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fac-Tricarbonyl(2,9-dimethyl-1,10-phenanthroline)(2,6-dimethylphenyl isocyanide)rhenium(I) hexafluorophosphate

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.005 Å
R factor = 0.021
wR factor = 0.051
Data-to-parameter ratio = 12.8

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

In the title compound, $[\mathrm{Re(C_9H_9N)(C_{14}H_{12}N_2)(CO)_3}]\mathrm{PF_6}$, the complex cation adopts a distorted octahedral geometry, with the Re atom at the center and a facial disposition of the three carbonyl ligands.

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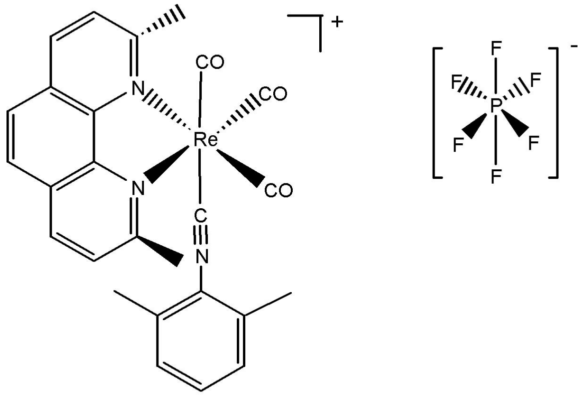
Comment

The title compound, (I), was synthesized as part of a series of diimine tricarbonylrhenium(I) complexes containing the ligand 2,6-dimethylphenyl isocyanide. These complexes are highly emissive. The emission lifetimes are in the microsecond timescale both at room temperature and at 77 K. These complexes have potential usage as dyes for solar energy conversion cells as well as sensors.



(I)

The cation (Fig. 1) shows distorted octahedral coordination, with the $\mathrm{Re^I}$ atom in the center and the three carbonyl ligands arranged so that the facial isomer is formed. The P—F distances in the anion are in the range 1.595 (3)–1.606 (3) Å.

Experimental

The title compound was prepared by modifying the procedure of Wrighton & Morse (1974). $[\mathrm{Re(CO)_5Cl}]$ (0.55 mmol) was added to an equimolar amount of 2,9-dimethyl-1,10-phenanthroline in a 125 ml round-bottomed flask. Absolute ethanol (50 ml) was added and the resulting mixture refluxed for 2–4 h. A light-yellow precipitate of $[\mathrm{ReCl(C_{14}H_{12}N_2)(CO)_3}]$ was formed. The solution was cooled to room temperature and filtered. The solid obtained was dried in a vacuum oven. Following the procedure of Shaver & Rillema (1992), the solid was combined with $\mathrm{AgCF_3SO_3}$ in a 1:1 ratio in ethanol. The mixture was refluxed for 4–6 h, resulting in the precipitation of AgCl and the formation of a $\mathrm{CF_3SO_3}^-$ salt. The solution was allowed to cool to room temperature and the AgCl was removed by filtration. The solution was added to an equimolar amount of 2,6-dimethylphenyl isocyanide and refluxed for another 3–5 h. The solvent was

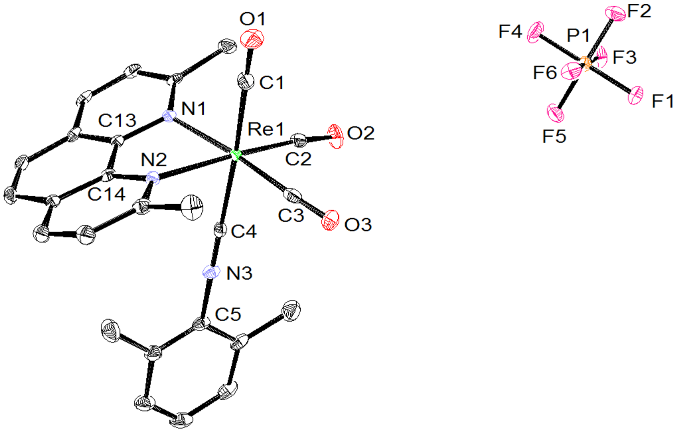


Figure 1
View of the title compound (50% probability displacement ellipsoids). H atoms have been omitted for clarity.

reduced in volume under vacuum. Saturated aqueous NH_4PF_6 (15 ml) was then added to the solution. The volume was diluted with distilled water to 50 ml (until precipitation was complete). The precipitate was filtered and dried in a vacuum oven. About 15 mg of the sample was dissolved in nitromethane (1 ml) in an uncovered small vial. The small vial was placed inside a larger vial containing diethyl ether. The vial was covered loosely and the solvent mixture was allowed to diffuse and slowly evaporate. Light-yellow crystals of the title compound were formed.

Crystal data

$[Re(C_9H_9N)(C_{14}H_{12}N_2)(CO)_3]PF_6$
$M_r = 754.63$
Monoclinic, $P2_1/n$
$a = 14.510$ (4) Å
$b = 11.223$ (4) Å
$c = 17.887$ (5) Å
$\beta = 113.53$ (2)°
$V = 2670.6$ (14) Å^3
$Z = 4$
$D_x = 1.877$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 24 reflections
$\theta = 1$–25°
$\mu = 4.69$ mm^{-1}
$T = 100$ K
Prism, yellow
0.5 × 0.3 × 0.3 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Non-profiled $\omega/2\theta$ scans
Absorption correction: refined from ΔF (Walker & Stuart, 1983)
$T_{min} = 0.198$, $T_{max} = 0.245$
4871 measured reflections
4682 independent reflections
3991 reflections with $I > 2\sigma(I)$
$R_{int} = 0.026$
$\theta_{max} = 25.0°$
$h = 0 \rightarrow 17$
$k = 0 \rightarrow 13$
$l = -21 \rightarrow 19$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.021$
$wR(F^2) = 0.051$
$S = 1.09$
4682 reflections
365 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0226P)^2 + 4.1906P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.57$ e Å^{-3}
$\Delta\rho_{min} = -0.60$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Re1—C2	1.920 (4)	Re1—N1	2.196 (3)
Re1—C1	1.994 (4)	Re1—N2	2.203 (3)
Re1—C4	2.063 (4)	N3—C4	1.152 (5)
C3—Re1—C2	84.56 (15)	C1—Re1—N1	92.21 (13)
C3—Re1—C1	90.93 (15)	C4—Re1—N1	87.48 (12)
C2—Re1—C1	95.39 (16)	C3—Re1—N2	101.03 (13)
C3—Re1—C4	89.12 (15)	C2—Re1—N2	169.88 (13)
C2—Re1—C4	89.59 (15)	C4—Re1—N2	82.12 (12)
C1—Re1—C4	175.01 (14)	N1—Re1—N2	75.63 (11)
C3—Re1—N1	175.53 (13)	C13—N1—Re1	111.7 (2)
C2—Re1—N1	98.32 (13)	C14—N2—Re1	110.8 (2)

H atoms were inserted at calculated positions (0.93–0.96 Å) and constrained with isotropic displacement parameters [$U_{iso}(H) = 1.2U_{eq}(C)$].

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: *SIR2002* (Burla *et al.*, 2003); 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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