

Yasuhiro Fuma and Masahiro Ebihara\*

Department of Chemistry, Faculty of Engineering, Gifu University, Yanagido, Gifu 501-1193, Japan

Correspondence e-mail: ebihara@apchem.gifu-u.ac.jp

Key indicators

Single-crystal X-ray study  
 T = 296 K  
 Mean  $\sigma(\text{C}-\text{C}) = 0.008 \text{ \AA}$   
 R factor = 0.035  
 wR factor = 0.073  
 Data-to-parameter ratio = 17.4

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

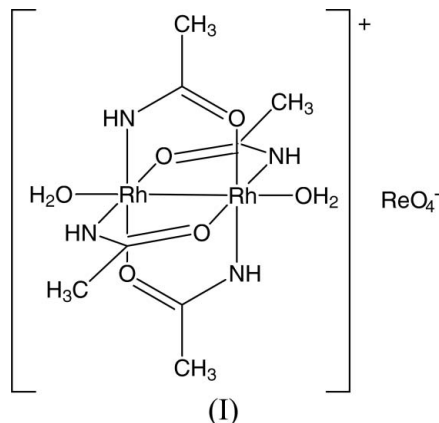
Tetra- $\mu$ -acetamido- $\kappa^4\text{N}:\text{O};\kappa^4\text{O}:\text{N}$ -diaqua-dirhodium(II,III) perrhenate

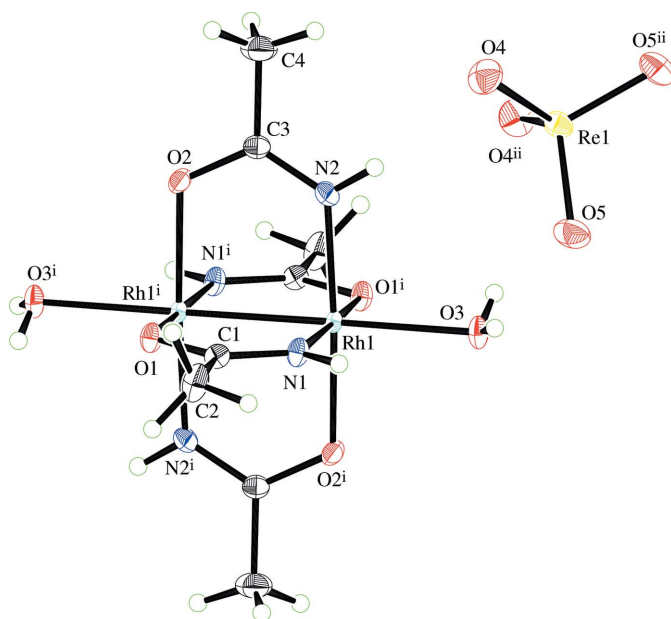
In the title compound,  $[\text{Rh}_2(\text{C}_2\text{H}_4\text{NO})_4(\text{H}_2\text{O})_2][\text{ReO}_4]$ , the cation lies on an inversion center and the anion on a twofold axis. The cations are connected two-dimensionally by  $\text{O}-\text{H}\cdots\text{O}$  and  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonding. Hydrogen bonds to perrhenate ions connect the two-dimensional sheets.

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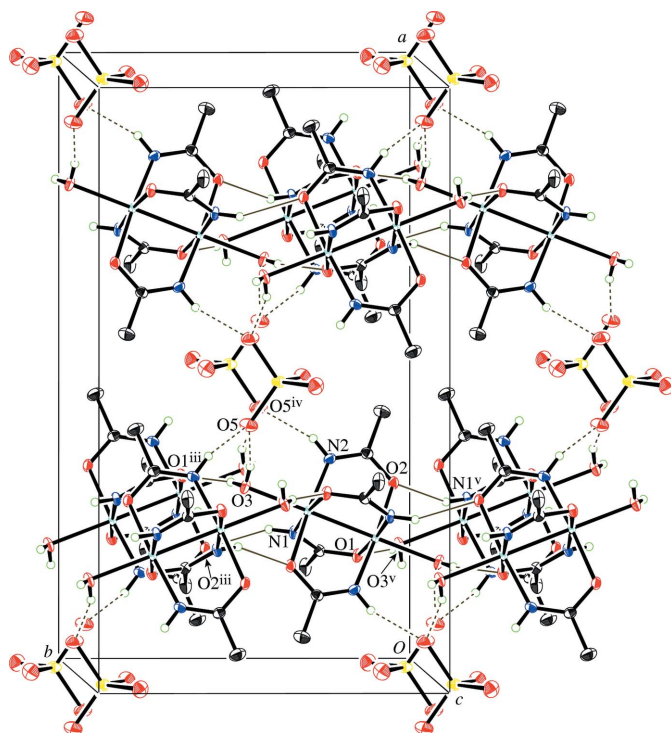
Comment

