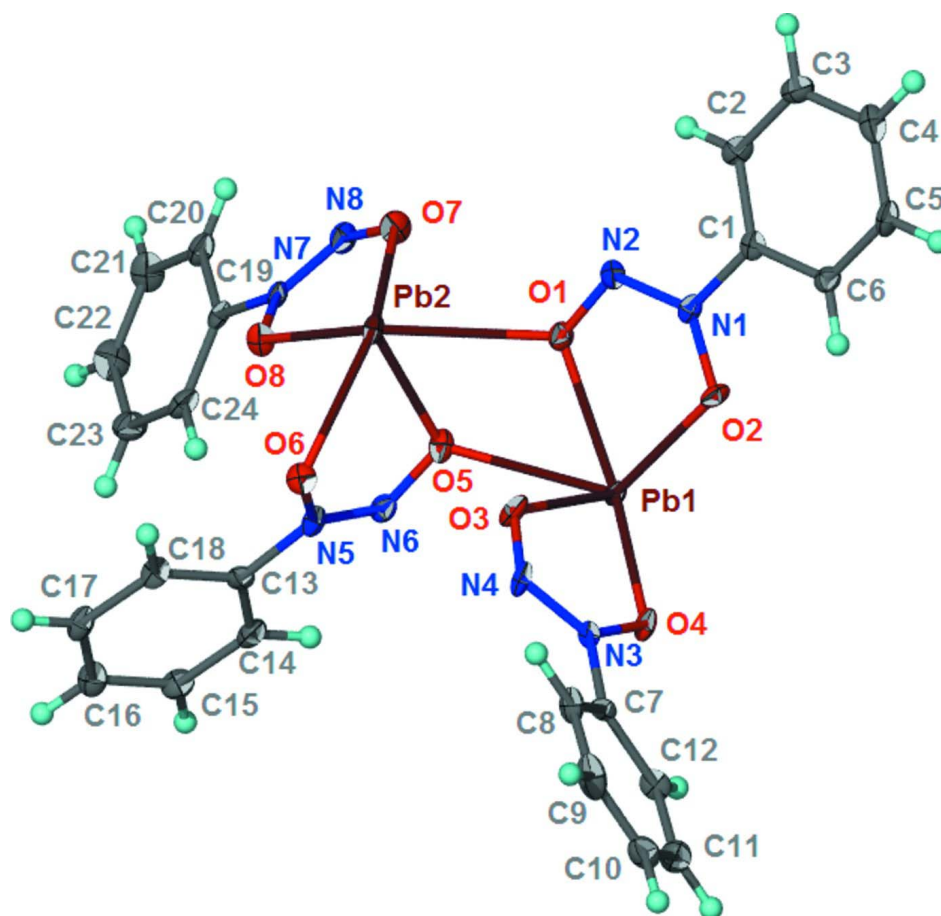


Figure 1

Anisotropic displacement ellipsoid plot (Barbour, 2001) of $\text{Pb}_2(\text{C}_6\text{H}_5\text{N}_2\text{O}_2)_4$ at the 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius.

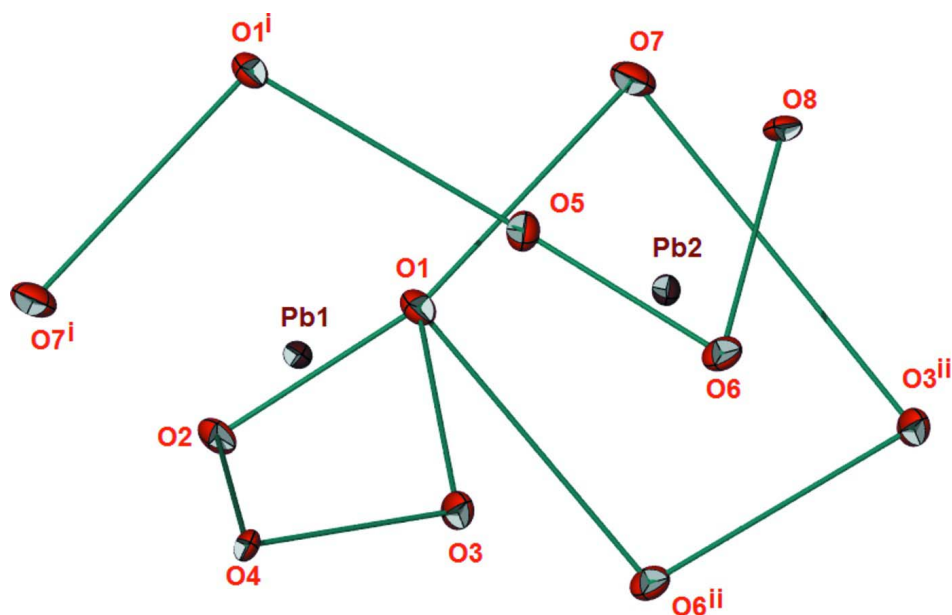


Figure 2

Detail of geometry of the lead atoms when intermolecular longer interactions are considered. Symmetry codes: (i) $1 - x, 1 - y, 1 - z$; (ii) $2 - x, 1 - y, 1 - z$.

