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Nonacarbonyl-μ_3-*N*,*N*-diethyldithiocarbamato-$\kappa^3 S:S:S'$-μ-hydrido-triosmium(3 *Os*—*Os*)

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.009 Å
R factor = 0.028
wR factor = 0.067
Data-to-parameter ratio = 13.6

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

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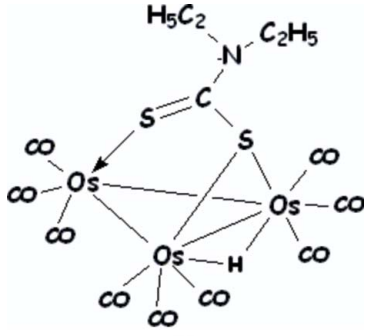
The molecular structure of the title compound, $[Os_3(C_5H_{10}NS_2)H(CO)_9]$, consists of an Os_3 triangle with a bridging dithiocarbamate ligand acting as a five-electron donor; a hydride ligand is located between two Os atoms. Three terminal carbonyl groups on each Os atom complete the structure. There are two independent molecules in the asymmetric unit.

Comment

Arce *et al.* (1985) have reported the synthesis and spectroscopic characterization of the osmiun cluster $[Os_3(\mu\text{-H})\{\mu^3,\eta^2\text{-}(S,S)\text{-}S_2CNEt_2\}(CO)_9]$. We describe here the crystal structure of this compound, (I) (Fig. 1).



(I)

The molecular structure is based on a triangle of three Os atoms; there are two short and one long Os—Os bonds. The Os atoms are bridged by a μ_2-hydride and a μ_3-dithiocarbamate ligand. The Os—Os distances are similar to distances reported in $[Os_3(\mu\text{-H})(CCSiMe_3)(CO)_9]$ [2.833 (1), 2.846 (1) and 2.843 (1) Å; Lewis *et al.*, 1992] and $[Os_3(\mu\text{-H})(\mu\text{-}\eta^2\text{-}C_{12}H_7)(CO)_{10}]$ [2.8349 (2), 2.8768 (2) and 2.8799 (3) Å; Adams *et al.*, 2003]. The μ^3,η^2-dithiocarbamate ligand behaves as a five-electron donor through the S atoms. The Os—S distances compare well with those found in $[Os_3(\mu\text{-H})\{\mu\text{-}SCH_2CH_2C(H)=C(H)CH_2CO\}(CO)_{10}]$ [2.419 (5) and 2.423 (4) Å; Adams & Perrin, 2000] and related complexes containing bridging sulfur–osmium linkages, *e.g.* $[Os_3(\mu\text{-H})\{\mu,\eta^2\text{-}SC=NCH=CHN(CH_3)\}(CO)_{10}]$ [2.415 (3) Å; Azam *et al.*, 2002] and $[Os_3(\mu\text{-H})(\eta\text{-}SC=NCH_2CH_2S)(CO)_{10}]$ [2.416 (11) and 2.419 (8) Å; Brodie *et al.*, 1986]. The C—S distances are also comparable to values reported for transition metal dithiocarbamates (Goh *et al.*, 2001; Lu *et al.*, 2004; Heard *et al.*, 2000). The C—N distance is indicative of double-bond character, as noted from the C=N distances of 1.37 (3)–1.40 (3) Å found in $Cp_6Cr_8S_8(S_2CNEt_2)_2$ (Goh *et al.*, 2001). The hydride ligand is unsymmetrically located between two

Os atoms. Three terminal carbonyl ligands around each metal atom complete the structure. The two molecules in the asymetric unit are essentially identical.

Experimental

The title compound was synthesized according to the procedure of Arce *et al.* (1985). Treatment of $[Os_3(\mu\text{-H})(\mu\text{-OCH=CH}_2)(CO)_{10}]$ with $HB_4{\cdot}Et_2O$ followed by the addition of tetraethylammonium *N*,*N*-diethyldithiocarbamate gave $[Os_3(\mu\text{-H})\{\mu^3,\eta^2\text{-(S,S)-}S_2CNEt_2\}(CO)_9]$, which was thermolytically decarbonylated to give the title compound. Crystals were grown from a hexane solution at 253 K.

