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# Tetra-$\mu_2$-acetato-$\kappa^8O$:$O'$-bis{[1-(1-adamantyliminiomethyl)-2-naphtholato-$\kappa O$]-rhodium(II)}

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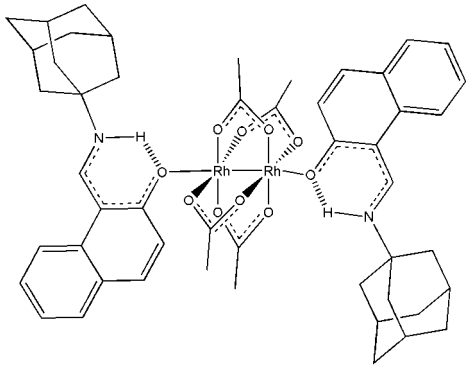
Key indicators: single-crystal X-ray study; $T$ = 90 K; mean $\sigma$(C–C) = 0.007 Å; $R$ factor = 0.039; $wR$ factor = 0.111; data-to-parameter ratio = 19.3.

The title compound, $[Rh_2(C_2H_3O_2)_4(C_{21}H_{23}NO)_2]$, is a centrosymmetric carboxylate-bridged dirhodium dimer, with each $Rh^{II}$ ion in a slightly distorted octahedral coordination environment. One axial bond involves the oxo O atom of the 1-(adamant-1-yliminiomethyl)-2-naphtholate ligand, with a longer than normal Rh—O bond distance which is likely to be due to the bond-lengthening effect of the Rh—Rh bond *trans* to this O atom. A strong intramolecular hydrogen bond exists between the H atom of the imino N atom and the oxo O atom of the ligand. In addition, the 1-(adamant-1-yliminiomethyl)-2-naphtholate ligand shows delocalization of $\pi$-electron density over the sequence of five atoms that extends from the N atom to the O atom.

## Related literature

For the structures of the ligand and a copper complex of the ligand, see: Acevedo-Arauz *et al.* (1992). For a related zinc complex, see: Zhao *et al.* (2003). For a review of rhodium(II) carboxylates, see: Boyar & Robinson (1983). For the structure of the parent aqua carboxylate compound, see: Cotton *et al.* (1971).




## Experimental

### Crystal data

$[Rh_2(C_2H_3O_2)_4(C_{21}H_{23}NO)_2]$
$M_r$ = 1052.80
Triclinic, $P\overline{1}$
$a$ = 9.5631 (13) Å
$b$ = 11.8424 (16) Å
$c$ = 11.8849 (16) Å
$\alpha$ = 107.160 (2)°
$\beta$ = 101.800 (2)°
$\gamma$ = 110.470 (2)°
$V$ = 1131.4 (3) Å$^3$
$Z$ = 1
Mo $K\alpha$ radiation
$\mu$ = 0.79 mm$^{-1}$
$T$ = 90 (2) K
0.48 × 0.45 × 0.32 mm

### Data collection

Bruker SMART APEXII diffractometer
Absorption correction: multi-scan (*TWINABS*; Bruker, 2007)
$T_{min}$ = 0.693, $T_{max}$ = 0.812
30719 measured reflections
5714 independent reflections
5378 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.054

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.111
$S$ = 1.15
5714 reflections
296 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 1.24 e Å$^{-3}$
$\Delta\rho_{min}$ = −1.36 e Å$^{-3}$

**Table 1**
Selected bond lengths (Å).

| | | | |
|---|---|---|---|
| Rh1—O5 | 2.250 (3) | C1—C10 | 1.445 (6) |
| Rh1—Rh1$^{i}$ | 2.3983 (7) | C2—C3 | 1.353 (6) |
| O5—C1 | 1.286 (5) | C9—C10 | 1.462 (5) |
| N1—C11 | 1.306 (5) | C10—C11 | 1.415 (5) |

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

**Table 2**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1···O5 | 0.83 (5) | 1.90 (5) | 2.555 (5) | 135 (5) |

Data collection: *APEX2* (Bruker, 2007); cell refinement: *cell_now* (Bruker, 2007) and *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1994); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2508).