Bis(μ -*N*-nitroso-*N*-phenylhydroxylaminate)- $\kappa^3 O, O': O'$; $\kappa^3 O': O, O'$ - bis[*N*-nitroso-*N*-phenylhydroxylaminate- $\kappa^2 O, O'$]lead(II)

Crystal data

[Pb₂(C₆H₅N₂O₂)₄]

$M_r = 962.86$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 9.6149$ (5) Å

$b = 11.5340$ (6) Å

$c = 13.2724$ (7) Å

$\alpha = 82.459$ (1)°

$\beta = 79.280$ (1)°

$\gamma = 67.369$ (1)°

$V = 1331.95$ (12) Å³

$Z = 2$

$F(000) = 896$

$D_x = 2.401$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 9907 reflections

$\theta = 2.4$ – 28.3 °

$\mu = 12.69$ mm⁻¹

$T = 100$ K

Prism, brown

$0.30 \times 0.15 \times 0.15$ mm

Data collection

Bruker SMART APEX
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ω scans

Absorption correction: multi-scan

(*SADABS*; Sheldrick, 1996)

$T_{\min} = 0.115$, $T_{\max} = 0.252$

16878 measured reflections

6094 independent reflections

5444 reflections with $I > 2\sigma(I)$

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

$\theta_{\max} = 27.5$ °, $\theta_{\min} = 2.3$ °

$h = -12 \rightarrow 12$

$k = -14 \rightarrow 14$

$l = -17 \rightarrow 17$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.026$

$wR(F^2) = 0.071$

$S = 1.06$

6094 reflections

379 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

$w = 1/[\sigma^2(F_o^2) + (0.0339P)^2 + 2.8506P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 1.74 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -2.24 \text{ e } \text{\AA}^{-3}$

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 | 0.634932 (18) | 0.462439 (15) | 0.364998 (12) | 0.00867 (6) |
| Pb2 | 0.819969 (18) | 0.523384 (15) | 0.612158 (13) | 0.01006 (6) |
| O1 | 0.6060 (4) | 0.6161 (3) | 0.4843 (2) | 0.0123 (7) |
| O2 | 0.5388 (4) | 0.6773 (3) | 0.3020 (2) | 0.0121 (7) |
| O3 | 0.8865 (4) | 0.4622 (3) | 0.3460 (3) | 0.0151 (7) |
| O4 | 0.7942 (4) | 0.3974 (3) | 0.2021 (3) | 0.0120 (7) |
| O5 | 0.7358 (4) | 0.3785 (3) | 0.5523 (3) | 0.0143 (7) |
| O6 | 1.0141 (4) | 0.3240 (3) | 0.5670 (3) | 0.0134 (7) |
| O7 | 0.5918 (4) | 0.5665 (3) | 0.7423 (3) | 0.0163 (7) |
| O8 | 0.8387 (4) | 0.3773 (3) | 0.7572 (3) | 0.0131 (7) |
| N1 | 0.5102 (4) | 0.7608 (4) | 0.3685 (3) | 0.0105 (8) |
| N2 | 0.5430 (5) | 0.7366 (4) | 0.4610 (3) | 0.0135 (8) |
| N3 | 0.9326 (4) | 0.3966 (4) | 0.1922 (3) | 0.0083 (7) |
| N4 | 0.9843 (5) | 0.4281 (4) | 0.2626 (3) | 0.0125 (8) |
| N5 | 0.9547 (5) | 0.2375 (4) | 0.5810 (3) | 0.0113 (8) |
| N6 | 0.8147 (4) | 0.2585 (4) | 0.5742 (3) | 0.0124 (8) |
| N7 | 0.7129 (4) | 0.3892 (4) | 0.8210 (3) | 0.0108 (8) |
| N8 | 0.5862 (5) | 0.4818 (4) | 0.8174 (3) | 0.0160 (9) |
| C1 | 0.4389 (5) | 0.8924 (4) | 0.3363 (4) | 0.0100 (9) |
| C2 | 0.3808 (5) | 0.9821 (5) | 0.4097 (4) | 0.0135 (9) |
| H2 | 0.3868 | 0.9575 | 0.4804 | 0.016* |
| C3 | 0.3142 (6) | 1.1081 (5) | 0.3774 (4) | 0.0155 (10) |
| H3 | 0.2736 | 1.1703 | 0.4264 | 0.019* |
| C4 | 0.3064 (6) | 1.1439 (5) | 0.2730 (4) | 0.0160 (10) |
| H4 | 0.2630 | 1.2304 | 0.2509 | 0.019* |
| C5 | 0.3626 (6) | 1.0524 (5) | 0.2020 (4) | 0.0154 (10) |
| H5 | 0.3563 | 1.0766 | 0.1313 | 0.018* |
| C6 | 0.4280 (5) | 0.9255 (4) | 0.2332 (4) | 0.0116 (9) |
| H6 | 0.4644 | 0.8630 | 0.1846 | 0.014* |
| C7 | 1.0322 (5) | 0.3611 (4) | 0.0962 (3) | 0.0093 (9) |
| C8 | 1.1523 (5) | 0.4028 (5) | 0.0675 (4) | 0.0142 (10) |
| H8 | 1.1735 | 0.4506 | 0.1116 | 0.017* |
| C9 | 1.2405 (6) | 0.3726 (5) | -0.0276 (4) | 0.0208 (11) |
| H9 | 1.3242 | 0.3991 | -0.0488 | 0.025* |

