

**{4,4'-Bis[*p*-(diethylamino)- α -styryl]-2,2'-bipyridine}-
tricarbonylchlororhenium(I)**

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Key indicators

Single-crystal X-ray study
 $T = 90$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 R factor = 0.028
 wR factor = 0.065
Data-to-parameter ratio = 25.0For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

In the title compound $[\text{ReCl}(\text{C}_{34}\text{H}_{38}\text{N}_4)(\text{CO})_3]$, the Re atom has a distorted octahedral configuration. The bipyridine part of the DEAS-bpy ligand and two carbonyl groups occupy the equatorial plane of the complex, with the third carbonyl ligand and the Cl atom in the axial positions. The organic ligand is essentially planar, giving rise to an extended π -conjugated system.

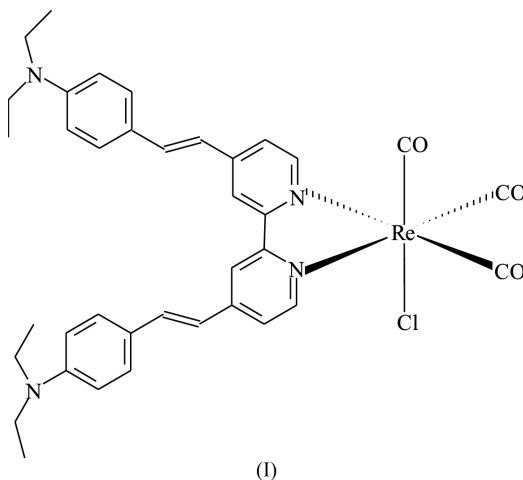
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Comment

Re^{I} -polypyridine-carbonyl complexes have interesting photophysical properties (Ziessel *et al.*, 1998); they can be used in photo- and electro-reduction reactions (Hawecker *et al.*, 1986; O'Toole *et al.*, 1985; Kutal *et al.*, 1987). It has also been suggested that they may form new materials possessing non-linear optical properties (Lehn, 1987). Therefore, knowledge of the molecular and crystal structure of such complexes is essential for understanding and explaining their properties. In the present communication, we report results of an X-ray structure investigation of the title complex, (I).



The Re atom in (I) has a distorted octahedral configuration, with the bipyridine moiety of the organic ligand and two carbonyl groups in the equatorial plane, and one carbonyl ligand and a Cl atom in the axial positions (Fig. 1). The bond angle $\text{N1}-\text{Re1}-\text{N2}$ of $74.55(8)^\circ$ is far from the ideal octahedral value. However, the $\text{C1}-\text{Re1}-\text{C3}$ angle is almost $89.4(1)^\circ$. The axial CO ligand is displaced out of the DEAS-bpy plane [$\text{C2}-\text{Re1}-\text{N1}$ is $99.01(9)^\circ$], while Cl1 is displaced towards the organic part of the molecule [$\text{N1}-\text{Re1}-\text{Cl1}$ is $82.94(6)^\circ$]. Moreover, the C3 atom is considerably displaced by 0.21 Å from a plane through the other equatorial atoms. Despite the significant distortion of the Re atom configuration

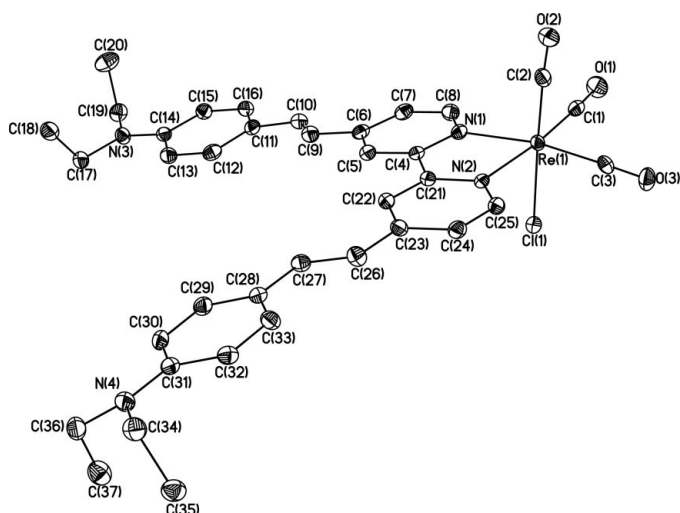


Figure 1
A view of (I) with displacement ellipsoids drawn at the 50% probability level. H atoms have been omitted for clarity.