Paddlewheel complexes with amidate ligands are useful modules in making assembled structures, because they have hydrogen-donating NH and hydrogen-accepting O sites in addition to the axial coordination sites. We have studied assembled complexes of acetamidate-bridged paddlewheel dirhodium complexes with halide linkers: one-dimensional zigzag chain structures of  $[\text{Rh}_2(\text{acam})_4(\mu\text{-X})]_n\text{H}_2\text{O}$  (Hacam = acetamide; X = Cl, Br and I; n = 0, 2, 3 and 7) (Yang *et al.*, 2000, 2001), a two-dimensional honeycomb structure of  $[\{\text{Rh}_2(\text{acam})_4\}_3(\mu_3\text{-Cl})_2] \cdot 4\text{H}_2\text{O}$  (Takazaki *et al.*, 2003), and a three-dimensional diamondoid structure of  $[\{\text{Rh}_2(\text{acam})_4\}_2(\mu_4\text{-I})] \cdot 6\text{H}_2\text{O}$  (Fuma *et al.*, 2004). In all these structures, direct hydrogen bonds between the NH and O atoms of the amidate ligands play an important role in constructing the structures. In the structure of  $[\text{Rh}_2(\text{acam})_4(\text{H}_2\text{O})_2][\text{Rh}_2(\text{acam})_4(\mu\text{-MCl}_4)] \cdot 2\text{H}_2\text{O}$  (M = Pd and Pt; Yang *et al.*, 2006), there are anionic chains of  $[\text{Rh}_2(\text{acam})_4(\mu\text{-MCl}_4)]^-$  and  $[\text{Rh}_2(\text{acam})_4(\text{H}_2\text{O})_2]^+$  that participate in hydrogen-bonding networks using the bridging amidate and axial aqua ligands. We recently reported variation of hydrogen-bonding networks in hexafluorophosphate salts of amidate-bridged dirhodium complexes with axial aqua ligands (Ebihara & Fuma, 2006). In this paper, we report a hydrogen-bonding network in  $[\text{Rh}_2(\text{acam})_4(\text{H}_2\text{O})_2][\text{ReO}_4]$ , (I).





**Figure 1**  
The cation and anion of the title complex, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level [symmetry codes: (i)  $\frac{1}{2} - x, \frac{1}{2} - y, 1 - z$ ; (ii)  $1 - x, y, \frac{3}{2} - z$ ].



**Figure 2**  
Crystal structure of (I). Methyl H atoms have been omitted for clarity. Hydrogen bonds between the rhodium complexes are drawn as thin lines and the other hydrogen bonds as dotted lines [symmetry codes: (iii)  $\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$ ; (iv)  $x, 1 - y, -\frac{1}{2} + z$ ; (v)  $\frac{1}{2} - x, -\frac{1}{2} + y, \frac{1}{2} - z$ ].

The structure of (I) is shown in Fig. 1. There are one independent  $\text{Rh}_2(\text{acam})_4$  unit that lies on an inversion center (1/4, 1/4, 1/2) and one independent perrhenate ion of which the Re atom lies on a twofold axis ( $\frac{1}{2}, y, \frac{3}{4}$ ). The bond distances around each Rh atom, including the metal–metal bond

(Table 1), are similar to the corresponding ones in  $[\text{Rh}_2(\text{acam})_4(\text{H}_2\text{O})_2]\text{ClO}_4$  (Baranovskii *et al.*, 1986),  $[\text{Rh}_2(\text{acam})_4(\text{H}_2\text{O})_2]\text{PF}_6$  and  $[\text{Rh}_2(\text{acam})_4(\text{H}_2\text{O})_2]\text{PF}_6 \cdot 2\text{H}_2\text{O}$  (Ebihara & Fuma, 2006). The dirhodium complexes and the perrhenate ions form a hydrogen-bonded network (Fig. 2 and Table 2). The cation donates an H atom of the axial aqua ligand (O3) to the amidate oxygen atom (O1<sup>iii</sup>) [symmetry code: (iii)  $\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$ ]. The complex also donates the amino H atom of N1 to the amidate O atom (O2<sup>iii</sup>). Atoms O1 and O2 accept these hydrogen bonds from O3<sup>v</sup> and N2<sup>v</sup> [symmetry code: (v)  $\frac{1}{2} - x, -\frac{1}{2} + y, \frac{1}{2} - z$ ]. The resulting two-dimensional sheet structure is extended parallel to the *bc* plane. This sheet structure is very similar to that in  $[\text{Rh}_2(\text{acam})_4(\text{H}_2\text{O})_2]\text{PF}_6 \cdot 2\text{H}_2\text{O}$  and different from those in  $[\text{Rh}_2(\text{acam})_4(\text{H}_2\text{O})_2]\text{ClO}_4$  and  $[\text{Rh}_2(\text{acam})_4(\text{H}_2\text{O})_2]\text{PF}_6$  (Ebihara & Fuma, 2006). Perrhenate ions are located between the sheets. Aqua ligand O3 hydrogen bonds to perrhenate O5 atom and amidate atom N2 donates an H atom to another perrhenate ion (O5<sup>iv</sup>) [symmetry code: (iv)  $x, 1 - y, -\frac{1}{2} + z$ ]. These hydrogen bonds of the perrhenate ions connect the hydrogen-bonded sheets.