| | | | | |
|-----|------------|-------------|-------------|-------------|
| C10 | 1.2081 (6) | 0.3047 (5) | -0.0917 (4) | 0.0173 (10) |
| H10 | 1.2680 | 0.2866 | -0.1573 | 0.021* |
| C11 | 1.0883 (6) | 0.2625 (5) | -0.0609 (4) | 0.0179 (10) |
| H11 | 1.0678 | 0.2140 | -0.1047 | 0.021* |
| C12 | 0.9989 (5) | 0.2915 (4) | 0.0341 (4) | 0.0130 (9) |
| H12 | 0.9163 | 0.2638 | 0.0558 | 0.016* |
| C13 | 1.0454 (5) | 0.1123 (4) | 0.6183 (4) | 0.0114 (9) |
| C14 | 0.9955 (6) | 0.0140 (5) | 0.6234 (4) | 0.0156 (10) |
| H14 | 0.9035 | 0.0261 | 0.5994 | 0.019* |
| C15 | 1.0836 (6) | -0.1037 (5) | 0.6649 (4) | 0.0160 (10) |
| H15 | 1.0517 | -0.1728 | 0.6689 | 0.019* |
| C16 | 1.2180 (6) | -0.1200 (5) | 0.7002 (4) | 0.0164 (10) |
| H16 | 1.2769 | -0.2001 | 0.7287 | 0.020* |
| C17 | 1.2661 (6) | -0.0205 (5) | 0.6942 (4) | 0.0147 (10) |
| H17 | 1.3585 | -0.0326 | 0.7179 | 0.018* |
| C18 | 1.1794 (5) | 0.0983 (5) | 0.6533 (4) | 0.0123 (9) |
| H18 | 1.2110 | 0.1675 | 0.6495 | 0.015* |
| C19 | 0.7167 (5) | 0.2865 (4) | 0.8969 (4) | 0.0102 (9) |
| C20 | 0.6247 (6) | 0.3103 (5) | 0.9917 (4) | 0.0151 (10) |
| H20 | 0.5615 | 0.3939 | 1.0083 | 0.018* |
| C21 | 0.6273 (6) | 0.2085 (5) | 1.0620 (4) | 0.0191 (11) |
| H21 | 0.5645 | 0.2225 | 1.1270 | 0.023* |
| C22 | 0.7215 (6) | 0.0868 (5) | 1.0372 (4) | 0.0217 (11) |
| H22 | 0.7216 | 0.0175 | 1.0847 | 0.026* |
| C23 | 0.8148 (6) | 0.0666 (5) | 0.9434 (4) | 0.0180 (10) |
| H23 | 0.8806 | -0.0166 | 0.9275 | 0.022* |
| C24 | 0.8139 (6) | 0.1657 (5) | 0.8724 (4) | 0.0151 (10) |
| H24 | 0.8785 | 0.1515 | 0.8081 | 0.018* |

