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Bis(*tert*-butylimido)[2-(dimethylaminomethyl)-pyrrol-1-yl]tungsten(VI)-μ-oxo-bis(*tert*-butylimido)-(η^2-*N,O-tert*-butylnitroso)tungsten(VI)

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.007 Å
R factor = 0.026
wR factor = 0.063
Data-to-parameter ratio = 25.4

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

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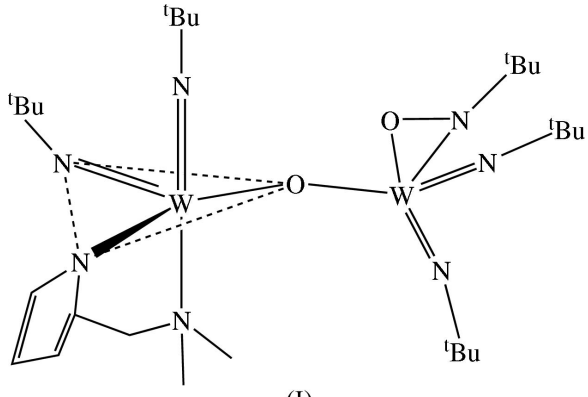
The structure of the title compound, $[W_2(C_4H_9N)_4(C_7H_{11}N_2)(C_4H_{10}NO)O]$, was determined at 150 K. It is an asymmetrical binuclear compound with a μ-oxo bridge linking two tungsten(VI) centers.

Comment

Organoimide chemistry has been investigated extensively and reviewed periodically (Nugent & Haymore, 1980; Nugent & Mayer, 1988). The organoimide compounds involving monoanionic monodentate ligands have been commonly observed. However, monoanionic bidentate organoimide complexes containing substituted pyrrole ligands are less reported (Huang *et al.*, 2000). Currently, we are investigating the chemistry of tungsten–imide complexes and bidentate 2-(dimethylaminomethyl)pyrrole (LH) and we present here the structure of the title compound, (I).



(I)

The title compound is a binuclear tungsten(VI) complex in which one of the tungsten centers involves a bidentate 2-(dimethylaminomethyl)pyrrolyl and two *tert*-butylimido ligands. The other tungsten center involves two *tert*-butylimido ligands and, intriguingly, an η^2-*N,O-tert*-butylnitroso ligand. The two tungsten centers are linked by a μ-oxo bridge with a bond angle of 159.69 (16) Å (Fig. 1).

In Fig. 1, the W atom on the left (W1) is in a distorted trigonal bipyramidal geometry, whereas that on the right (W2) is in a distorted tetrahedral geometry with the η^2-*N,O-tert*-butylnitroso ligand occupying one coordination site. The asymmetrical binuclear compound has no molecular symmetry.

Centrosymmetric pairs of intermolecular hydrogen bonds of the type N—H···O are present in the crystal structure (Table 1). Fig. 2 shows the crystal packing.

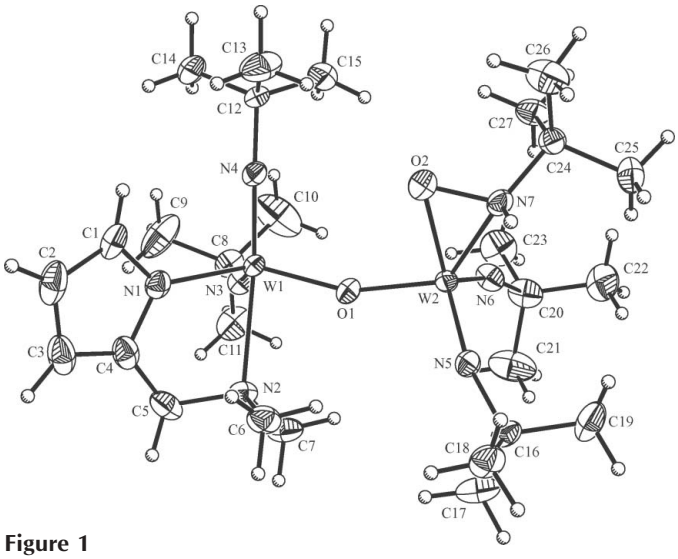


Figure 1
The structure of (I), showing 50% probability displacement ellipsoids for the non-H atoms.

