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Bis[1,3-bis(pentafluorophenyl)triazenido]-tricarbonylosmium(II)

Bai-Wang Sun,[a]* Hui Liu,[a] Guo-Ying Yang,[b] Siau Gek Ang[b] and How Ghee Ang[b]

[a]Department of Chemistry and Chemical Engineering, Southeast University, Nanjing 210096, People's Republic of China, and [b]Department of Chemistry, Faculty of Science, National University of Singapore, 3 Science Drive 3, Singapore 117543

Correspondence e-mail: chmsunbw@seu.edu.cn

Key indicators

Single-crystal X-ray study
T = 223 K
Mean σ(C–C) = 0.007 Å
R factor = 0.022
wR factor = 0.065
Data-to-parameter ratio = 13.5

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

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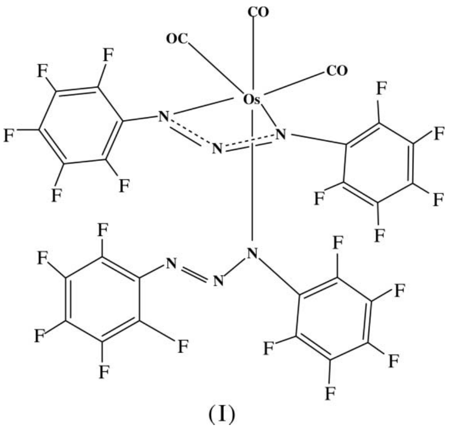
The title compound, [Os($C_{12}F_{10}N_3$)$_2$(CO)$_3$], has been synthesized by the reaction of $C_6F_5N{=}NNHC_6F_5$ with $H_2(Os)_3(CO)_{10}$. The OsC_3N_3 coordination is octahedral around the Os center. Two bonding modes, chelating and monodentate, are observed for the triazenyl groups.

Comment

Transition metal–carbonyl clusters have been widely used in reactions with various organic ligands containing N and S atoms, leading to the formation of many new coordination compounds with novel structures and properties (Akther *et al.*, 2003; Ang *et al.*, 1996; Canal *et al.*, 2003; Fink *et al.*, 1999; Kiriakidou-Kazemifar *et al.*, 2001; Kramer *et al.*, 2002; Pomogailo *et al.*, 2002; Tunik *et al.*, 2001). Our group has recently reported a hydrazino-edge-bridged triangular triosmium cluster as part of our investigation of metal compounds with N-containing pentafluorophenyl ligands (Sun *et al.*, 2005). Here we report a new osmium compound, (I) (Fig. 1), with bis(pentafluorophenyl)triazenide ligands.



(I)

In this osmium compound, the coordination around the Os center is distorted octahedral, composed of three terminal carbonyl groups and two mutually *cis* triazenyl ligands. Two N atoms from the bidentate $C_6F_5N_3C_6F_5$ ligand and two C atoms from two carbonyl groups define the equatorial plane, the average atom displacement being 0.0322 (19) Å. The Os center lies above this plane by 0.0195 (21) Å. The axial positions are occupied by one N atom from the monodentate $C_6F_5N_3C_6F_5$ ligand and one C atom of the third carbonyl group. The two equatorial Os—C bond lengths are 1.925 (4) and 1.913 (4) Å for Os1—C1 and Os1—C3, respectively. The axial Os1—C2 bond length is 1.934 (5) Å, longer than the above two. The C—O bond lengths and the Os—C—O angles are similar to those observed in other osmium–carbonyl

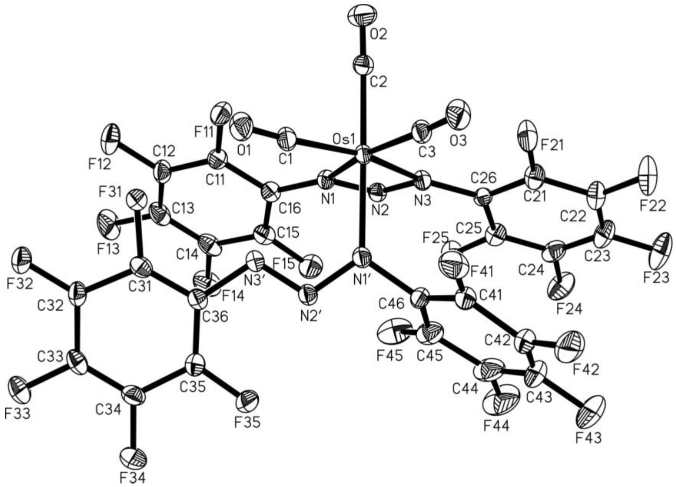


Figure 1
A view of (I) with 30% probability displacement ellipsoids.

compounds (Akther *et al.*, 2003; Canal *et al.*, 2003; Fink *et al.*, 1999; Kiriakidou-Kazemifar *et al.*, 2001; Kramer *et al.*, 2002; Pomogailo *et al.*, 2002; Sun *et al.*, 2005; Tunik *et al.*, 2003). Selected bond angles and lengths are listed in Table 1.