Atomic displacement parameters (Å²)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|--------------|--------------|--------------|--------------|--------------|
| Pb1 | 0.00879 (9) | 0.01181 (9) | 0.00641 (9) | -0.00508 (7) | -0.00091 (6) | -0.00028 (6) |
| Pb2 | 0.01043 (9) | 0.01290 (10) | 0.00908 (10) | -0.00683 (7) | -0.00345 (7) | 0.00231 (7) |
| O1 | 0.0151 (17) | 0.0133 (16) | 0.0089 (16) | -0.0059 (14) | -0.0030 (13) | 0.0017 (13) |
| O2 | 0.0169 (17) | 0.0132 (16) | 0.0069 (15) | -0.0060 (13) | -0.0007 (13) | -0.0032 (13) |
| O3 | 0.0140 (17) | 0.0257 (19) | 0.0091 (16) | -0.0100 (15) | -0.0017 (13) | -0.0051 (14) |
| O4 | 0.0080 (15) | 0.0203 (17) | 0.0103 (16) | -0.0077 (13) | -0.0027 (12) | -0.0005 (13) |
| O5 | 0.0142 (17) | 0.0167 (17) | 0.0143 (17) | -0.0077 (14) | -0.0058 (14) | 0.0022 (14) |
| O6 | 0.0122 (16) | 0.0150 (16) | 0.0155 (17) | -0.0084 (13) | -0.0013 (13) | 0.0009 (13) |
| O7 | 0.0172 (18) | 0.0186 (18) | 0.0096 (17) | -0.0051 (14) | 0.0011 (14) | 0.0016 (14) |
| O8 | 0.0081 (15) | 0.0190 (17) | 0.0104 (16) | -0.0053 (13) | 0.0017 (12) | 0.0015 (13) |
| N1 | 0.0095 (18) | 0.0153 (19) | 0.0080 (19) | -0.0064 (16) | -0.0009 (15) | -0.0004 (15) |
| N2 | 0.013 (2) | 0.014 (2) | 0.013 (2) | -0.0046 (16) | -0.0042 (16) | 0.0004 (16) |
| N3 | 0.0049 (17) | 0.0130 (18) | 0.0070 (18) | -0.0045 (15) | 0.0010 (14) | 0.0004 (15) |
| N4 | 0.0120 (19) | 0.022 (2) | 0.0062 (19) | -0.0099 (17) | -0.0033 (15) | 0.0020 (16) |
| N5 | 0.0138 (19) | 0.016 (2) | 0.0060 (18) | -0.0087 (16) | -0.0020 (15) | 0.0022 (15) |
| N6 | 0.0101 (19) | 0.015 (2) | 0.014 (2) | -0.0058 (16) | -0.0024 (15) | -0.0025 (16) |

| | | | | | | |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| N7 | 0.0113 (19) | 0.0161 (19) | 0.0047 (18) | -0.0047 (16) | -0.0021 (14) | 0.0000 (15) |
| N8 | 0.019 (2) | 0.019 (2) | 0.0083 (19) | -0.0047 (17) | -0.0031 (16) | 0.0006 (16) |
| C1 | 0.007 (2) | 0.013 (2) | 0.011 (2) | -0.0056 (17) | -0.0005 (17) | 0.0002 (17) |
| C2 | 0.012 (2) | 0.019 (2) | 0.010 (2) | -0.0079 (19) | 0.0031 (18) | -0.0032 (18) |
| C3 | 0.014 (2) | 0.017 (2) | 0.016 (2) | -0.0059 (19) | -0.0004 (19) | -0.006 (2) |
| C4 | 0.014 (2) | 0.015 (2) | 0.022 (3) | -0.0094 (19) | -0.008 (2) | 0.006 (2) |
| C5 | 0.017 (2) | 0.022 (3) | 0.009 (2) | -0.008 (2) | -0.0055 (18) | 0.0035 (19) |
| C6 | 0.011 (2) | 0.013 (2) | 0.012 (2) | -0.0057 (18) | -0.0021 (18) | -0.0023 (18) |
| C7 | 0.010 (2) | 0.008 (2) | 0.007 (2) | -0.0012 (17) | 0.0019 (17) | -0.0004 (16) |
| C8 | 0.010 (2) | 0.018 (2) | 0.016 (2) | -0.0072 (19) | -0.0045 (18) | 0.0034 (19) |
| C9 | 0.010 (2) | 0.027 (3) | 0.024 (3) | -0.008 (2) | -0.004 (2) | 0.008 (2) |
| C10 | 0.015 (2) | 0.018 (2) | 0.014 (2) | -0.003 (2) | 0.0000 (19) | 0.001 (2) |
| C11 | 0.013 (2) | 0.022 (3) | 0.017 (3) | -0.003 (2) | -0.0014 (19) | -0.004 (2) |
| C12 | 0.010 (2) | 0.016 (2) | 0.014 (2) | -0.0075 (18) | 0.0027 (18) | 0.0010 (18) |
| C13 | 0.011 (2) | 0.013 (2) | 0.008 (2) | -0.0029 (18) | 0.0016 (17) | -0.0024 (17) |
| C14 | 0.015 (2) | 0.022 (3) | 0.012 (2) | -0.008 (2) | -0.0017 (19) | -0.0048 (19) |
| C15 | 0.019 (2) | 0.017 (2) | 0.017 (2) | -0.011 (2) | -0.003 (2) | -0.0024 (19) |
| C16 | 0.014 (2) | 0.021 (3) | 0.015 (2) | -0.008 (2) | -0.0030 (19) | 0.001 (2) |
| C17 | 0.013 (2) | 0.024 (3) | 0.010 (2) | -0.010 (2) | -0.0018 (18) | 0.0005 (19) |
| C18 | 0.010 (2) | 0.019 (2) | 0.012 (2) | -0.0111 (19) | 0.0003 (18) | -0.0008 (19) |
| C19 | 0.011 (2) | 0.016 (2) | 0.008 (2) | -0.0091 (18) | -0.0026 (17) | -0.0006 (18) |
| C20 | 0.015 (2) | 0.022 (3) | 0.009 (2) | -0.008 (2) | -0.0026 (18) | -0.0011 (19) |
| C21 | 0.013 (2) | 0.029 (3) | 0.015 (3) | -0.010 (2) | 0.0030 (19) | 0.003 (2) |
| C22 | 0.028 (3) | 0.019 (3) | 0.021 (3) | -0.015 (2) | -0.006 (2) | 0.008 (2) |
| C23 | 0.024 (3) | 0.014 (2) | 0.017 (3) | -0.007 (2) | -0.003 (2) | -0.0024 (19) |
| C24 | 0.017 (2) | 0.021 (2) | 0.010 (2) | -0.010 (2) | -0.0013 (19) | -0.0040 (19) |

