

Decacarbonyl- $1\kappa^3C, 2\kappa^3C, 3\kappa^4C$ -(μ -pentafluorophenyl-hydrazine- $1\kappa N:2\kappa N'$)-triangulo-triosmium: a hydrazino-edge-bridged triangular triosmium clusterBai-Wang Sun,^{a*} Mei-Su Zhang,^a
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Key indicators

Single-crystal X-ray study

 $T = 285\text{ K}$ Mean $\sigma(C-C) = 0.013\text{ \AA}$ R factor = 0.040 wR factor = 0.112

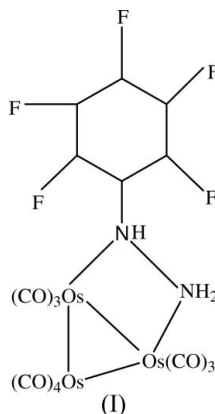
Data-to-parameter ratio = 19.3

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

The title compound, $[\text{Os}_3(\text{C}_6\text{F}_5\text{NHNH}_2)(\text{CO})_{10}]$, contains a near regular triangle of Os atoms. Two of the metal atoms are bridged by the hydrazine group of the $\text{C}_6\text{F}_5\text{NHNH}_2$ ligand. Ten carbonyl groups complete the cluster, resulting in a distorted octahedral coordination for each Os atom.

Comment

In recent years, transition-metal carbonyl clusters have received considerable attention owing to their important role in catalytic reactions (Ojima *et al.*, 1991) as well as the preparation of materials with novel magnetic properties (Pomogailo *et al.*, 2002). Different organic ligands containing N and S atoms can stabilize the metal cluster framework by means of chelating and bridging (Akther *et al.*, 2003; Deeming *et al.*, 1992; Au *et al.*, 1995).



We report here the synthesis and structure of the title compound, (I), containing a triangle of Os atoms and the organic ligand $\text{C}_6\text{F}_5\text{NHNH}_2$, which acts as a μ^1, μ^1 - N, N -bidentate bridge connecting two Os atoms. Presumably, the electron-withdrawing pentafluorobenzene group helps to stabilize the cluster through its substituted hydrazine group. The molecule of (I) (Fig. 1) consists of an Os_3 triangle with ten terminal CO ligands, and a substituted hydrazine group. Each Os coordination environment is distorted octahedral (including the Os–Os bonds), with Os3 bonded to four terminal carbonyl ligands, and Os1 and Os2 bonded to three terminal carbonyl ligands and one N atom from the $\text{C}_6\text{F}_5\text{NHNH}_2$ grouping.

The Os–Os bond lengths in (I) range from 2.8456 (6) to 2.8912 (9) Å (Table 1), the Os1–Os2 bond spanned by the ligand being the longest. The Os–Os–Os bond angles are all close to 60° . The N1–Os2–Os1 and Os2–Os1–N2 bond angles are $65.18(2)^\circ$ and $68.39(2)^\circ$, respectively. These values are similar to the equivalent data for related clusters (Akther *et al.*, 2003).

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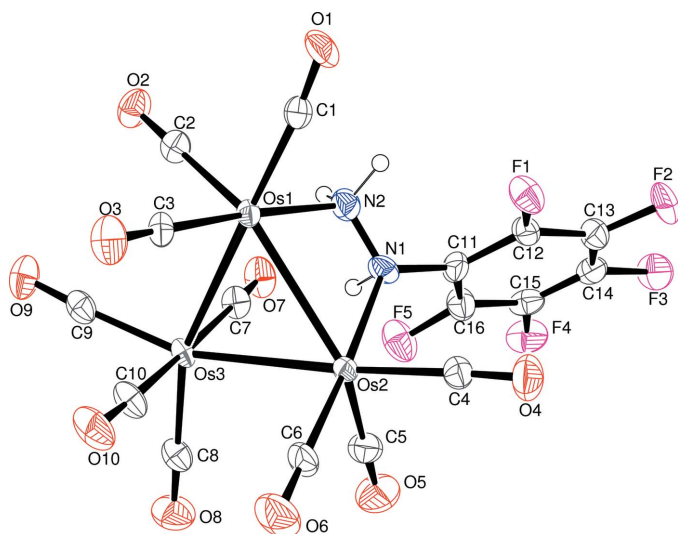


Figure 1
A view of (I), showing 30% probability displacement ellipsoids.

