

(2,2'-Bipyridine- κ^2N,N')tricarbonyl(acetonitrile- κN)rhenium(I) hexafluoroantimonate dichloromethane hemisolvate**Yandan Chen,* Liyi Zhang and Zhongning Chen**

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Key indicators

Single-crystal X-ray study

 $T = 293\text{ K}$ Mean $\sigma(\text{C}-\text{C}) = 0.023\text{ \AA}$ R factor = 0.061 wR factor = 0.161

Data-to-parameter ratio = 14.3

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

$[\text{Re}(\text{C}_2\text{H}_3\text{N})(\text{C}_{10}\text{H}_8\text{N}_2)_2(\text{CO})_3][\text{SbF}_6] \cdot 0.5\text{CH}_2\text{Cl}_2$ was isolated from the reaction between $[\text{Re}(\text{bpy})(\text{CO})_3\text{Cl}]$, where bpy = 2,2'-bipyridine, and AgSbF_6 in acetonitrile. The rhenium atom has a distorted octahedral geometry.

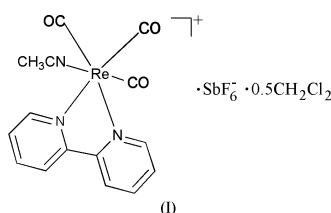
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Comment

Rhenium(I)-tricarbonyl-diimine systems are of increasing interest because of their rich photoluminescent properties and their possible effect in electrochemically promoted reductions of carbon dioxide (Yam *et al.*, 1997; Gibson *et al.*, 1998). Here we report the crystal structure of a rhenium(I) complex, $[\text{Re}(\text{CO})_3(\text{C}_{10}\text{H}_8\text{N}_2)_2(\text{CH}_3\text{CN})\text{SbF}_6] \cdot 0.5\text{CH}_2\text{Cl}_2$, (I), which was prepared according to literature methods (Fredericks *et al.*, 1979) except for using AgSbF_6 to remove the chloro ligand.



The asymmetric unit of (I) contains two similar units. A perspective drawing of one of the complex cations of (I) with the atomic numbering scheme, is depicted in Fig. 1. Selected bond distances are presented in Table 1.

The three carbonyl ligands at each Re atom are arranged in a facial configuration. Each Re atom adopts a slightly distorted octahedral geometry with $\text{C11}-\text{Re1}-\text{N11}$ and $\text{C101}-\text{Re2}-\text{N101}$ bond angles of $177.8(5)^\circ$ and $172.5(5)^\circ$, respectively. The $\text{N1}-\text{Re1}-\text{N2}$ [$75.5(4)^\circ$] and $\text{N101}-\text{Re2}-\text{N102}$ [$74.6(4)^\circ$] bond angles are far from 90° owing to the bite angle of the chelating bipyridyl (bpy) ligand (Yam *et al.*, 1995, 1996). The average bond distances for $\text{Re}-\text{N}$ [$2.163(11)\text{ \AA}$] and $\text{Re}-\text{C}$ [$1.920(16)\text{ \AA}$] are found to be comparable to those in previously determined rhenium(I)-tricarbonyl-diimine structures (Chen *et al.*, 1989; Yam *et al.*, 1997; Hevia *et al.*, 2002).

Experimental

An equimolar mixture of $[\text{Re}(\text{bpy})(\text{CO})_3\text{Cl}]$ and AgSbF_6 in acetonitrile was refluxed under anaerobic condition for 16 h, after which the resulting solution was filtered to give a yellow solution. The filtrate was evaporated under vacuum to afford a yellow-orange solid, which was redissolved in dichloromethane and layered with diethyl ether to form well shaped yellow crystals suitable for X-ray diffraction analysis.

Crystal data

[Re(C₂H₃N)(C₁₀H₈N₂)₂(CO)₃]
[SbF₆]_{0.5}CH₂Cl₂
M_r = 745.68
Triclinic, P1̄
a = 8.4548 (4) Å
b = 12.4176 (5) Å
c = 22.6477 (9) Å
α = 86.510 (1)°
β = 79.359 (1)°
γ = 73.167 (1)°
V = 2236.66 (16) Å³

Z = 4
D_x = 2.214 Mg m⁻³
Mo Kα radiation
Cell parameters from 6027 reflections
θ = 0.9–25.1°
μ = 6.81 mm⁻¹
T = 293 (2) K
Prism, yellow
0.62 × 0.38 × 0.28 mm

Data collection

Siemens SMART CCD diffractometer
ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
T_{min} = 0.057, T_{max} = 0.149
11706 measured reflections

7840 independent reflections
6151 reflections with I > 2σ(I)
R_{int} = 0.032
θ_{max} = 25.1°
h = -10 → 9
k = -14 → 14
l = -26 → 26

Refinement

Refinement on F²
R[F² > 2σ(F²)] = 0.061
wR(F²) = 0.161
S = 1.13
7840 reflections
550 parameters
H-atom parameters constrained

w = 1/[σ²(F_o²) + (0.0667P)² + 20.7333P]
where P = (F_o² + 2F_c²)/3
(Δ/σ)_{max} = 0.001
Δρ_{max} = 1.26 e Å⁻³
Δρ_{min} = -2.02 e Å⁻³

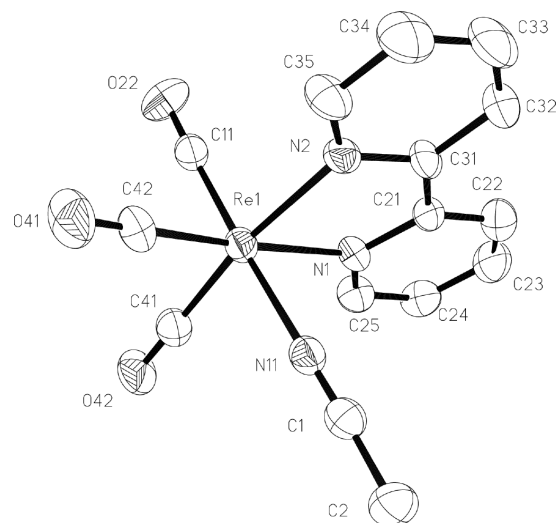


Figure 1

A view of one of the complex cations of the asymmetric unit of (I), with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

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

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Table 1

Selected geometric parameters (Å).

Re1—C11	1.917 (16)	Re2—C102	1.907 (16)
Re1—C42	1.922 (15)	Re2—C104	1.908 (16)
Re1—C41	1.927 (14)	Re2—C101	1.939 (17)
Re1—N11	2.157 (12)	Re2—N12	2.145 (12)
Re1—N1	2.163 (10)	Re2—N101	2.169 (10)
Re1—N2	2.169 (10)	Re2—N102	2.175 (10)

The H atoms were positioned geometrically (C—H distance fixed at 0.96 or 0.93 Å), assigned isotropic displacement parameters and allowed to ride on their respective C atoms. The highest peak and deepest hole are 1.08 Å from Re1 and 0.92 Å from Re2, respectively.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SMART* and *SAINT* (Siemens, 1994); data reduction: *SAINT* and *XPREF* (Siemens, 1994); program(s) used to solve structure: *SHELXTL* (Siemens, 1994); program(s) used to refine structure: *SHELXTL*;

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