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2,2′-Bipyridine(octacarbonyl)dirhenium(*Re*–*Re*)

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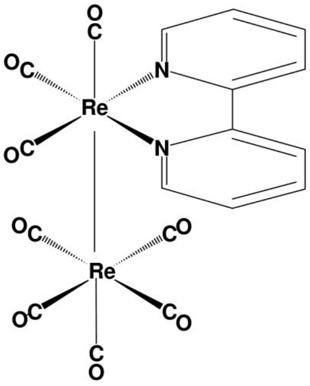
Key indicators

Single-crystal X-ray study
T = 220 K
Mean σ(C–C) = 0.019 Å
R factor = 0.046
wR factor = 0.136
Data-to-parameter ratio = 12.6

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

In the title compound, $[Re_2(C_{10}H_8N_2)(CO)_8]$, both Re atoms have distorted octahedral coordination geometry. The carbonyls of the $Re(CO)_5$ moiety are fully staggered relative to those of the $Re(CO)_3$ unit. We are currently interested in the application of Re-diimine complexes as catalysts for the photo- and electro-chemical reduction of CO_2. The (2,2′-bipyridine)$Re(CO)_3^{\cdot}$ radical, a key intermediate in the [Cl(2,2′-bipyridine)$Re(CO)_3$] catalytic CO_2 reduction cycle, is formed by Cl^- loss from [Cl(2,2′-bipyridine)$Re(CO)_3]^{-\cdot}$. We synthesized the title compound as a precursor to the (2,2′-bipyridine)$Re(CO)_3^{\cdot}$ radical and so that we can examine the structural features of the (2,2′-bipyridine)$Re(CO)_3$ moiety following irradiation of the title compound.



(I)

Experimental

The title compound (I) was prepared by refluxing $[Re_2(CO)_{10}]$ with excess bipyridyl in xylene for 3 d. The xylene was removed and the residual starting material extracted by dissolution in diethyl ether and precipitating the product with pentane. Crystals were obtained by slow concentration of an acetone solution in the dark.

Crystal data

$[Re_2(C_{10}H_8N_2)(CO)_8]$
$M_r = 752.66$
Triclinic, $P\bar{1}$
$a = 10.155$ (5) Å
$b = 13.586$ (5) Å
$c = 14.972$ (8) Å
$\alpha = 80.55$ (5)°
$\beta = 73.07$ (4)°
$\gamma = 85.40$ (5)°
$V = 1948.1$ (16) Å^3
$Z = 4$
$D_x = 2.566$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 43 reflections
$\theta = 12.5$–14.5°
$\mu = 12.47$ mm^{-1}
$T = 220$ (2) K
Plate, red
0.65 × 0.24 × 0.09 mm

Data collection

Stoe Stadi-4 four-circle diffractometer
ω/θ scans
Absorption correction: refined from ΔF (*DIFABS*; Walker & Stuart, 1983)
$T_{\min} = 0.049$, $T_{\max} = 0.310$
6852 measured reflections
6852 independent reflections
5903 reflections with $I > 2\sigma(I)$
$\theta_{\max} = 25.0^\circ$
$h = -11 \rightarrow 12$
$k = -15 \rightarrow 16$
$l = 0 \rightarrow 17$
3 standard reflections
frequency: 60 min
intensity variation: random $\pm 3.2\%$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.136$
$S = 1.11$
6852 reflections
542 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.082P)^2 + 20.60P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.03$
$\Delta\rho_{\max} = 2.74$ e Å^{-3}
$\Delta\rho_{\min} = -2.72$ e Å^{-3}

The crystal was transferred into the nitrogen stream of an Oxford Cryosystems open-flow cryostat (Cosier & Glazer, 1986) operating at 150 (2) K. The crystal used was unsuitable for absorption correction using ψ scans ($\mu \times t$mid $\simeq 3.0$) and it was not possible to index and measure crystal faces with sufficient accuracy for a numerical correction. Furthermore, because of the low symmetry we could not employ *HABITUS* (Herrendorf, 1995) to optimize the crystal shape and dimensions. We reluctantly, therefore, had to use *DIFABS* (Walker & Stuart, 1983) to apply empirical corrections. The final residual electron density extrema lie near Re2. H atoms were placed geometrically and refined riding at a distance of 0.95 Å from their parent C atoms with $U_{\rm iso}(\rm H) = 1.2U_{\rm eq}(\rm C)$.

Data collection: *Stadi*4 (Stoe & Cie, 1997); cell refinement: *Stadi*4; data reduction: *X-RED* (Stoe & Cie, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2001); software used to prepare material for publication: *SHELXL*97 and *PLATON*.

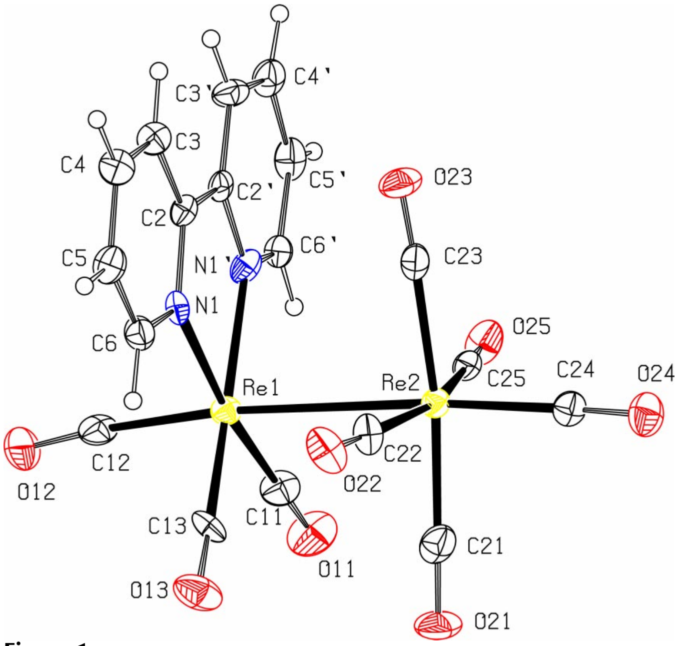


Figure 1
A general view of one of the two independent molecules of the title compound showing the atom numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

We thank EPSRC for the award of a diffractometer.

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