## References

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  doi:10.1107/S1600536807046302 

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# supplementary materials

*Acta Cryst.* (2007). E**63**, m2606-m2607 [ **doi:10.1107/S1600536807046302** ]

# Tetra-$\mu_2$-acetato-$\kappa^8O:O'$-bis{[1-(1-adamantyliminiomethyl)-2-naphtholato-$\kappa O$]rhodium(II)}

**J. U. Franco, M. M. Olmstead and J. C. Hammons**

## Comment

Dirhodium carboxylate bridged species with a variety of axially ligated bases have been widely studied (Cotton *et al.*, 1971; Boyar & Robinson, 1983). Their diamagnetism and short Rh(II)···Rh(II) distance is explained by a Rh—Rh single bond. Weak axial ligation is attributed to the *trans*-influence of this metal-metal bond. Some of the recent interest in these species has to do with their ability to interact with nucleoside bases. Since a very large number of related compounds has been characterized by X-ray crystallography, a wealth of information about ligand basicities has been made available. The title compound has a Schiff base axial ligand which cannot coordinate in the usual bidendate manner due to the unavailability of *cis*-coordination sites. Instead, it coordinates through the O donor rather than the N donor atom. The imino nitrogen H atom forms a strong intramolecular H bond to the coordinated O (Fig. 1). This type of coordination has been previously reported in a Zn complex (Zhao, *et al.*, 2003). A bidentate mode of coordination of this ligand has been reported in the bis-Cu(II) complex (Acevedo-Arauz *et al.*, 1992).

The selected geometric parameters show that the Rh—Rh distance in the title compound, 2.3983 (7) Å, is in the normal range for complexes of this type. The axial ligand Rh—O distance is 2.250 (3) Å, considerably longer than the average Rh—*O*(acetato) distance of 2.043 (3) Å. This is commonly attributed to the *trans*- bond lengthening effect of the Rh—Rh bond. The bond distances within the axial ligand also show delocalization of the π electron density over the five atoms, N1, C11, C10, C1 and O5. Resonance structures that contribute to this delocalization give rise to zwitterions that place positive charge on N1 and negative charge on C10 or O5, thus enhancing the hydrogen bond and causing C9—C10 to be long and C2—C3 to be short. The entire 2-napthol-iminomethyl group is planar with an average deviation from the plane of 0.032 (5) Å. This pattern of protonation and bond distances persists in the free ligand as well (Acevedo-Arauz *et al.*, 1992).

## Experimental

The ligand, 1-(1-adamantyl)iminomethyl)-2-napthol, was prepared in the follwing manner. 1-Aminoadamantane (100 mg) and 2-hydroxy-1-naphthaldehyde (114 mg) were placed in a round bottom flask. Then 40 ml of ethanol was added to the mixture, and the solution was stirred for 3 hrs. The solution was then cooled to room temperature and the volume of solution was reduced by half. After the solution was placed in an ice bath, a yellow precipitate formed, which was filtered and collected. To prepare the title compound, in a 100 ml round bottom flask, 46 mg of 1-(1-adamantyl)iminomethyl)-2-napthol was dissolved in 30 ml of methanol, followed by addition of 16 mg of $[Rh(OAc)_2]_2{\cdot}2(H_2O)_2$ dissolved in 15 ml of methanol. The reaction mixture was stirred for 3 hrs at room temperature. The solution was then filtered to remove any solids and the solvent removed by use of a rotary evaporator. The product material was dissolved in diethyl ether and allowed to slowly evaporate. This method afforded green crystals, yield, 47%.

### Refinement

The C-bound H atoms were positioned geometrically with C—H = 0.95–1.00 Å, and allowed to ride on their parent atoms with $U_{iso}$(H) = 1.2 $U_{eq}$(C), 1.5$U_{eq}$(C) for methyl groups. The hydrogen atom bonded to N1 was freely refined. The structure is a rotational twin which was separated by use of cell_now and TWINABS (Bruker, 2007) into two domains related by a 180° rotation about the real [0 − 1 1] axis. The twin parameter refined to 0.3968 (13) using all observations involving domain 1 (HKLF 5).

### Figures

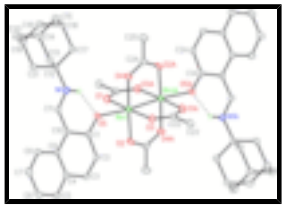


Fig. 1. A view of the title compound with the atom numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Hydrogen atoms have been omitted for clarity except for the imino H atom that is involved in hydrogen bonding, which is shown as a sphere of arbitrary size. Symmetry code: (A) −*x*,-*y*, 1 − *z*.