Geometric parameters (Å, °)

| | | | |
|--------|-----------|---------|-----------|
| Pb1—O2 | 2.382 (3) | C5—H5 | 0.9500 |
| Pb1—O3 | 2.384 (3) | C6—H6 | 0.9500 |
| Pb1—O4 | 2.427 (3) | C7—C12 | 1.378 (7) |
| Pb1—O1 | 2.433 (3) | C7—C8 | 1.388 (7) |
| Pb1—O5 | 2.757 (3) | C8—C9 | 1.387 (7) |
| Pb2—O8 | 2.371 (3) | C8—H8 | 0.9500 |
| Pb2—O5 | 2.389 (3) | C9—C10 | 1.376 (8) |
| Pb2—O6 | 2.403 (3) | C9—H9 | 0.9500 |
| Pb2—O7 | 2.453 (3) | C10—C11 | 1.392 (7) |
| Pb2—O1 | 2.718 (3) | C10—H10 | 0.9500 |
| O1—N2 | 1.305 (5) | C11—C12 | 1.387 (7) |
| O2—N1 | 1.309 (5) | C11—H11 | 0.9500 |
| O3—N4 | 1.307 (5) | C12—H12 | 0.9500 |
| O4—N3 | 1.310 (5) | C13—C14 | 1.380 (7) |
| O5—N6 | 1.320 (5) | C13—C18 | 1.394 (7) |
| O6—N5 | 1.307 (5) | C14—C15 | 1.399 (7) |
| O7—N8 | 1.311 (5) | C14—H14 | 0.9500 |
| O8—N7 | 1.312 (5) | C15—C16 | 1.393 (7) |
| N1—N2 | 1.293 (6) | C15—H15 | 0.9500 |

