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A molecular corner: *fac*-tricarbonylchlorido-bis[3-(4-pyridyl)pentane-2,4-dione-κ*N*]rhenium(I) chloroform solvate

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Key indicators

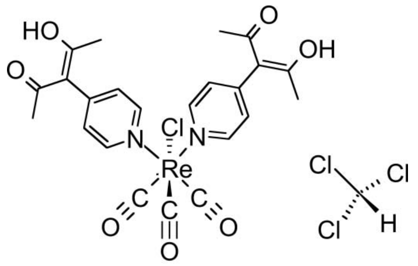
Single-crystal X-ray study
T = 110 K
Mean σ(C–C) = 0.014 Å
R factor = 0.058
wR factor = 0.151
Data-to-parameter ratio = 16.3

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

The title compound, $[ReCl(C_{10}H_{11}NO_2)_2(CO)_3]\cdot CHCl_3$, has Re—N distances of 2.202 (8) and 2.237 (7) Å, an N—Re—N angle of 84.1 (3)° and 2-hydroxy-4-oxopent-2-en-3-yl (acacH) units which are uncoordinated. The acacH units are in the enol tautomeric form, with delocalized single and double bonds.

Comment

The molecule 3-(4-pyridyl)pentane-2,4-dione, PyacH, was first prepared by Nozawa (1986), and has been used in a number of metal complexes (Turner *et al.*, 1997; Mackay *et al.*, 1995; Chen *et al.*, 2003, 2004; Vreshch *et al.*, 2003, 2004, 2005). Here, we describe the title compound (I), which is the first reported PyacH metal complex in which the pyridine unit is bound to the metal atom but the acacH unit is not. This makes (I) suitable as a 'molecular corner' for the construction of metal-organic frameworks or related supramolecular species.



(I)

This molecule (Fig. 1 and Table 1) is similar in structure to the molecules $[Re(CO)_3Cl(py)_2]$, (II), and $[Re(CO)_3Cl(4,4'\text{-}bpy)_2]$, (III) (Bélanger *et al.*, 1998). The N1—Re1—N2 angle in (I) is essentially identical to that in (II) [84.2 (2)°], but the corresponding angle in (III) is slightly wider at 87.0 (2)° (Bélanger *et al.*, 1998). The Re—N distances in (I) also agree well with those in (II) and (III).

Both 2-hydroxy-4-oxopent-2-en-3-yl (acacH) units are present in the enol tautomeric form. The similarity of the C—C and C—O distances about the approximately central axes of the acacH units, as well as the somewhat central locations of the refined OH hydrogen positions (Table 2), indicate delocalization of single and double bonds.

Experimental

Compound (I) was prepared by a procedure similar to that used by Zingales *et al.* (1967) for $Re(CO)_3Cl(py)_2$. PyacH was prepared by the method of Mackay *et al.* (1995). A solution of $Re(CO)_5Cl$ (0.0812 g, 2.24×10^{-4} mol) and PyacH (0.105 g, 5.91×10^{-4} mol) in $CHCl_3$ (100 ml) was placed in a flask that was wrapped with Al foil to protect it from light. The mixture was refluxed for 72 h under N_2, with stirring, during which time it turned from dark yellow to very pale yellow. The solution was concentrated to about 10 ml and added to pentane (80 ml). The resulting white flocculent precipitate was collected, washed with pentane and dried, yielding 0.135 g (91%) of (I) as an off-white powder. Analysis found: C 41.59 H, 3.51, N 4.15%; $C_{23}H_{22}ClN_2O_7Re$ requires: C 41.85, H 3.36, N 4.24%. FT–IR: ν_{CO} 2020, 1908, and 1877 cm^{-1}. Colorless plates of the chloroform solvate of (I) suitable for X-ray analysis were obtained after *ca* 15 days by layering a $CHCl_3$ solution of (I) with hexane.

Crystal data

$[ReCl(C_{10}H_{11}NO_2)_2(CO)_3]\cdot CHCl_3$
$M_r = 779.44$
Monoclinic, $P2_1/c$
$a = 10.889$ (4) Å
$b = 11.607$ (4) Å
$c = 24.306$ (10) Å
$\beta = 102.477$ (14)°
$V = 3000$ (2) Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 4.45$ mm^{-1}
$T = 110$ K
0.30 × 0.25 × 0.02 mm

Data collection

Nonius KappaCCD diffractometer with Oxford Cryostream
Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997)
$T_{min} = 0.308$, $T_{max} = 0.907$
27300 measured reflections
5749 independent reflections
4148 reflections with $I > 2\sigma(I)$
$R_{int} = 0.071$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.151$
$S = 1.37$
5749 reflections
353 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max} = 1.97$ e Å^{-3}
$\Delta\rho_{min} = -1.80$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Re1—Cl1	2.465 (2)	C12—O5	1.301 (10)
Re1—N1	2.237 (7)	C20—O6	1.309 (14)
Re1—N2	2.202 (8)	C22—O7	1.290 (13)
Re1—C1	1.954 (12)	C10—C11	1.412 (12)
Re1—C2	1.901 (10)	C11—C12	1.402 (11)
Re1—C3	1.915 (12)	C20—C21	1.426 (15)
C10—O4	1.302 (10)	C21—C22	1.369 (14)
Cl1—Re1—N2	86.08 (19)	N1—Re1—N2	84.1 (3)
Cl1—Re1—N1	84.81 (19)		
C3—Re1—N1—C8	−43.5 (7)	C5—C6—C11—C12	−76.2 (12)
C1—Re1—N2—C14	38.7 (7)	C15—C16—C21—C20	69.7 (14)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H5O···O4	1.13 (11)	1.46 (11)	2.459 (9)	144 (8)
O7—H7O···O6	1.06 (15)	1.55 (14)	2.486 (12)	144 (13)

H atoms on C were placed in idealized positions with C—H distances 0.95–1.00 Å and thereafter treated as riding. A torsional parameter was refined for each methyl group. Coordinates of H atoms on O were refined. Isotropic displacement parameters of the H atoms were assigned as 1.2 times U_{eq} of the bonded atoms (1.5 for methyl and OH). The maximum difference map peak was 0.86 Å from Re1 and the deepest hole 0.92 Å from Re1.

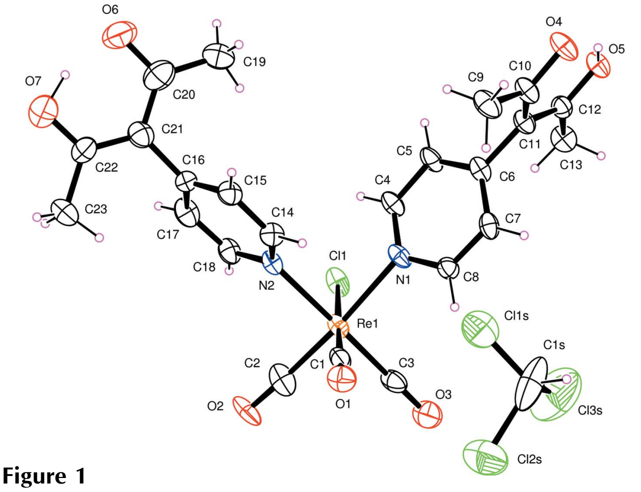


Figure 1
Molecular structure of (I), showing the numbering scheme and displacement ellipsoids at the 50% level. H atoms are represented with an arbitrary radius.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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