Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

fac-Aqua(2,2′-bipyrazine)tricarbonylrhenium(I) hexafluorophosphate dihydrate

D. Paul Rillema,* Robert A. Kirgan, Bradley Smucker and Curtis Moore

Department of Chemistry, Wichita State University, 1845 N. Fairmount, Wichita, KS 67260, USA

Correspondence e-mail:
paul.rillema@wichita.edu

Received 3 February 2007
Accepted 10 April 2007

Key indicators

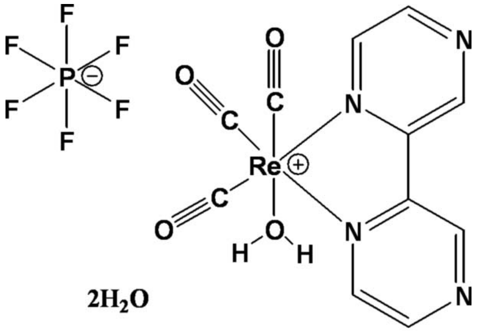
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
Disorder in solvent or counterion
R factor = 0.016
wR factor = 0.037
Data-to-parameter ratio = 11.6

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

The crystal structure of the title compound, $[Re(C_8H_6N_4)(CO)_3(H_2O)]PF_6\cdot 2H_2O$, shows the Re center to be in a distorted octahedral geometry with a facial distribution of the carbonyl ligands. Two other water molecules are hydrogen bonded to the coordinated water molecule. The cation lies on a crystallographic mirror plane, across which the anion is disordered.

Comment

During recrystallization of $[Re(CO)_3(bipyrazine)(pyridine)](PF_6)$, a light-induced reaction took place, producing the title compound, (I). Pyridine was replaced by a water molecule.



(I)

The cation of (I) (Fig. 1 and Table 1) shows a distorted octahedal geometry defined by two N and one O donors and three carbonyl ligands, which are arranged so that a *fac* isomer is formed. The Re atom lies on a crystallographic mirror plane, which also contains one carbonyl ligand and the coordinated water molecule. The anion is disordered across the mirror plane.

A comparison of bond lengths with those in two similiar complexes having 2,2′-bipyridine [(II)] and 1,10-phenanthroline [(III)] (Salignac *et al.*, 2003) in place of 2,2′-bipyrazine [(I)] reveals that the Re—*L* bond lengths, where *L* is the nitrogen ligand, are virtually identical [2.161 and 2.165 Å in (II), and 2.161 and 2.183 Å in (III)]. The Re—CO bond distances in the equatorial positions are 1.914 and 1.901 Å in (II), and 1.938 and 1.931 Å in (III), and in the axial positions 1.882 Å in (II) and 1.898 Å in (III).

The most interesting aspect of the title complex is the water molecule bound in the axial position. The Re—O bond distances (Table 2) show how the bipyridine and phenanthroline ligands donate electron density, producing a larger bond distance. When bipyrazine is present, there is a withdrawing effect on the Re center, causing the bond to be

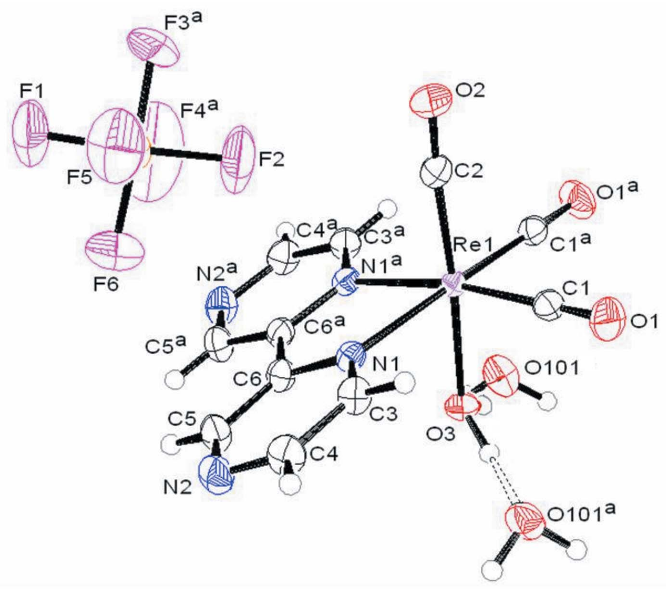


Figure 1
The structure of the formula unit of (I), with 50% probability displacement ellipsoids. The hydrogen bond is shown as a double dashed line. Only one disorder component is shown for the anion. [Symmetry code: (a) $x, \frac{1}{2} - y, z$.]

shorter. This effect is also seen in the hydrogen-bond distances between the coordinated and uncoordinated water molecules. The bipyrazine group gives a slightly shorter hydrogen bond than that in the bipyridine-containing complex. This shows the effect of the ring system not only on the inner-sphere environment but also on the outer-sphere environment.