| | | | |
|------------|-------------|-------------|-----------|
| N1—C1 | 1.447 (6) | C16—C17 | 1.382 (7) |
| N3—N4 | 1.286 (6) | C16—H16 | 0.9500 |
| N3—C7 | 1.445 (6) | C17—C18 | 1.400 (7) |
| N5—N6 | 1.291 (6) | C17—H17 | 0.9500 |
| N5—C13 | 1.447 (6) | C18—H18 | 0.9500 |
| N7—N8 | 1.278 (6) | C19—C24 | 1.387 (7) |
| N7—C19 | 1.444 (6) | C19—C20 | 1.390 (7) |
| C1—C6 | 1.383 (7) | C20—C21 | 1.396 (7) |
| C1—C2 | 1.396 (7) | C20—H20 | 0.9500 |
| C2—C3 | 1.388 (7) | C21—C22 | 1.390 (8) |
| C2—H2 | 0.9500 | C21—H21 | 0.9500 |
| C3—C4 | 1.400 (7) | C22—C23 | 1.382 (8) |
| C3—H3 | 0.9500 | C22—H22 | 0.9500 |
| C4—C5 | 1.388 (7) | C23—C24 | 1.381 (7) |
| C4—H4 | 0.9500 | C23—H23 | 0.9500 |
| C5—C6 | 1.392 (7) | C24—H24 | 0.9500 |
| O2—Pb1—O3 | 91.40 (12) | C4—C5—C6 | 120.8 (4) |
| O2—Pb1—O4 | 92.26 (11) | C4—C5—H5 | 119.6 |
| O3—Pb1—O4 | 64.06 (11) | C6—C5—H5 | 119.6 |
| O2—Pb1—O1 | 64.21 (11) | C1—C6—C5 | 118.7 (4) |
| O3—Pb1—O1 | 77.66 (11) | C1—C6—H6 | 120.6 |
| O4—Pb1—O1 | 134.66 (11) | C5—C6—H6 | 120.6 |
| O2—Pb1—O5 | 125.02 (11) | C12—C7—C8 | 122.4 (4) |
| O3—Pb1—O5 | 72.98 (11) | C12—C7—N3 | 118.0 (4) |
| O4—Pb1—O5 | 123.21 (10) | C8—C7—N3 | 119.5 (4) |
| O1—Pb1—O5 | 61.03 (10) | C9—C8—C7 | 117.9 (5) |
| O8—Pb2—O5 | 79.95 (12) | C9—C8—H8 | 121.0 |
| O8—Pb2—O6 | 70.65 (11) | C7—C8—H8 | 121.0 |
| O5—Pb2—O6 | 64.08 (11) | C10—C9—C8 | 120.8 (5) |
| O8—Pb2—O7 | 63.85 (11) | C10—C9—H9 | 119.6 |
| O5—Pb2—O7 | 85.48 (12) | C8—C9—H9 | 119.6 |
| O6—Pb2—O7 | 128.79 (11) | C9—C10—C11 | 120.4 (5) |
| O8—Pb2—O1 | 130.44 (11) | C9—C10—H10 | 119.8 |
| O5—Pb2—O1 | 62.13 (11) | C11—C10—H10 | 119.8 |
| O6—Pb2—O1 | 113.71 (11) | C12—C11—C10 | 119.7 (5) |
| O7—Pb2—O1 | 81.54 (11) | C12—C11—H11 | 120.1 |
| N2—O1—Pb1 | 121.0 (3) | C10—C11—H11 | 120.1 |
| N2—O1—Pb2 | 121.7 (3) | C7—C12—C11 | 118.8 (4) |
| Pb1—O1—Pb2 | 111.54 (12) | C7—C12—H12 | 120.6 |
| N1—O2—Pb1 | 116.1 (3) | C11—C12—H12 | 120.6 |
| N4—O3—Pb1 | 122.2 (3) | C14—C13—C18 | 122.3 (5) |
| N3—O4—Pb1 | 115.8 (3) | C14—C13—N5 | 120.7 (4) |
| N6—O5—Pb2 | 115.1 (3) | C18—C13—N5 | 116.9 (4) |
| N6—O5—Pb1 | 121.7 (3) | C13—C14—C15 | 118.5 (5) |
| Pb2—O5—Pb1 | 111.59 (12) | C13—C14—H14 | 120.8 |
| N5—O6—Pb2 | 109.6 (3) | C15—C14—H14 | 120.8 |
| N8—O7—Pb2 | 119.6 (3) | C16—C15—C14 | 120.2 (5) |

