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Key indicators

Single-crystal X-ray study
 T = 100 K
 Mean $\sigma(\text{C}-\text{C}) = 0.011 \text{ \AA}$
 R factor = 0.033
 wR factor = 0.074
 Data-to-parameter ratio = 12.1

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

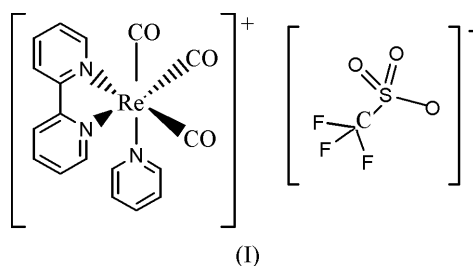
fac-Bipyridyltricarbonyl(pyridine)rhenium(I) trifluoromethanesulfonate

The structure of the title compound, $[\text{Re}(\text{C}_5\text{H}_5\text{N})(\text{C}_{10}\text{H}_8\text{N}_2)(\text{CO})_3](\text{CF}_3\text{O}_3\text{S})$, an emissive molecule, shows the Re centre in the cation to be in a distorted octahedral geometry with a facial disposition of carbonyl ligands.

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Comment

The title compound, (I), was prepared as a standard molecule for comparison with the properties of other heterocyclic tricarbonylrhenium(I) complexes containing pyridine as the sixth ligand. Complexes of this type have long-lived excited states, often of the order of microseconds, at room temperature and at 77 K. Such molecules have potential usage as dyes in solid-state regenerative solar cells and as sensors.



The cation (Fig. 1 and Table 1) shows a distorted octahedral geometry defined by three N donors and three carbonyl ligands that are arranged so that a *fac*-isomer is formed.

Experimental

The title compound was prepared according to the procedures described by Wrighton & Morse (1974). $\text{Re}(\text{CO})_5\text{Cl}$ (0.200 g,

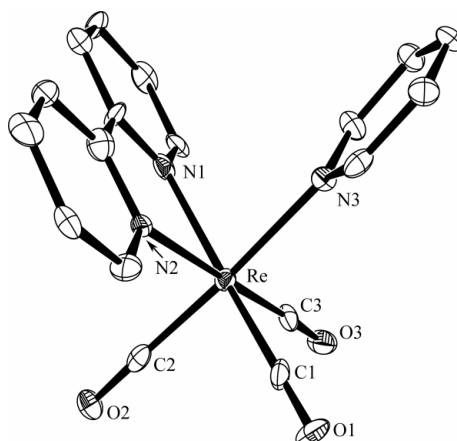


Figure 1
 View of the cation in (I) (50% probability displacement ellipsoids). H atoms have been omitted for clarity.

553 μmol) and 2,2'-bipyridine (bpy, 0.0864 g, 553 μmol) were dissolved in absolute ethanol (20 ml) and heated at reflux under Ar for 5 h. The solvent was removed by rotary evaporation, leaving solid $\text{Re}(\text{CO})_3(\text{bpy})\text{Cl}$ in the flask. Following the procedure of Shaver & Rillema (1992), the solid was then combined with AgCF_3SO_3 in a 1:1 ratio, added to absolute ethanol (20 ml) and refluxed for 8 h, resulting in precipitation of AgCl and formation of the trifluoromethanesulfonate (CF_3SO_3^-) salt. AgCl was removed by filtration and the solvent was removed by rotary evaporation. The solid was then combined with absolute ethanol (20 ml) and pyridine (0.040 ml), the solution was refluxed for 6 h and the solvent was removed by rotary evaporation. The remaining solid was dissolved in CH_2Cl_2 and then purified by column chromatography over neutral alumina. The initial yellow fraction that eluted with CH_2Cl_2 was discarded; the desired fraction eluted with a 3:1(v/v) $\text{CH}_2\text{Cl}_2/\text{CH}_3\text{CN}$ mixture. Removal of solvent by slow evaporation produced crystals of the title compound.

Crystal data

$[\text{Re}(\text{C}_5\text{H}_5\text{N})(\text{C}_{10}\text{H}_8\text{N}_2)(\text{CO})_3](\text{CF}_3\text{O}_3\text{S})$	$D_x = 2.121 \text{ Mg m}^{-3}$
$M_r = 654.58$	Mo $K\alpha$ radiation
Orthorhombic, $Pbca$	Cell parameters from 24 reflections
$a = 12.178 (2) \text{ \AA}$	$\theta = 10\text{--}12^\circ$
$b = 14.414 (3) \text{ \AA}$	$\mu = 6.10 \text{ mm}^{-1}$
$c = 23.357 (5) \text{ \AA}$	$T = 100 (2) \text{ K}$
$V = 4100.1 (14) \text{ \AA}^3$	Needle, green–yellow
$Z = 8$	$0.38 \times 0.15 \times 0.08 \text{ mm}$

Data collection

Enraf–Nonius CAD-4 diffractometer	2357 reflections with $I > 2\sigma(I)$
Non-profiled $\omega/2\theta$ scans	$\theta_{\text{max}} = 25.0^\circ$
Absorption correction: ψ scan (North <i>et al.</i> , 1968)	$h = 0 \rightarrow 14$
$T_{\text{min}} = 0.505$, $T_{\text{max}} = 0.612$	$k = 0 \rightarrow 17$
3601 measured reflections	$l = 0 \rightarrow 27$
3601 independent reflections	3 standard reflections
	frequency: 60 min
	intensity decay: none

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.033$	$w = 1/[\sigma^2(F_o^2) + (0.0313P)^2]$
$wR(F^2) = 0.074$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{\text{max}} = 0.001$
3601 reflections	$\Delta\rho_{\text{max}} = 1.01 \text{ e \AA}^{-3}$
298 parameters	$\Delta\rho_{\text{min}} = -0.74 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (\AA , $^\circ$).

Re–N1	2.174 (6)	Re–C1	1.929 (7)
Re–N2	2.158 (6)	Re–C2	1.918 (8)
Re–N3	2.194 (6)	Re–C3	1.912 (8)
N1–Re–N2	74.5 (2)	N2–Re–C3	176.6 (3)
N1–Re–N3	80.7 (2)	N3–Re–C1	93.3 (3)
N1–Re–C1	167.2 (3)	N3–Re–C2	176.1 (3)
N1–Re–C2	95.4 (3)	N3–Re–C3	94.5 (3)
N1–Re–C3	102.2 (3)	C1–Re–C2	90.7 (3)
N2–Re–N3	85.4 (2)	C1–Re–C3	89.5 (3)
N2–Re–C1	93.9 (3)	C2–Re–C3	85.6 (3)
N2–Re–C2	94.3 (2)		

H atoms were included in the riding-model approximation, with $\text{C–H} = 0.95 \text{ \AA}$ and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{iso}}(\text{C})$. The highest peak in the difference map was at (0.2234, 0.7605, 0.3385).

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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