Experimental

$Re(CO)_5Cl$ (100 mg, 0.277 mmol) and silver trifluoromethanesulfonate (71 mg, 0.277 mmol) were combined in ethanol (20 ml) and refluxed overnight in the dark. The AgCl was removed by filtration. 2,2′-Bipyrazine (44 mg, 0.278 mmol) was then added and the mixture was refluxed for 1 h. Next, pyridine (22 mg, 0.278 mmol) was added and the mixture was refluxed overnight. The volume was reduced and the solution added to an aqueous saturated NH_4PF_6 solution. The solid that formed was filtered off immediately, washed with diethyl ether and dried (yield *ca* 65%). Crystals were obtained from an ethanol–water mixture (1:1 *v*/*v*) by slow evaporation. During this recrystallization, water substituted for pyridine and the title compound was obtained.

Crystal data

$[Re(C_8H_6N_4)(CO)_3(H_2O)]$-
$PF_6{\cdot}2H_2O$
M_r = 627.42
Orthorhombic, *Pnma*
a = 15.5869 (6) Å
b = 13.1068 (5) Å
c = 9.0349 (4) Å
V = 1845.78 (13) $Å^3$
Z = 4
Mo $K\alpha$ radiation
μ = 6.77 mm^{-1}
T = 293 (2) K
0.42 × 0.24 × 0.20 mm

Data collection

Bruker Kappa-APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2006)
T_{min} = 0.158, T_{max} = 0.450
49927 measured reflections
1892 independent reflections
1880 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.016
$wR(F^2)$ = 0.037
S = 1.17
1892 reflections
163 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.68 e $Å^{-3}$
$\Delta\rho_{min}$ = −0.67 e $Å^{-3}$

Table 1
Selected geometric parameters (Å, °).

C1—Re1	1.918 (3)	O3—Re1	2.143 (3)
C2—Re1	1.912 (4)	N1—Re1	2.167 (2)
C2—Re1—C1	88.01 (12)	C1—Re1—N1	97.49 (11)
$C1^i$—Re1—C1	89.30 (17)	O3—Re1—N1	78.43 (8)
C2—Re1—O3	175.71 (15)	C1—Re1—$N1^i$	170.92 (10)
C1—Re1—O3	95.03 (10)	N1—Re1—$N1^i$	75.14 (12)
C2—Re1—N1	98.20 (12)		

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

Table 2
Re—O and O···O distances (Å).

Compound	Re—O	O···O
(I)	2.143 (3)	2.611 (3)
(II)	2.190	2.623
(III)	2.181	

Water H atoms were found in a difference map and their positions were fixed, while their U_{iso}(H) values were refined. Other H atoms were placed in idealized positions, with C—H = 0.93 Å, and refined as riding, with $U_{iso}(H) = 1.2U_{eq}(C)$. The PF_6^- anion was modeled as disordered equally over two orientations with a common F1—P1—F2 axis.

Data collection: *APEX2* (Bruker, 2006); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2006); program(s) used to solve structure: *SHELXTL* (Bruker, 2006); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Farrugia, 1997); software used to prepare material for publication: *SHELXTL*.

The authors acknowledge funding from the National Science Foundation for Research Sites for Educators of Chemistry, and the Department of Education for Graduate Assistance in Areas of National Need.

References

Bruker (2006). *APEX2*, *SAINT*, *SHELXTL* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Salignac, B., Grundler, P. V., Cayemittes, S., Frey, U., Scopelleti, R., Merbach, A. E., Hedinger, R., Hegetschweiler, K., Alberto, R., Prinz, U., Raabe, G., Kolle, U. & Hall, S. (2003). *Inorg. Chem.* **42**, 3516–3526.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.

Acta Cryst. (2007). E**63**, m1404–m1405 Rillema *et al.* • $[Re(C_8H_6N_4)(CO)_3(H_2O)]PF_6{\cdot}2H_2O$ **m1405**