### Tetra-μ$_2$-acetato-κ$^8$*O*:*O*'-bis{[1-(1-adamantyliminomethyl)- 2-naphthol-κ*O*]rhodium(II)}

*Crystal data*

| | |
|---|---|
| $[Rh_2(C_2H_3O_2)_4(C_{21}H_{23}NO)_2]$ | $Z$ = 1 |
| $M_r$ = 1052.80 | $F_{000}$ = 542 |
| Triclinic, $P\overline{1}$ | $D_x$ = 1.545 Mg m$^{-3}$ |
| Hall symbol: -P 1 | Mo $K\alpha$ radiation<br>λ = 0.71073 Å |
| $a$ = 9.5631 (13) Å | Cell parameters from 9981 reflections |
| $b$ = 11.8424 (16) Å | θ = 3.1–31.7º |
| $c$ = 11.8849 (16) Å | μ = 0.79 mm$^{-1}$ |
| α = 107.160 (2)º | $T$ = 90 (2) K |
| β = 101.800 (2)º | Block, green |
| γ = 110.470 (2)º | 0.48 × 0.45 × 0.32 mm |
| $V$ = 1131.4 (3) Å$^3$ | |

*Data collection*

| | |
|---|---|
| Bruker SMART APEXII diffractometer | 5714 independent reflections |
| Radiation source: fine-focus sealed tube | 5378 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{int}$ = 0.054 |
| Detector resolution: 8.3 pixels mm$^{-1}$ | $\theta_{max}$ = 27.5º |
| $T$ = 90(2) K | $\theta_{min}$ = 3.0º |
| ω scans | $h$ = −12→11 |
| Absorption correction: multi-scan (TWINABS; Bruker, 2007) | $k$ = −15→14 |

$T_{min}$ = 0.693, $T_{max}$ = 0.812
30719 measured reflections

$l$ = −15→15

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.111
$S$ = 1.15
5714 reflections
296 parameters
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
$w = 1/[\sigma^2(F_o^2) + 5.1888P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.24 e Å$^{-3}$
$\Delta\rho_{min}$ = −1.36 e Å$^{-3}$
Extinction correction: none

*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 (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Rh1 | 0.03312 (4) | 0.07891 (3) | 0.45399 (3) | 0.01078 (9) |
| O1 | −0.1137 (4) | 0.1462 (3) | 0.5242 (3) | 0.0181 (6) |
| O2 | −0.1574 (4) | −0.0572 (3) | 0.2973 (3) | 0.0172 (6) |
| O3 | 0.1782 (4) | 0.0040 (3) | 0.3903 (3) | 0.0167 (6) |
| O4 | 0.2190 (4) | 0.2066 (3) | 0.6167 (3) | 0.0174 (6) |
| O5 | 0.1100 (4) | 0.2411 (3) | 0.3856 (3) | 0.0163 (6) |
| N1 | 0.2639 (4) | 0.4913 (3) | 0.4931 (3) | 0.0126 (6) |
| H1 | 0.229 (6) | 0.420 (5) | 0.500 (5) | 0.013 (12)* |
| C1 | 0.0905 (5) | 0.2486 (4) | 0.2782 (4) | 0.0138 (8) |
| C2 | −0.0022 (5) | 0.1327 (4) | 0.1631 (4) | 0.0137 (7) |
| H2 | −0.0487 | 0.0492 | 0.1668 | 0.016* |
| C3 | −0.0240 (5) | 0.1408 (4) | 0.0501 (4) | 0.0149 (8) |
| H3 | −0.0881 | 0.0627 | −0.0234 | 0.018* |
| C4 | 0.0458 (5) | 0.2625 (4) | 0.0370 (4) | 0.0137 (7) |
| C5 | 0.0267 (5) | 0.2650 (4) | −0.0832 (4) | 0.0191 (9) |