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| N7—O8—Pb2 | 117.1 (3) | C16—C15—H15 | 119.9 |
| N2—N1—O2 | 125.6 (4) | C14—C15—H15 | 119.9 |
| N2—N1—C1 | 116.3 (4) | C17—C16—C15 | 120.4 (5) |
| O2—N1—C1 | 118.0 (4) | C17—C16—H16 | 119.8 |
| N1—N2—O1 | 112.5 (4) | C15—C16—H16 | 119.8 |
| N4—N3—O4 | 123.6 (4) | C16—C17—C18 | 120.2 (5) |
| N4—N3—C7 | 117.9 (4) | C16—C17—H17 | 119.9 |
| O4—N3—C7 | 118.5 (4) | C18—C17—H17 | 119.9 |
| N3—N4—O3 | 114.4 (4) | C13—C18—C17 | 118.3 (5) |
| N6—N5—O6 | 124.4 (4) | C13—C18—H18 | 120.9 |
| N6—N5—C13 | 116.9 (4) | C17—C18—H18 | 120.9 |
| O6—N5—C13 | 118.3 (4) | C24—C19—C20 | 121.8 (5) |
| N5—N6—O5 | 112.6 (4) | C24—C19—N7 | 118.3 (4) |
| N8—N7—O8 | 124.7 (4) | C20—C19—N7 | 119.9 (4) |
| N8—N7—C19 | 118.0 (4) | C19—C20—C21 | 118.5 (5) |
| O8—N7—C19 | 117.1 (4) | C19—C20—H20 | 120.8 |
| N7—N8—O7 | 113.5 (4) | C21—C20—H20 | 120.8 |
| C6—C1—C2 | 121.7 (4) | C22—C21—C20 | 120.2 (5) |
| C6—C1—N1 | 118.8 (4) | C22—C21—H21 | 119.9 |
| C2—C1—N1 | 119.5 (4) | C20—C21—H21 | 119.9 |
| C3—C2—C1 | 118.8 (5) | C23—C22—C21 | 119.9 (5) |
| C3—C2—H2 | 120.6 | C23—C22—H22 | 120.0 |
| C1—C2—H2 | 120.6 | C21—C22—H22 | 120.0 |
| C2—C3—C4 | 120.4 (5) | C24—C23—C22 | 121.0 (5) |
| C2—C3—H3 | 119.8 | C24—C23—H23 | 119.5 |
| C4—C3—H3 | 119.8 | C22—C23—H23 | 119.5 |
| C5—C4—C3 | 119.6 (5) | C23—C24—C19 | 118.6 (5) |
| C5—C4—H4 | 120.2 | C23—C24—H24 | 120.7 |
| C3—C4—H4 | 120.2 | C19—C24—H24 | 120.7 |
| | | | |
| O2—Pb1—O1—N2 | 5.5 (3) | Pb1—O4—N3—C7 | -178.8 (3) |
| O3—Pb1—O1—N2 | 103.2 (3) | O4—N3—N4—O3 | -0.4 (6) |
| O4—Pb1—O1—N2 | 71.0 (4) | C7—N3—N4—O3 | 178.2 (4) |
| O5—Pb1—O1—N2 | -179.6 (3) | Pb1—O3—N4—N3 | 0.8 (5) |
| O2—Pb1—O1—Pb2 | -148.12 (16) | Pb2—O6—N5—N6 | 28.7 (5) |
| O3—Pb1—O1—Pb2 | -50.45 (13) | Pb2—O6—N5—C13 | -143.8 (3) |
| O4—Pb1—O1—Pb2 | -82.70 (17) | O6—N5—N6—O5 | -0.8 (6) |
| O5—Pb1—O1—Pb2 | 26.76 (11) | C13—N5—N6—O5 | 171.8 (4) |
| O8—Pb2—O1—N2 | 130.8 (3) | Pb2—O5—N6—N5 | -29.0 (4) |
| O5—Pb2—O1—N2 | 175.6 (3) | Pb1—O5—N6—N5 | 111.0 (4) |
| O6—Pb2—O1—N2 | -145.0 (3) | Pb2—O8—N7—N8 | -8.9 (6) |
| O7—Pb2—O1—N2 | 86.3 (3) | Pb2—O8—N7—C19 | 167.6 (3) |
| O8—Pb2—O1—Pb1 | -75.71 (17) | O8—N7—N8—O7 | 0.3 (7) |
| O5—Pb2—O1—Pb1 | -30.95 (12) | C19—N7—N8—O7 | -176.1 (4) |
| O6—Pb2—O1—Pb1 | 8.49 (16) | Pb2—O7—N8—N7 | 8.3 (5) |
| O7—Pb2—O1—Pb1 | -120.29 (14) | N2—N1—C1—C6 | 167.7 (4) |
| O3—Pb1—O2—N1 | -81.4 (3) | O2—N1—C1—C6 | -11.0 (6) |
| O4—Pb1—O2—N1 | -145.5 (3) | N2—N1—C1—C2 | -13.4 (6) |