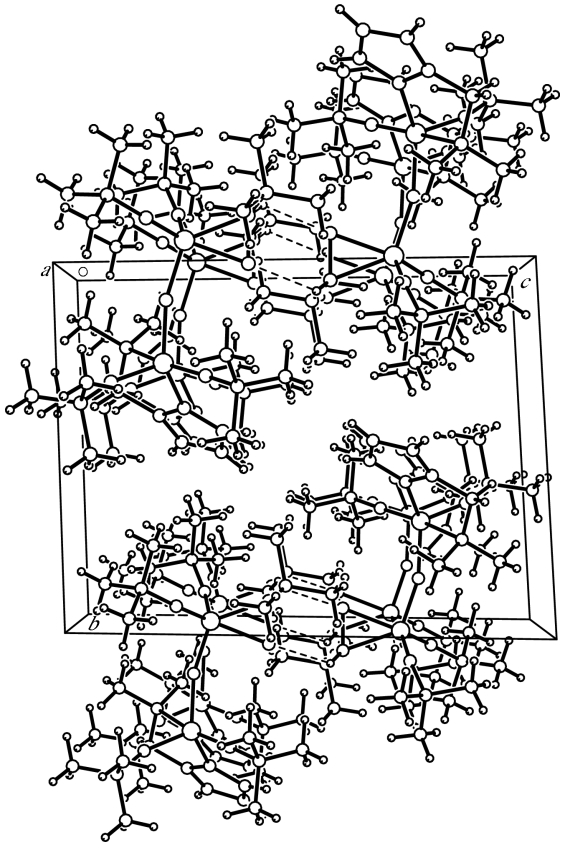


Figure 2
The packing of (I), viewed along the *a* axis. Dashed lines indicate hydrogen bonds.

Experimental

Crystals of the title complex were obtained from the reaction of 2-(dimethylaminomethyl)pyrrole (0.66 g, 5 mmol) and bis(*tert*-butlyimido)bis(*tert*-butylamido)tungsten (2.5 g, 5 mmol) in diethyl ether at 195 K for 12 h followed by repeated recrystallization from a diethyl ether solution.

Crystal data

$[W_2(C_4H_9N)_4(C_7H_{11}N_2)(C_4H_{10}NO)O]$
$M_r = 879.50$
Triclinic, $P\overline{1}$
$a = 9.6263$ (3) Å
$b = 12.0153$ (3) Å
$c = 15.6375$ (4) Å
$\alpha = 86.334$ (2)°
$\beta = 79.479$ (2)°
$\gamma = 83.175$ (2)°
$V = 1764.02$ (8) Å^3
$Z = 2$
$D_x = 1.656$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5880 reflections
$\theta = 2.3$–28.8°
$\mu = 6.55$ mm^{-1}
$T = 150$ (2) K
Block, colorless
0.53 × 0.34 × 0.27 mm

Data collection

Bruker SMART APEX-II diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{min} = 0.080$, $T_{max} = 0.171$
25718 measured reflections
9142 independent reflections
7668 reflections with $I > 2\sigma(I)$
$R_{int} = 0.029$
$\theta_{max} = 28.9°$
$h = -12 \rightarrow 13$
$k = -16 \rightarrow 16$
$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.026$
$wR(F^2) = 0.063$
$S = 1.04$
9142 reflections
360 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0236P)^2 + 3.8003P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 3.04$ e Å^{-3}
$\Delta\rho_{min} = -1.90$ e Å^{-3}

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N7—H7···O2^{i}	0.93	2.53	3.220 (4)	132

Symmetry code: (i) $1-x, -y, 1-z$.

All H atoms were positioned geometrically (C—H = 0.95–0.99 Å) and refined with a riding model, with $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl H atoms and $1.2U_{eq}(C)$ for all other H atoms. The maximum and minimum electron-density peaks are 0.74 and 0.62 Å, respectively, from atom W2.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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