| | | | | |
|---|---|---|---|---|
| H5 | −0.0342 | 0.1852 | −0.1556 | 0.023* |
| C6 | 0.0950 (5) | 0.3812 (5) | −0.0971 (4) | 0.0224 (9) |
| H6 | 0.0811 | 0.3821 | −0.1782 | 0.027* |
| C7 | 0.1860 (5) | 0.4990 (4) | 0.0113 (4) | 0.0203 (8) |
| H7 | 0.2326 | 0.5800 | 0.0031 | 0.024* |
| C8 | 0.2074 (5) | 0.4973 (4) | 0.1279 (4) | 0.0167 (8) |
| H8 | 0.2715 | 0.5776 | 0.1993 | 0.020* |
| C9 | 0.1380 (5) | 0.3811 (4) | 0.1462 (4) | 0.0137 (7) |
| C10 | 0.1576 (5) | 0.3739 (4) | 0.2688 (4) | 0.0119 (7) |
| C11 | 0.2387 (5) | 0.4898 (4) | 0.3803 (4) | 0.0145 (7) |
| H11 | 0.2773 | 0.5718 | 0.3727 | 0.017* |
| C12 | 0.3360 (5) | 0.6120 (4) | 0.6110 (4) | 0.0129 (7) |
| C13 | 0.4989 (5) | 0.7094 (4) | 0.6205 (4) | 0.0147 (8) |
| H13A | 0.4867 | 0.7366 | 0.5492 | 0.018* |
| H13B | 0.5712 | 0.6666 | 0.6166 | 0.018* |
| C14 | 0.5699 (5) | 0.8310 (4) | 0.7452 (4) | 0.0163 (8) |
| H14 | 0.6757 | 0.8949 | 0.7517 | 0.020* |
| C15 | 0.5894 (5) | 0.7881 (4) | 0.8553 (4) | 0.0170 (8) |
| H15A | 0.6635 | 0.7470 | 0.8540 | 0.020* |
| H15B | 0.6347 | 0.8658 | 0.9356 | 0.020* |
| C16 | 0.4272 (5) | 0.6893 (4) | 0.8450 (4) | 0.0162 (8) |
| H16 | 0.4409 | 0.6612 | 0.9163 | 0.019* |
| C17 | 0.3573 (5) | 0.5682 (4) | 0.7201 (4) | 0.0154 (8) |
| H17A | 0.4296 | 0.5255 | 0.7166 | 0.018* |
| H17B | 0.2534 | 0.5037 | 0.7137 | 0.018* |
| C18 | 0.3131 (5) | 0.7544 (4) | 0.8501 (4) | 0.0183 (8) |
| H18A | 0.2085 | 0.6911 | 0.8437 | 0.022* |
| H18B | 0.3564 | 0.8313 | 0.9309 | 0.022* |
| C19 | 0.2936 (5) | 0.7986 (4) | 0.7411 (4) | 0.0160 (8) |
| H19 | 0.2208 | 0.8420 | 0.7445 | 0.019* |
| C20 | 0.4564 (5) | 0.8958 (4) | 0.7494 (4) | 0.0179 (8) |
| H20A | 0.4428 | 0.9235 | 0.6786 | 0.022* |
| H20B | 0.5021 | 0.9751 | 0.8285 | 0.022* |
| C21 | 0.2217 (5) | 0.6765 (4) | 0.6165 (4) | 0.0143 (8) |
| H21A | 0.1176 | 0.6130 | 0.6109 | 0.017* |
| H21B | 0.2047 | 0.7026 | 0.5449 | 0.017* |
| C22 | 0.1874 (5) | −0.0912 (4) | 0.4156 (4) | 0.0144 (7) |
| C23 | 0.2955 (5) | −0.1445 (4) | 0.3671 (4) | 0.0207 (9) |
| H23A | 0.2345 | −0.2388 | 0.3146 | 0.031* |
| H23B | 0.3819 | −0.1308 | 0.4380 | 0.031* |
| H23C | 0.3402 | −0.0985 | 0.3173 | 0.031* |
| C24 | 0.2413 (5) | 0.1683 (4) | 0.7049 (4) | 0.0153 (8) |
| C25 | 0.3815 (5) | 0.2619 (5) | 0.8237 (4) | 0.0240 (9) |
| H25A | 0.3459 | 0.2701 | 0.8961 | 0.036* |
| H25B | 0.4281 | 0.3484 | 0.8204 | 0.036* |
| H25C | 0.4613 | 0.2280 | 0.8315 | 0.036* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Rh1 | 0.01272 (15) | 0.00895 (15) | 0.00890 (14) | 0.00323 (11) | 0.00245 (11) | 0.00394 (10) |
| O1 | 0.0214 (15) | 0.0171 (15) | 0.0216 (15) | 0.0101 (12) | 0.0096 (12) | 0.0117 (12) |
| O2 | 0.0191 (14) | 0.0162 (14) | 0.0112 (13) | 0.0039 (12) | −0.0004 (11) | 0.0071 (11) |
| O3 | 0.0204 (15) | 0.0151 (14) | 0.0166 (14) | 0.0081 (12) | 0.0086 (12) | 0.0070 (12) |
| O4 | 0.0187 (14) | 0.0152 (14) | 0.0126 (13) | 0.0031 (12) | 0.0019 (12) | 0.0057 (11) |