## Experimental

$[\text{Rh}_2(\text{acam})_4(\text{H}_2\text{O})_2] \cdot 6\text{H}_2\text{O}$  was synthesized according to the published method (Doyle *et al.*, 1990).  $[\text{Rh}_2(\text{acam})_4(\text{H}_2\text{O})_2]\text{ClO}_4$  was prepared by the method of Baranovskii *et al.* (1986). An aqueous solution (10 ml) of  $[\text{Rh}_2(\text{acam})_4(\text{H}_2\text{O})_2]\text{ClO}_4$  (15 mg, 0.026 mmol),  $[\text{Rh}_2(\text{acam})_4(\text{H}_2\text{O})_2] \cdot 6\text{H}_2\text{O}$  (16 mg, 0.028 mmol) and  $\text{NH}_4\text{ReO}_4$  (20 mg, 0.075 mmol) was left for 7 d at 323 K to obtain  $[\{\text{Rh}_2(\text{acam})_4\}_2(\mu_4\text{-ReO}_4)]$  (Fuma & Ebihara, 2006) with a small number of crystals of (I).

### Crystal data

$[\text{Rh}_2(\text{C}_2\text{H}_4\text{NO})_4(\text{H}_2\text{O})_2][\text{ReO}_4]$   
 $M_r = 724.30$   
 Monoclinic,  $C2/c$   
 $a = 19.718$  (2) Å  
 $b = 11.578$  (1) Å  
 $c = 8.2521$  (9) Å  
 $\beta = 97.664$  (5)°  
 $V = 1867.1$  (3) Å<sup>3</sup>

$Z = 4$   
 $D_x = 2.577$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation  
 $\mu = 8.27$  mm<sup>-1</sup>  
 $T = 296$  (2) K  
 Plate, brown  
 $0.15 \times 0.07 \times 0.03$  mm

### Data collection

Rigaku/MSC Mercury CCD diffractometer  
 $\omega$  scans  
 Absorption correction: integration (NUMABS; Higashi, 1999)  
 $T_{\min} = 0.120$ ,  $T_{\max} = 0.738$

7434 measured reflections  
 2139 independent reflections  
 2037 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.042$   
 $\theta_{\max} = 27.5^\circ$

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.035$   
 $wR(F^2) = 0.073$   
 $S = 1.15$   
 2139 reflections  
 123 parameters  
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0302P)^2 + 7.291P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 1.42$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -1.53$  e Å<sup>-3</sup>  
 Extinction correction: SHELXL97  
 Extinction coefficient: 0.00091 (11)

**Table 1**

Selected bond lengths (Å).

Rh1–Rh1 <sup>i</sup>	2.4053 (7)	Rh1–N1	1.979 (4)
Rh1–O1 <sup>i</sup>	2.040 (3)	Rh1–N2	1.971 (4)
Rh1–O2 <sup>i</sup>	2.033 (3)	Re1–O4	1.713 (5)
Rh1–O3	2.237 (4)	Re1–O5	1.718 (4)

 Symmetry code: (i)  $-x + \frac{1}{2}, -y + \frac{1}{2}, -z + 1$ .

**Table 2**

Hydrogen-bond geometry (Å, °).

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
O3–H10···O1 <sup>iii</sup>	0.76 (8)	2.10 (8)	2.841 (6)	168 (8)
O3–H9···O5	0.73 (7)	2.06 (8)	2.717 (6)	150 (8)
N1–H1···O2 <sup>iii</sup>	0.86	2.33	3.139 (6)	158
N2–H2···O5 <sup>iv</sup>	0.86	2.37	3.122 (6)	147

 Symmetry codes: (iii)  $-x + \frac{1}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$ ; (iv)  $x, -y + 1, z - \frac{1}{2}$ .

Aqua H atoms were located in difference syntheses and their positional parameters are refined [O–H = 0.73 (7) and 0.76 (8) Å], with  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$ . The remaining H atoms were positioned geometrically, with N–H = 0.86 Å and C–H = 0.96 Å, and constrained to ride on their parent atoms with  $U_{\text{iso}}(\text{H}) = xU_{\text{eq}}(\text{C,N})$ , where  $x = 1.5$  for methyl H and  $x = 1.2$  for all other H atoms. The highest peak and deepest hole are located 0.85 and 0.81 Å, respectively, from atom Re1.

Data collection: *CrystalClear* (Molecular Structure Corporation & Rigaku, 2001); cell refinement: *CrystalClear*; data reduction: *TEXSAN* (Rigaku/MSK, 2004); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure:

*SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97* and *TEXSAN*.

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