Crystal data

$[Os_3(C_5H_{10}NS_2)H(CO)_9]$
M_r = 971.96
Monoclinic, $P2_1/c$
a = 15.0412 (1) Å
b = 12.5376 (1) Å
c = 23.2430 (2) Å
β = 103.191 (1)°
V = 4267.53 (6) Å^3
Z = 8
D_x = 3.026 Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 9424 reflections
θ = 3.9–70.6°
μ = 35.26 mm^{-1}
T = 100 (2) K
Prism, orange
0.06 × 0.03 × 0.02 mm

Data collection

Bruker CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.323, T_{max} = 0.494
31582 measured reflections
7285 independent reflections
7049 reflections with $I > 2\sigma(I)$
R_{int} = 0.053
θ_{max} = 67.1°
$h = -17 \rightarrow 17$
$k = -14 \rightarrow 13$
$l = -26 \rightarrow 23$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.067
S = 1.18
7285 reflections
535 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.024P)^2 + 5.3186P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.66 e Å^{-3}
$\Delta\rho_{min}$ = −2.38 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Os1—S2	2.4318 (13)	Os3—H1	1.90 (8)
Os1—Os2	2.8171 (3)	S1—C10	1.689 (6)
Os1—Os3	2.8519 (3)	S2—C10	1.796 (6)
Os1—H1	1.98 (8)	C10—N1	1.316 (7)
Os2—S1	2.4461 (14)	C11—N1	1.486 (7)
Os2—Os3	2.7993 (3)	C13—N1	1.488 (8)
Os3—S2	2.4218 (14)		
Os2—Os1—Os3	59.176 (8)	N1—C10—S1	122.6 (4)
Os3—Os2—Os1	61.032 (8)	N1—C10—S2	116.7 (4)
Os2—Os3—Os1	59.792 (8)	S1—C10—S2	120.7 (3)
Os3—S2—Os1	71.97 (4)		

All H atoms were initially located in a difference Fourier map. The two hydrides H1 and H2 were refined independently with isotropic displacement parameters. All other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.98–1.00 Å and with $U_{iso}(H) = 1.2–1.5U_{eq}(C)$. The highest electron-density peak is located 0.93 Å from atom Os5 and the deepest hole is located 0.96 Å from Os4.

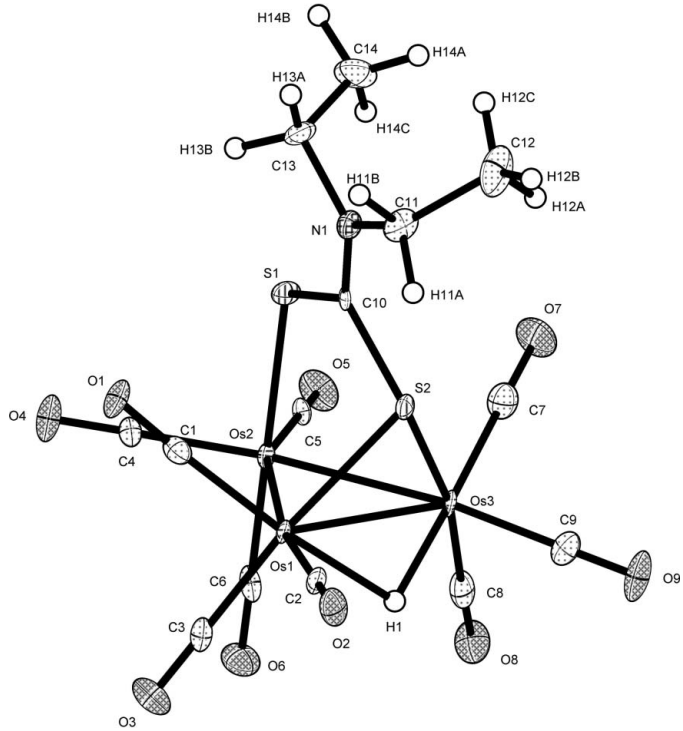


Figure 1
One of the independent molecules of the title compound. Displacement ellipsoids are drawn at the 50% probability level and H atoms are represented by circles of arbitrary size.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

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