| O5 | 0.0225 (15) | 0.0168 (15) | 0.0103 (13) | 0.0097 (12) | 0.0043 (12) | 0.0059 (11) |
| N1 | 0.0147 (15) | 0.0086 (16) | 0.0134 (15) | 0.0036 (13) | 0.0056 (13) | 0.0044 (13) |
| C1 | 0.0134 (18) | 0.0135 (19) | 0.0151 (18) | 0.0079 (15) | 0.0038 (15) | 0.0049 (15) |
| C2 | 0.0148 (18) | 0.0082 (17) | 0.0151 (18) | 0.0043 (14) | 0.0031 (15) | 0.0030 (14) |
| C3 | 0.0153 (18) | 0.0148 (19) | 0.0125 (17) | 0.0078 (15) | 0.0041 (15) | 0.0018 (15) |
| C4 | 0.0138 (18) | 0.0156 (19) | 0.0128 (18) | 0.0078 (15) | 0.0049 (15) | 0.0051 (15) |
| C5 | 0.020 (2) | 0.021 (2) | 0.0137 (18) | 0.0083 (17) | 0.0051 (16) | 0.0048 (16) |
| C6 | 0.024 (2) | 0.030 (2) | 0.0126 (19) | 0.0107 (19) | 0.0068 (17) | 0.0091 (17) |
| C7 | 0.024 (2) | 0.022 (2) | 0.020 (2) | 0.0091 (18) | 0.0098 (18) | 0.0139 (17) |
| C8 | 0.0190 (19) | 0.0156 (19) | 0.0131 (18) | 0.0051 (16) | 0.0050 (16) | 0.0060 (15) |
| C9 | 0.0124 (17) | 0.0164 (19) | 0.0130 (18) | 0.0079 (15) | 0.0047 (15) | 0.0046 (15) |
| C10 | 0.0134 (17) | 0.0135 (18) | 0.0107 (17) | 0.0059 (15) | 0.0071 (14) | 0.0055 (14) |
| C11 | 0.0142 (18) | 0.0111 (18) | 0.0166 (19) | 0.0043 (15) | 0.0044 (15) | 0.0056 (15) |
| C12 | 0.0138 (17) | 0.0129 (18) | 0.0099 (17) | 0.0053 (15) | 0.0028 (14) | 0.0035 (14) |
| C13 | 0.0120 (17) | 0.0156 (19) | 0.0128 (17) | 0.0035 (15) | 0.0042 (15) | 0.0042 (15) |
| C14 | 0.0139 (18) | 0.0149 (19) | 0.0139 (18) | 0.0022 (15) | 0.0045 (15) | 0.0029 (15) |
| C15 | 0.0120 (18) | 0.020 (2) | 0.0129 (18) | 0.0047 (16) | 0.0016 (15) | 0.0035 (16) |
| C16 | 0.0138 (18) | 0.020 (2) | 0.0116 (18) | 0.0062 (16) | 0.0022 (15) | 0.0055 (15) |
| C17 | 0.0169 (19) | 0.0150 (19) | 0.0145 (18) | 0.0059 (16) | 0.0052 (15) | 0.0078 (15) |
| C18 | 0.0162 (19) | 0.021 (2) | 0.0132 (18) | 0.0056 (17) | 0.0056 (16) | 0.0034 (16) |
| C19 | 0.0186 (19) | 0.0154 (19) | 0.0146 (18) | 0.0090 (16) | 0.0067 (16) | 0.0042 (15) |
| C20 | 0.021 (2) | 0.0132 (19) | 0.0147 (18) | 0.0060 (16) | 0.0060 (16) | 0.0018 (15) |
| C21 | 0.0136 (18) | 0.0149 (19) | 0.0125 (17) | 0.0066 (15) | 0.0023 (15) | 0.0041 (15) |
| C22 | 0.0156 (18) | 0.0130 (19) | 0.0133 (18) | 0.0056 (15) | 0.0034 (15) | 0.0056 (15) |
| C23 | 0.024 (2) | 0.023 (2) | 0.021 (2) | 0.0137 (18) | 0.0105 (18) | 0.0100 (17) |
| C24 | 0.0159 (18) | 0.0132 (19) | 0.0127 (18) | 0.0046 (15) | 0.0042 (15) | 0.0024 (15) |
| C25 | 0.019 (2) | 0.023 (2) | 0.017 (2) | −0.0001 (17) | −0.0010 (17) | 0.0059 (18) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| Rh1—O2 | 2.043 (3) | C12—C13 | 1.538 (5) |
| Rh1—O1 | 2.042 (3) | C13—C14 | 1.542 (5) |
| Rh1—O4 | 2.043 (3) | C13—H13A | 0.9900 |
| Rh1—O3 | 2.044 (3) | C13—H13B | 0.9900 |
| Rh1—O5 | 2.250 (3) | C14—C20 | 1.534 (6) |
| Rh1—Rh1$^{i}$ | 2.3983 (7) | C14—C15 | 1.537 (6) |
| O1—C22$^{i}$ | 1.263 (5) | C14—H14 | 1.0000 |
| O2—C24$^{i}$ | 1.268 (5) | C15—C16 | 1.535 (6) |
| O3—C22 | 1.275 (5) | C15—H15A | 0.9900 |