The torsion angles $C1-Os1-Os2-C4 = 116.13 (24)^\circ$, $C2-Os1-Os2-C5 = 139.04 (26)^\circ$, $C3-Os1-Os2-C6 = 110.22 (29)^\circ$ and $C2-Os1-Os3-C1 = 89.35 (23)^\circ$. The dihedral angles between the Os_3 ring and the $Os1/Os2/N1/N2$ mean plane and the pentafluorobenzene mean plane are $74.80 (11)$ and $54.03 (19)^\circ$, respectively, and the dihedral angle between the $Os1/Os2/N1/N2$ mean plane and the pentafluorobenzene mean plane is $79.01 (22)^\circ$.

Experimental

$[Os_3(CO)_{10}(MeCN)_2]$ (0.25 g, 0.21 mmol) was added to a CH_2Cl_2 solution (20 ml) of $C_6F_5NHNH_2$ (0.044 g, 0.22 mmol) and the mixture was stirred at room temperature for one hour. The solvent was removed under reduced pressure and the residue was purified by TLC on silica gel (eluant dichloromethane). Upon crystallization from dichloromethane, yellow single crystals of (I) were obtained. Analysis calculated for $C_{16}H_3F_5N_2O_{10}Os_3$: C 18.3, H 0.29, N 2.67%; found: C 18.30, H 0.32, N 2.69%. IR (KBr, cm^{-1}): ν CO 2113 (*m*), 2072 (*m*), 2065 (*s*), 2032 (*m*), 2024 (*vs*), 2011 (*vs*). 1H NMR ($CDCl_3/TMS$): δ 1.949 (*d*, 2H), 2.035 (*t*, 1H).

Crystal data

$[Os_3(C_6H_3F_5N_2)(CO)_{10}]$
 $M_r = 1048.80$
Monoclinic, $P2_1/c$
 $a = 14.816 (3) \text{ \AA}$
 $b = 7.6177 (15) \text{ \AA}$
 $c = 19.153 (4) \text{ \AA}$
 $\beta = 90.46 (3)^\circ$
 $V = 2161.6 (7) \text{ \AA}^3$
 $Z = 4$

$D_x = 3.223 \text{ Mg m}^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 1924 reflections
 $\theta = 1.4\text{--}30.0^\circ$
 $\mu = 17.69 \text{ mm}^{-1}$
 $T = 285 (2) \text{ K}$
Block, yellow
 $0.18 \times 0.16 \times 0.15 \text{ mm}$

Data collection

Bruker SMART CCD diffractometer
 φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 1998)
 $T_{\min} = 0.056$, $T_{\max} = 0.069$
17316 measured reflections

6269 independent reflections
5341 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.060$
 $\theta_{\max} = 30.0^\circ$
 $h = -20 \rightarrow 13$
 $k = -10 \rightarrow 10$
 $l = -26 \rightarrow 22$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.040$
 $wR(F^2) = 0.112$
 $S = 1.06$
6269 reflections
325 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0533P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 2.48 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -3.03 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (\AA , $^\circ$).

Os1—Os3	2.8456 (6)	Os1—N2	2.112 (8)
Os1—Os2	2.8912 (9)	Os2—N1	2.108 (6)
Os2—Os3	2.8657 (6)		
Os1—Os3—Os2	60.82 (2)	N1—N2—Os1	109.2 (6)
Os3—Os1—Os2	59.93 (3)	N2—N1—Os2	117.4 (6)
Os3—Os2—Os1	59.244 (16)		

H atoms were positioned geometrically ($N-H = 0.90\text{--}0.91 \text{ \AA}$) and refined as riding, with $U_{\text{iso}}(H) = 1.2U_{\text{eq}}(N)$. The highest peak is located 0.84 \AA from atom Os1 and the deepest hole is 0.77 \AA from Os1.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINTE* (Bruker, 1998); data reduction: *SAINTE*; 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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