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| O1—Pb1—O2—N1 | -5.8 (3) | O2—N1—C1—C2 | 168.0 (4) |
| O5—Pb1—O2—N1 | -11.3 (3) | C6—C1—C2—C3 | -1.7 (7) |
| O2—Pb1—O3—N4 | -92.5 (3) | N1—C1—C2—C3 | 179.3 (4) |
| O4—Pb1—O3—N4 | -0.7 (3) | C1—C2—C3—C4 | -0.4 (7) |
| O1—Pb1—O3—N4 | -155.7 (3) | C2—C3—C4—C5 | 1.6 (7) |
| O5—Pb1—O3—N4 | 141.1 (4) | C3—C4—C5—C6 | -0.7 (7) |
| O2—Pb1—O4—N3 | 90.9 (3) | C2—C1—C6—C5 | 2.6 (7) |
| O3—Pb1—O4—N3 | 0.4 (3) | N1—C1—C6—C5 | -178.5 (4) |
| O1—Pb1—O4—N3 | 35.8 (3) | C4—C5—C6—C1 | -1.3 (7) |
| O5—Pb1—O4—N3 | -44.6 (3) | N4—N3—C7—C12 | 160.2 (4) |
| O8—Pb2—O5—N6 | -42.0 (3) | O4—N3—C7—C12 | -21.1 (6) |
| O6—Pb2—O5—N6 | 31.3 (3) | N4—N3—C7—C8 | -22.9 (6) |
| O7—Pb2—O5—N6 | -106.2 (3) | O4—N3—C7—C8 | 155.8 (4) |
| O1—Pb2—O5—N6 | 171.0 (3) | C12—C7—C8—C9 | 0.2 (7) |
| O8—Pb2—O5—Pb1 | 174.02 (15) | N3—C7—C8—C9 | -176.6 (4) |
| O6—Pb2—O5—Pb1 | -112.72 (16) | C7—C8—C9—C10 | 0.8 (7) |
| O7—Pb2—O5—Pb1 | 109.80 (14) | C8—C9—C10—C11 | -1.6 (8) |
| O1—Pb2—O5—Pb1 | 26.99 (11) | C9—C10—C11—C12 | 1.4 (8) |
| O2—Pb1—O5—N6 | -166.4 (3) | C8—C7—C12—C11 | -0.3 (7) |
| O3—Pb1—O5—N6 | -87.0 (3) | N3—C7—C12—C11 | 176.5 (4) |
| O4—Pb1—O5—N6 | -45.4 (3) | C10—C11—C12—C7 | -0.5 (7) |
| O1—Pb1—O5—N6 | -172.1 (3) | N6—N5—C13—C14 | 14.3 (6) |
| O2—Pb1—O5—Pb2 | -25.19 (19) | O6—N5—C13—C14 | -172.6 (4) |
| O3—Pb1—O5—Pb2 | 54.25 (14) | N6—N5—C13—C18 | -162.4 (4) |
| O4—Pb1—O5—Pb2 | 95.90 (15) | O6—N5—C13—C18 | 10.7 (6) |
| O1—Pb1—O5—Pb2 | -30.82 (12) | C18—C13—C14—C15 | -0.3 (7) |
| O8—Pb2—O6—N5 | 59.4 (3) | N5—C13—C14—C15 | -176.8 (4) |
| O5—Pb2—O6—N5 | -28.6 (3) | C13—C14—C15—C16 | 0.2 (7) |
| O7—Pb2—O6—N5 | 31.2 (3) | C14—C15—C16—C17 | -0.4 (8) |
| O1—Pb2—O6—N5 | -67.2 (3) | C15—C16—C17—C18 | 0.6 (7) |
| O8—Pb2—O7—N8 | -9.1 (3) | C14—C13—C18—C17 | 0.5 (7) |
| O5—Pb2—O7—N8 | 71.9 (3) | N5—C13—C18—C17 | 177.1 (4) |
| O6—Pb2—O7—N8 | 20.7 (4) | C16—C17—C18—C13 | -0.7 (7) |
| O1—Pb2—O7—N8 | 134.4 (3) | N8—N7—C19—C24 | 146.6 (5) |
| O5—Pb2—O8—N7 | -81.3 (3) | O8—N7—C19—C24 | -30.1 (6) |
| O6—Pb2—O8—N7 | -147.2 (3) | N8—N7—C19—C20 | -33.5 (6) |
| O7—Pb2—O8—N7 | 8.6 (3) | O8—N7—C19—C20 | 149.8 (4) |
| O1—Pb2—O8—N7 | -42.1 (3) | C24—C19—C20—C21 | -2.3 (7) |
| Pb1—O2—N1—N2 | 6.9 (5) | N7—C19—C20—C21 | 177.8 (4) |
| Pb1—O2—N1—C1 | -174.5 (3) | C19—C20—C21—C22 | 0.6 (8) |
| O2—N1—N2—O1 | -1.9 (6) | C20—C21—C22—C23 | 1.2 (8) |
| C1—N1—N2—O1 | 179.6 (4) | C21—C22—C23—C24 | -1.4 (8) |
| Pb1—O1—N2—N1 | -4.3 (5) | C22—C23—C24—C19 | -0.2 (8) |
| Pb2—O1—N2—N1 | 146.7 (3) | C20—C19—C24—C23 | 2.1 (7) |
| Pb1—O4—N3—N4 | -0.2 (5) | N7—C19—C24—C23 | -178.1 (4) |