| | | | |
|---|---|---|---|
| O4—C24 | 1.268 (5) | C15—H15B | 0.9900 |
| O5—C1 | 1.286 (5) | C16—C17 | 1.541 (6) |
| N1—C11 | 1.306 (5) | C16—C18 | 1.542 (6) |
| N1—C12 | 1.489 (5) | C16—H16 | 1.0000 |
| N1—H1 | 0.83 (5) | C17—H17A | 0.9900 |
| C1—C2 | 1.444 (5) | C17—H17B | 0.9900 |
| C1—C10 | 1.445 (6) | C18—C19 | 1.535 (6) |
| C2—C3 | 1.353 (6) | C18—H18A | 0.9900 |
| C2—H2 | 0.9500 | C18—H18B | 0.9900 |
| C3—C4 | 1.431 (6) | C19—C20 | 1.541 (6) |
| C3—H3 | 0.9500 | C19—C21 | 1.543 (5) |
| C4—C5 | 1.413 (6) | C19—H19 | 1.0000 |
| C4—C9 | 1.423 (5) | C20—H20A | 0.9900 |
| C5—C6 | 1.377 (6) | C20—H20B | 0.9900 |
| C5—H5 | 0.9500 | C21—H21A | 0.9900 |
| C6—C7 | 1.413 (6) | C21—H21B | 0.9900 |
| C6—H6 | 0.9500 | C22—O1$^{i}$ | 1.263 (5) |
| C7—C8 | 1.366 (6) | C22—C23 | 1.512 (6) |
| C7—H7 | 0.9500 | C23—H23A | 0.9800 |
| C8—C9 | 1.406 (6) | C23—H23B | 0.9800 |
| C8—H8 | 0.9500 | C23—H23C | 0.9800 |
| C9—C10 | 1.462 (5) | C24—O2$^{i}$ | 1.268 (5) |
| C10—C11 | 1.415 (5) | C24—C25 | 1.510 (6) |
| C11—H11 | 0.9500 | C25—H25A | 0.9800 |
| C12—C17 | 1.536 (5) | C25—H25B | 0.9800 |
| C12—C21 | 1.538 (5) | C25—H25C | 0.9800 |
| O2—Rh1—O1 | 89.35 (12) | H13A—C13—H13B | 108.2 |
| O2—Rh1—O4 | 176.06 (12) | C20—C14—C15 | 109.1 (3) |
| O1—Rh1—O4 | 90.08 (12) | C20—C14—C13 | 109.0 (3) |
| O2—Rh1—O3 | 91.00 (12) | C15—C14—C13 | 109.5 (3) |
| O1—Rh1—O3 | 176.25 (12) | C20—C14—H14 | 109.7 |
| O4—Rh1—O3 | 89.31 (12) | C15—C14—H14 | 109.7 |
| O2—Rh1—O5 | 97.98 (11) | C13—C14—H14 | 109.7 |
| O1—Rh1—O5 | 91.01 (11) | C16—C15—C14 | 109.7 (3) |
| O4—Rh1—O5 | 85.92 (11) | C16—C15—H15A | 109.7 |
| O3—Rh1—O5 | 92.64 (11) | C14—C15—H15A | 109.7 |
| O2—Rh1—Rh1$^{i}$ | 87.16 (8) | C16—C15—H15B | 109.7 |
| O1—Rh1—Rh1$^{i}$ | 87.71 (9) | C14—C15—H15B | 109.7 |
| O4—Rh1—Rh1$^{i}$ | 88.93 (9) | H15A—C15—H15B | 108.2 |
| O3—Rh1—Rh1$^{i}$ | 88.58 (9) | C15—C16—C17 | 109.6 (3) |
| O5—Rh1—Rh1$^{i}$ | 174.69 (8) | C15—C16—C18 | 109.7 (4) |
| C22$^{i}$—O1—Rh1 | 119.3 (3) | C17—C16—C18 | 109.5 (3) |
| C24$^{i}$—O2—Rh1 | 119.9 (2) | C15—C16—H16 | 109.3 |
| C22—O3—Rh1 | 118.0 (3) | C17—C16—H16 | 109.3 |
| C24—O4—Rh1 | 118.0 (3) | C18—C16—H16 | 109.3 |
| C1—O5—Rh1 | 135.9 (3) | C12—C17—C16 | 109.1 (3) |
| C11—N1—C12 | 124.9 (3) | C12—C17—H17A | 109.9 |