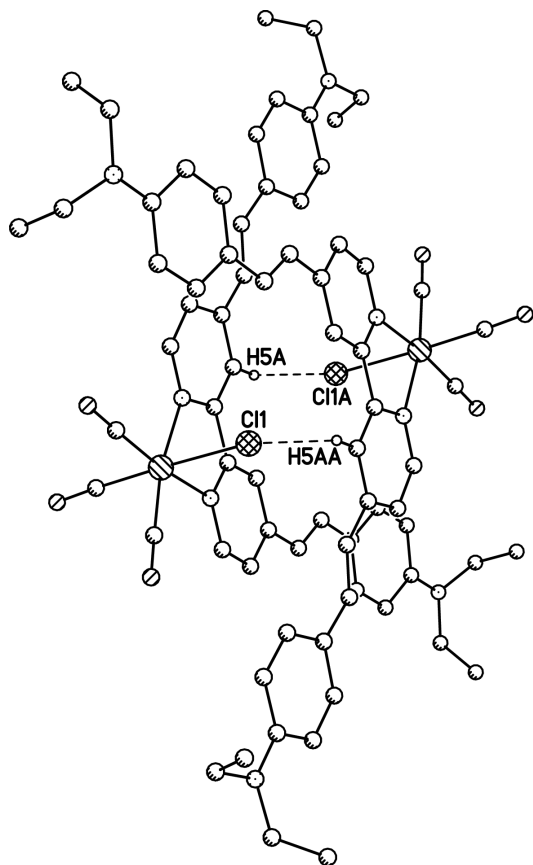


Figure 2
A view of the dimer as found in the crystal. H atoms, except those involved in contact with Cl1, have been omitted for clarity.

from octahedral, all Re—C and C—O distances are similar, though the axial Re—C bond is somewhat shorter than the equatorial bonds. The C—O bond lengths indicate significant π -back donation from the Re atom. This can be explained by strong electron-donating properties of the organic ligand.

DEAS-bpy contains two identical parts, each consisting of the diethylaminophenyl and pyridine groups connected by an ethylene moiety. The two parts are slightly twisted with respect to each other, the dihedral angle between them being 3° . Each half of DEAS-bpy is distorted from planarity. The relevant torsion angles C23—C26—C27—C28, C24—C23—C26—C27, C26—C27—C28—C29, C6—C9—C10—C11, C5—C6—C9—C10, C9—C10—C11—C16 are $177.5(2)$, $-173.6(3)$, $-171.7(3)$, $174.9(2)$, $-173.8(3)$, and $173.4(3)^\circ$, respectively. This non-planarity is sufficiently small to conserve the extended π -conjugated system of the organic ligand. Accordingly, the amine N3 and N4 atoms are almost coplanar with the C atoms to which they are bonded [the sums of the bond angles are $360.0(6)$ and $357.6(6)^\circ$, respectively]. Both methyl groups of each diethylamino substituent are displaced in opposite directions with respect to the mean DEAS-bpy plane.

In the crystal, molecules of (I) form dimers along the crystallographic c axis; the shortest contact in a dimer is $\text{Cl1} \cdots \text{H5A}(1-x, 1-y, -z)$ 2.65 \AA .

Experimental

Compound (I) was synthesized as described in the literature (Juris *et al.*, 1988). Crystals appropriate for X-ray structure analysis were grown by diffusion of ether into a solution of compound (I) in dichloromethane.

Crystal data

$[\text{ReCl}(\text{C}_{34}\text{H}_{38}\text{N}_4)(\text{CO})_3]$
 $M_r = 808.36$
 Monoclinic, $P2_1/c$
 $a = 14.4908(5) \text{ \AA}$
 $b = 23.1755(5) \text{ \AA}$
 $c = 9.6971(5) \text{ \AA}$
 $\beta = 91.899(2)^\circ$
 $V = 3254.8(2) \text{ \AA}^3$
 $Z = 4$

$D_x = 1.650 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 Cell parameters from 846 reflections
 $\theta = 2-30^\circ$
 $\mu = 3.86 \text{ mm}^{-1}$
 $T = 90.0(1) \text{ K}$
 Plate, red
 $0.3 \times 0.2 \times 0.1 \text{ mm}$

Data collection

CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: (SADABS; Bruker, 1998)
 $T_{\min} = 0.391$, $T_{\max} = 0.699$
 36 470 measured reflections
 10 508 independent reflections

8209 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.058$
 $\theta_{\max} = 32.5^\circ$
 $h = -21 \rightarrow 21$
 $k = -29 \rightarrow 34$
 $l = -14 \rightarrow 13$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.028$
 $wR(F^2) = 0.065$
 $S = 0.97$
 10508 reflections
 420 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0323P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.006$
 $\Delta\rho_{\max} = 1.75 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -1.72 \text{ e \AA}^{-3}$
 Extinction correction: SHELXL97
 Extinction coefficient: 0.00096 (8)

The positions of all H atoms were found from difference Fourier syntheses. Methyl groups were refined with torsional freedom, other H atoms with a riding model. $U(\text{H})$ was set at 1.2 (1.5 for methyl groups) times $U_{\text{eq}}(\text{C})$. The largest positive and negative features of the final difference synthesis lie within 0.9 \AA of the Re atom.

Data collection: SMART (Bruker, 1998); cell refinement: SMART; data reduction: SHELXTL (Sheldrick, 1997); program(s) used to solve structure: SHELXTL; program(s) used to refine structure:

SHELXTL; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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