The triazenyl groups present two different bonding modes in this mononuclear compound, *viz*. 1,3-bidentate and monodentate. The N—N—N angle is 103.8 (3)° for the bidentate equatorial $C_6F_5N_3C_6F_5$ and 113.1 (3)° for the monodentate axial one. In (I), the N═N bond (Table 1) is longer than the characteristic value for a double bond (1.24 Å), whereas the N—N bonds are shorter than the characteristic value for a single bond (1.44 Å; International Tables for X-ray Crystallography, 1985, Vol. III). However, these values are in good agreement with those found in related compounds (Hörner *et al.*, 2004; Walton *et al.*, 1991; Zhang *et al.*, 1999).

The dihedral angle between the N1/N2/N3/Os1 least-squares plane and the C11–C16 ring is 29.86 (16)°. The N1/N2/N3/Os1 plane and the C21–C26 ring are inclined at 20.7 (2)°. For the N3′/N2′/N1′ plane and the C31–C36 ring, this dihedral angle is 13.7 (4)°, and for N1′/N2′/N3′ and the C41–C46 ring, the value is 66.8 (2)°.

Experimental

The ligand $C_6F_5N{=}NNHC_6F_5$ was prepared as described by Hörner *et al.* (2004) using $C_6F_5NH_2$ as the starting material. The product was recrystallized from tetrahydrofuran (THF) and well defined crystals were obtained by slow evaporation of a THF/hexane (1:1) solution. For the preparation of (I), $[H_2Os_3(CO)_{10}]$ (0.150 g, 0.176 mmol) was added to a hexane solution (6 ml) of $C_6F_5N{=}NNHC_6F_5$ (0.111 g, 0.293 mmol) and the mixture was stirred at 373 K for 22 h. The solvent was removed under reduced pressure and the residue was purified by TLC on silica gel (eluent: dichloromethane/ hexane, ratio 1:9). Upon crystallization, yellow single crystals of (I) were obtained. Analysis calculated for $C_{27}F_{20}N_6O_3Os$: C 25.15, N 5.33%; found: C 25.57, N 5.98%. IR (KBr, cm^{-1}): ν (CO) 2126 (*vs*), 2063 (*vs*), 2022 (*s*), 2082 (*m*), 2049 (*vs*), 2011 (*vs*); ν(C_6 F_5) 1515 (*vs*), 992 (*vs*); ν(N═N) 1276 (*vs*); ^{13}C NMR ($CDCl_3$/TMS): δ 187.7, 177.5, 169.3, 166.2 (CO); 145.1–149.3 (*m*, C_6F_5).^{19}F NMR ($CDCl_3$/TMS): δ −72.5 (*d*, 2F), −75.5 (*d*, 6F), −79.7 (*m*, 4F), −83.2 to −87.6 (*m*, 8F).

Crystal data

$[Os(C_{12}F_{10}N_3)_2(CO)_3]$
M_r = 1026.53
Orthorhombic, $P2_12_12_1$
a = 12.4352 (7) Å
b = 14.7412 (8) Å
c = 16.5795 (9) Å
V = 3039.2 (3) Å^3
Z = 4
D_x = 2.243 Mg m^{-3}
Mo *Kα* radiation
μ = 4.36 mm^{-1}
T = 223 (2) K
Block, yellow
0.20 × 0.15 × 0.10 mm

Data collection

Bruker AXS SMART CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1998)
T_{min} = 0.462, T_{max} = 0.648
21297 measured reflections
6961 independent reflections
6717 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.022
$wR(F^2)$ = 0.065
S = 1.10
6961 reflections
514 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0328P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 1.03 e Å^{-3}
$\Delta\rho_{min}$ = −0.55 e Å^{-3}
Absolute structure: Flack (1983)
Flack parameter: −0.002 (5)

Table 1
Selected geometric parameters (Å, °).

Os1—C3	1.913 (4)	N1′—C46	1.424 (5)
Os1—C1	1.925 (4)	N2′—N3′	1.276 (5)
Os1—C2	1.934 (5)	N3′—C36	1.404 (5)
Os1—N1	2.113 (3)	N3—N2	1.315 (5)
Os1—N1′	2.113 (4)	N3—C26	1.402 (5)
Os1—N3	2.138 (4)	N2—N1	1.307 (5)
N1′—N2′	1.325 (5)	N1—C16	1.402 (5)
C3—Os1—C1	87.53 (18)	N1—Os1—N1′	85.13 (13)
C3—Os1—C2	90.9 (2)	C3—Os1—N3	107.96 (16)
C1—Os1—C2	91.89 (19)	C1—Os1—N3	164.26 (15)
C3—Os1—N1	166.00 (16)	C2—Os1—N3	90.74 (16)
C1—Os1—N1	106.47 (16)	N1—Os1—N3	58.09 (14)
C2—Os1—N1	88.38 (18)	N1′—Os1—N3	84.35 (14)
C3—Os1—N1′	95.02 (17)	N3′—N2′—N1′	113.1 (3)
C1—Os1—N1′	91.62 (16)	N1—N2—N3	103.8 (3)
C2—Os1—N1′	173.28 (18)		

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

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