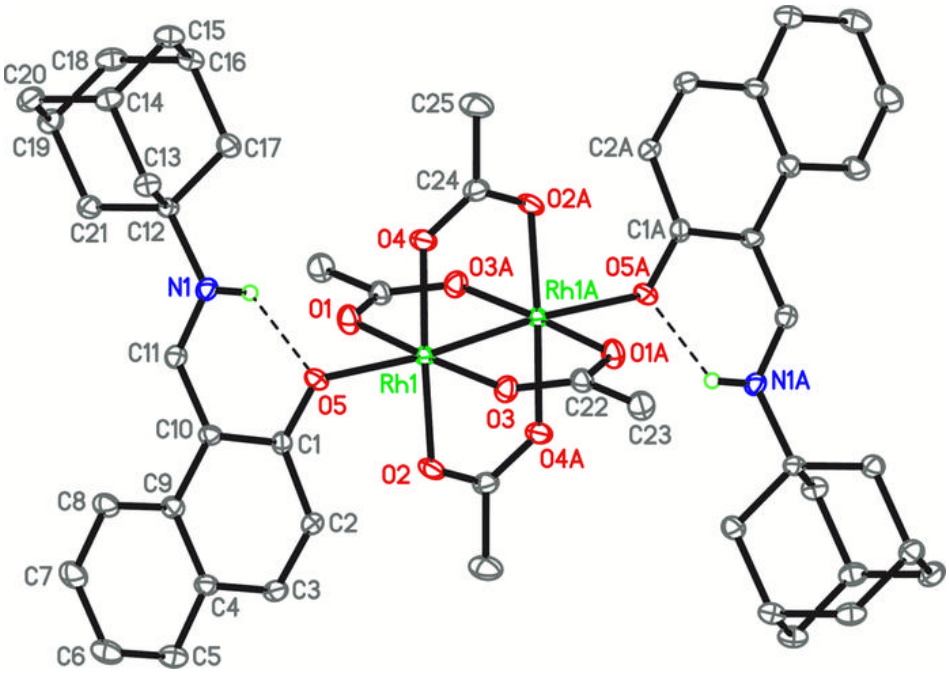
| | | | |
|---|---|---|---|
| C11—N1—H1 | 118 (3) | C16—C17—H17A | 109.9 |
| C12—N1—H1 | 117 (3) | C12—C17—H17B | 109.9 |
| O5—C1—C2 | 121.2 (4) | C16—C17—H17B | 109.9 |
| O5—C1—C10 | 121.0 (4) | H17A—C17—H17B | 108.3 |
| C2—C1—C10 | 117.8 (4) | C19—C18—C16 | 109.2 (3) |
| C3—C2—C1 | 121.2 (4) | C19—C18—H18A | 109.8 |
| C3—C2—H2 | 119.4 | C16—C18—H18A | 109.8 |
| C1—C2—H2 | 119.4 | C19—C18—H18B | 109.8 |
| C2—C3—C4 | 122.6 (4) | C16—C18—H18B | 109.8 |
| C2—C3—H3 | 118.7 | H18A—C18—H18B | 108.3 |
| C4—C3—H3 | 118.7 | C18—C19—C20 | 110.1 (3) |
| C5—C4—C9 | 120.0 (4) | C18—C19—C21 | 108.6 (3) |
| C5—C4—C3 | 120.5 (4) | C20—C19—C21 | 109.3 (3) |
| C9—C4—C3 | 119.5 (4) | C18—C19—H19 | 109.6 |
| C6—C5—C4 | 121.1 (4) | C20—C19—H19 | 109.6 |
| C6—C5—H5 | 119.5 | C21—C19—H19 | 109.6 |
| C4—C5—H5 | 119.5 | C14—C20—C19 | 110.2 (3) |
| C5—C6—C7 | 118.9 (4) | C14—C20—H20A | 109.6 |
| C5—C6—H6 | 120.6 | C19—C20—H20A | 109.6 |
| C7—C6—H6 | 120.6 | C14—C20—H20B | 109.6 |
| C8—C7—C6 | 120.5 (4) | C19—C20—H20B | 109.6 |
| C8—C7—H7 | 119.8 | H20A—C20—H20B | 108.1 |
| C6—C7—H7 | 119.8 | C12—C21—C19 | 109.2 (3) |
| C7—C8—C9 | 122.4 (4) | C12—C21—H21A | 109.8 |
| C7—C8—H8 | 118.8 | C19—C21—H21A | 109.8 |
| C9—C8—H8 | 118.8 | C12—C21—H21B | 109.8 |
| C8—C9—C4 | 117.1 (4) | C19—C21—H21B | 109.8 |
| C8—C9—C10 | 124.5 (4) | H21A—C21—H21B | 108.3 |
| C4—C9—C10 | 118.3 (4) | O1$^{i}$—C22—O3 | 126.4 (4) |
| C11—C10—C1 | 118.8 (4) | O1$^{i}$—C22—C23 | 116.8 (4) |
| C11—C10—C9 | 120.6 (4) | O3—C22—C23 | 116.8 (4) |
| C1—C10—C9 | 120.6 (4) | C22—C23—H23A | 109.5 |
| N1—C11—C10 | 124.2 (4) | C22—C23—H23B | 109.5 |
| N1—C11—H11 | 117.9 | H23A—C23—H23B | 109.5 |
| C10—C11—H11 | 117.9 | C22—C23—H23C | 109.5 |
| N1—C12—C17 | 106.9 (3) | H23A—C23—H23C | 109.5 |
| N1—C12—C21 | 109.2 (3) | H23B—C23—H23C | 109.5 |
| C17—C12—C21 | 109.3 (3) | O2$^{i}$—C24—O4 | 126.0 (4) |
| N1—C12—C13 | 111.5 (3) | O2$^{i}$—C24—C25 | 116.3 (4) |
| C17—C12—C13 | 109.2 (3) | O4—C24—C25 | 117.7 (4) |
| C21—C12—C13 | 110.7 (3) | C24—C25—H25A | 109.5 |
| C12—C13—C14 | 109.4 (3) | C24—C25—H25B | 109.5 |
| C12—C13—H13A | 109.8 | H25A—C25—H25B | 109.5 |
| C14—C13—H13A | 109.8 | C24—C25—H25C | 109.5 |
| C12—C13—H13B | 109.8 | H25A—C25—H25C | 109.5 |
| C14—C13—H13B | 109.8 | H25B—C25—H25C | 109.5 |

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

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1···O5 | 0.83 (5) | 1.90 (5) | 2.555 (5) | 135 (5) |

**Fig. 1**


C15
C18
C16
C20
C14
C19
C13
C17
C21
C12
N1
C11
C10
C1
C9
C8
C2
C7
C3
C4
C6
C5
C25
C24
O2A
O4
O3A
Rh1A
O5A
C2A
C1A
O1
O1A
Rh1
O5
O3
C22
C23
N1A
O2
O4A